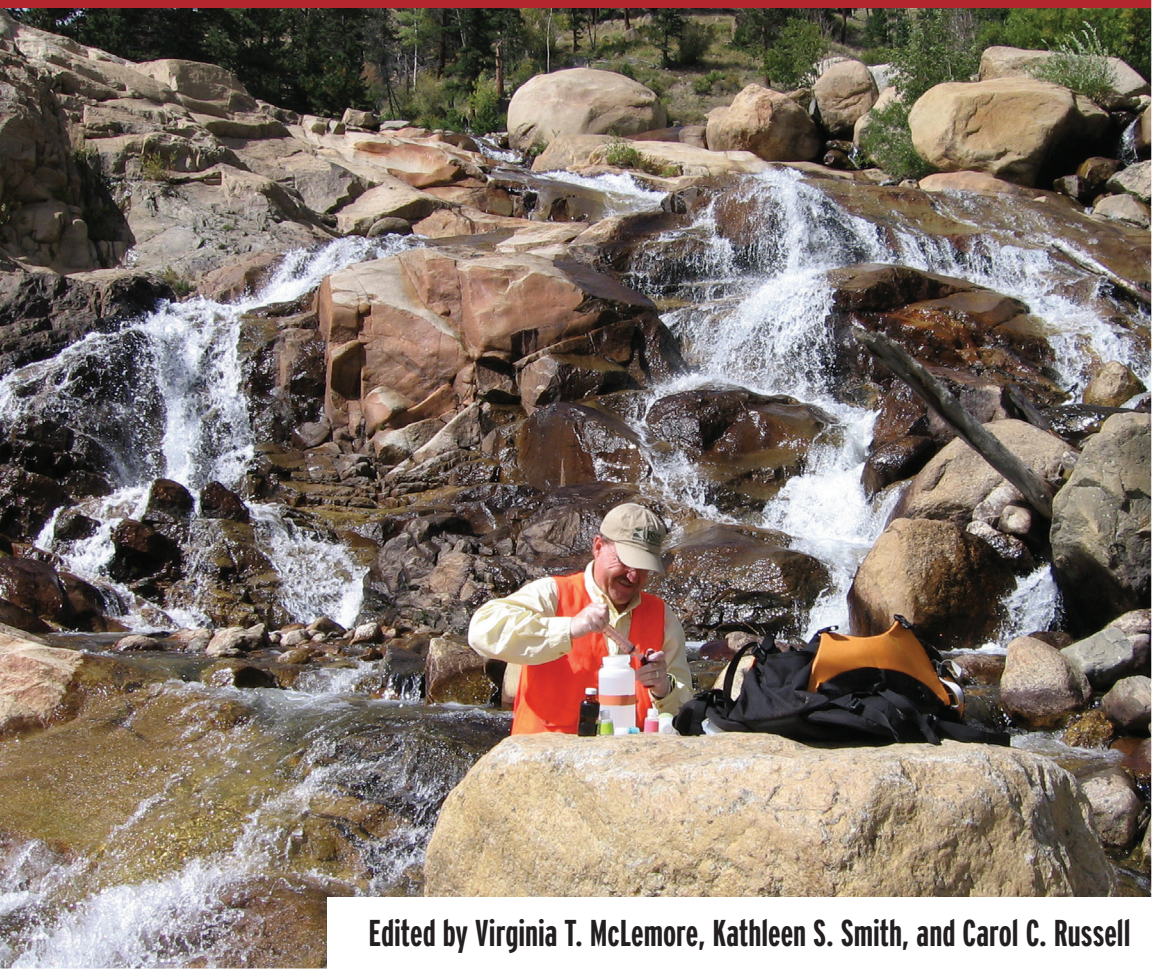
Management Technologies for Metal Mining Influenced Water

Sampling and Monitoring for the Mine Life Cycle

Volume 6



Edited by Virginia T. McLemore, Kathleen S. Smith, and Carol C. Russell

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DISCLAIMER

This handbook is not intended to provide all of the information needed to conduct sampling and monitoring at mining influenced sites. 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. Any use of trade, firm, or product names is for descriptive purposes only and does not imply endorsement by the U.S. government. The views expressed in this handbook are those of the authors and editors and do not necessarily represent the views of the U.S. Environmental Protection Agency or of the U.S. government. This handbook has been peer reviewed and approved for publication consistent with U.S. Geological Survey Fundamental Science Practices (http://pubs.usgs.gov/circ/1367/, accessed August 23, 2013).

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Appendix 5 **Case Studies of Sampling and Monitoring**

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Preface

Sampling and Monitoring for the Mine Life Cycle is volume 6 in the Management Technologies for Metal Mining Influenced Water series prepared through the Acid Drainage Technology Initiative–Metal Mining Sector (ADTI-MMS). Published handbooks in the series include *Basics of Metal Mining Influenced Water*; *Mitigation of Metal Mining Influenced Water*; *Mine Pit Lakes: Characteristics, Predictive Modeling, and Sustainability*; *Techniques for Predicting Metal Mining Influenced Water*; and *Geochemical Modeling for Mine Site Characterization and Remediation*. Each handbook undergoes a consensus review process that involves approval by the appropriate ADTI-MMS technical committee and ADTI-MMS membership.

ADTI-MMS 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. ADTI-MMS is part of the Acid Drainage Technology Initiative (ADTI), which is the United States' regional member of the Global Alliance sponsored by the International Network for Acid Prevention (INAP). The mission of ADTI-MMS is to identify, evaluate, develop, and disseminate information about cost-effective, environmentally sound methods and technologies to manage mine wastes and related metallurgical materials for abandoned, active, and future mining and associated operations, and to promote understanding of these technologies. Drainage quality issues are considered for ore and waste as well as from alkaline, neutral, and acidic conditions in the mining and related metallurgical processing environment. ADTI-MMS does not address policy or regulatory matters. The co-chairs of the ADTI-MMS Sampling and Monitoring Technical Committee during the preparation of this handbook were Virginia T. McLemore (New Mexico Bureau of Geology and Mineral Resources [NMBGMR]), Kathleen S. Smith (U.S. Geological Survey [USGS]), and Carol C. Russell (U.S. Environmental Protection Agency [USEPA]).

This technical handbook is intended for planners, regulators, consultants, land managers, researchers, students, stakeholders, and anyone with an interest in mining influenced waters. The aim of this handbook is to provide specialized information applicable to sampling and monitoring activities at mining sites. This handbook is intended to supplement and enhance existing sampling and monitoring publications, and is not intended to provide policy or regulatory information.

This handbook greatly benefited from contributions and reviews by numerous experts listed in the front material. We thank them all for their hard work and patience during the preparation of this handbook. We extend special thanks to Charles Bucknam, Linda Figueroa, Jane Olivier, Doug Peters, and Dennis Turner for their leadership, support, encouragement, and tenacity. The NMBGMR (New Mexico Institute of Mining and Technology), U.S. Office of Surface Mining Reclamation and Enforcement (initial funding for report writing and obtaining MEND documents), and USEPA (travel funds) provided direct funding in support of this handbook. We acknowledge the in-kind support provided by companies, institutions, and agencies for contributors and reviewers to participate in the preparation of this handbook. We especially thank the NMBGMR, USGS (Mineral Resources Program and Toxic Substances Hydrology Program), and USEPA (Region 8 and the Office of Research and Development) for providing salary support for this work.

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CHAPTER 1

Introduction

Kathleen S. Smith and Virginia T. McLemore

1.1 SYNOPSIS

This handbook provides an overview of basic aspects of sampling for environmental purposes and monitoring of environmentally relevant variables at mining sites. While focusing mainly on environmental sampling and monitoring of surface water, also considered are groundwater, process water streams, rock (native, mined, and processed), soil, and other media including air and biological organisms. The objectives of this handbook are to

- Endorse the principle that sampling and monitoring programs be designed to
  - Account for all aspects of the mine life cycle, and
  - Plan for closure throughout the mine life cycle;
- Discuss considerations for environmental sampling and monitoring at mining sites during the various phases of mining;
- Supply technical information and suggest resources that relate to sampling and monitoring at mining sites; and
- Provide an approach for and guidance on design of sampling and monitoring programs at mining sites.

This handbook will be useful to mine managers and planners, laboratory staff, consultants, regulators, researchers, undergraduate and graduate students, communities affected by mining activities, nongovernmental organizations, and the general public.

This is a technical document and is not intended to be used for policy or regulatory purposes. Furthermore, this handbook is not designed to be an all-inclusive compendium of information about sampling and monitoring of acidic drainage and other mining influenced water (MIW; term coined by Schmiermund and Drozd 1997) or natural drainage. Instead, it provides key information, technical details, and resources that can assist readers in making informed decisions about sampling and monitoring at mining sites. This handbook is intended to supplement and enhance current literature, and strives to provide an awareness of the critical components and complexities involved in environmental sampling and monitoring at mining sites. It differs from most information sources by providing an approach to address all types of MIW and other sampling media throughout the mine life cycle.

1.2 ORGANIZATION OF THIS HANDBOOK

This handbook is organized into a main text and six appendices. Sidebars and illustrations are included throughout the main text to provide additional detail about important concepts, to

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present examples and brief case studies, and to suggest resources for further information. References cited are listed at the end of each chapter.

The main text includes topical chapters that discuss various aspects of environmental sampling and monitoring at mining sites.

- Chapter 2 describes sampling and monitoring needs during the mine life cycle. The phases of mining are described, and the issues, challenges, sampling, monitoring, and characterization needs are specified for each mining phase.
- Chapter 3 discusses sampling considerations that relate to mining sites. Specialized sampling issues and relevant research findings for mining sites are described, and additional resources are suggested for further information.
- Chapter 4 provides an overview of risk and of international sampling and monitoring guidance documents. A stepwise framework is presented for developing and implementing a sampling and monitoring program at mining sites.
- Chapter 5 describes the planning process for sampling and monitoring at mining sites. It includes a discussion of the importance of conceptual models in this process.
- Chapter 6 covers the various aspects of implementing a sampling program at mining sites.
- Chapter 7 provides an overview of the decision-making process. It includes a discussion of the steps involved in decision making and highlights some of the specialized considerations for mining sites.
- Chapter 8 discusses key issues and research topics that need to be pursued at mining sites.

Appendices are included to supplement the main text. The appendices are available on the included CD-ROM and are an integral part of this handbook.

- Appendix 1 is a list of Web-site resources that address sampling and monitoring topics.
- Appendix 2 is a summary specifically devoted to ASTM standard methods that pertain to sampling and monitoring at mining sites.
- Appendix 3 contains technical summaries written by subject-matter experts that describe field measurements, collection methods, and analytical techniques and procedures, and discuss principles, limitations, and uses of various sampling and monitoring techniques. This appendix is designed to be a valuable resource to supplement concepts and methods discussed in the main text.
- Appendix 4 contains some examples of historical sampling plans for mining-related sites and also includes a USEPA (U.S. Environmental Protection Agency) Region 8 review form for Quality Management Plans.
- Appendix 5 is a collection of case studies from mining-related sites that demonstrate sampling and monitoring applications and discuss lessons learned. The case studies are written by scientists, and illustrate concepts and methods discussed in this handbook.
- Appendix 6 illustrates applications of GEMs (geo-environmental models) at mining sites using case histories.

1.3 ROLE OF THIS HANDBOOK

At present, there are few authoritative guides available that address environmental sampling and monitoring approaches for MIW throughout the mine life cycle. The Mine Environment Neutral Drainage (MEND) program has worked to develop technologies to prevent and control acidic drainage since 1989 and has published numerous significant reports concerning MIW. The Acid

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Drainage Technology Initiative–Coal Mining Sector has produced two handbooks on acid mine drainage (AMD) as it relates to coal mining (Skousen et al. 1998; Kleinmann 2000). Since the 1980s, numerous general sampling and monitoring techniques have been developed throughout the world by USEPA, USGS (U.S. Geological Survey), and other organizations (TAEM and SENES 1997; USBR 2001; Tremblay and Hogan 2001a, 2001b; Popeck 2003; Downing 2010). The *Global Acid Rock Drainage Guide* (GARD Guide; INAP 2009) includes a section on monitoring but does not describe sampling methods and sampling strategy in detail, except briefly in some of the individual chapters. Hence, there are limited descriptions of the specialized approach necessary for environmental sampling and monitoring at mining sites. This handbook endeavors to fill that niche by providing specialized information that addresses sampling of all types of MIW and other sampling media throughout the mine life cycle.

1.4 IMPORTANCE OF DEFINING QUESTIONS

A recurring theme throughout this handbook is the importance of defining the questions being addressed and the objectives of the investigation. Prior to beginning a sampling and monitoring program, it is essential to know what data and information are necessary, how they are to be used, what sampling media will target which issues, the important analytes, and the desired degree of confidence in the answer(s). Sampling and monitoring decisions cannot be made, and protocols cannot be prepared, until these issues have been addressed. Various aspects of question definition are discussed later in this handbook.

It is good practice to think beyond regulatory data requirements and attempt to use data-collection opportunities for a variety of purposes. For example, additional data collection can provide valuable insights in developing conceptual models that guide sampling and monitoring

ADDITIONAL INFORMATION ABOUT SAMPLING AND MONITORING AT MINING SITES

"ADTI-MMS Sampling and Monitoring for the Mine Life Cycle" by V.T. McLemore, C.C. Russell, and K.S. Smith in *Securing the Future and 8th ICARD*, conference proceedings, June 23–26, 2009, Skelleftea, Sweden: www.proceedings-stfandicard-2009.com/pdfer/Virginia_McLemore_B4_T3_ADTI-MMS-Sampling-and-Monitoring-for-the-Mine-life-Cycle.pdf

MEND Manual and other reports: www.mend-nedem.org

"Monitoring" in *Global Acid Rock Drainage Guide* (GARD Guide) by the International Network for Acid Prevention: www.gardguide.com/index.php/Chapter_8

"Sampling and Monitoring for Closure" by V.T. McLemore, K.S. Smith, C.C. Russell, and the Sampling and Monitoring Committee of the ADTI-MMS in *Understanding and Responding to Hazardous Substances at Mine Sites in the Western United States*, 2007, edited by J.V. DeGraff. Reviews in Engineering Geology, Vol. 17. Boulder, CO: Geological Society of America. pp. 171–180. doi: 10.1130/2007.4017(11)

USEPA *Abandoned Mine Site Characterization and Cleanup Handbook* (2000), EPA 910-B-00-001: www.epa.gov/superfund/policy/remedy/pdfs/amscch.pdf

USEPA *Framework for Metals Risk Assessment* (2007), EPA 120/R-07/001: www.epa.gov/raf/metalsframework/pdfs/metals-risk-assessment-final.pdf

USGS mine drainage activities: http://mine-drainage.usgs.gov/

USGS Mineral Resources Data System (MRDS): http://tin.er.usgs.gov/mrds/

USGS *National Field Manual for the Collection of Water-Quality Data* (variously dated): http://water.usgs.gov/owq/FieldManual/

USGS watershed contamination from hard rock mining: http://toxics.usgs.gov/regional/mining/

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activities. Although regulatory guidelines require specific sampling and monitoring data, broader data collection can ultimately aid in making more reliable decisions.

1.5 SAMPLING AND MONITORING FOR CLOSURE

This handbook endorses the concept of sampling and monitoring for closure during all phases of the mine life cycle on a site-specific basis, from exploration (Hartman 1992) to mine planning, actual mining (operations), reclamation, and closure. Designing for ultimate closure of new mines or mine expansions is a relatively new concept. Because water-quality impacts are a major environmental concern at many mines, detailed assessment of potential impacts on water quality, as well as impacts on the environment in general, are required in most countries before mining can commence. These assessments entail numerous sampling and monitoring programs that begin during the exploration phase and continue throughout the mine life cycle. Geological and environmental variations between mines have fostered a broad range of techniques to understand, sample, monitor, predict, model, mitigate, and control MIW. It is important to ensure that the samples and data quality collected during the exploration phase also are acceptable for subsequent environmental modeling and prediction studies, and that the data can be used for planning for remediation and mine closure.

Mining sites generally are very complex with a variety of specialized sampling and monitoring requirements. The lifetime of a mine extends from the exploration phase to closure and postclosure, and can involve a time frame of many years. Over the lifetime of a mine, a variety of media types are sampled, companies and personnel can come and go, and regulatory requirements can change. These factors make the planning and maintenance of a sampling and monitoring program very challenging. Chapter 2 describes sampling and monitoring needs during the various phases of mining, and the Timing of Environmentally Related Sampling sidebar discusses some considerations in timing of data collection at mining sites.

1.6 TERMINOLOGY

A *sample* is a representative portion, subset, or fraction of a body of material representing a defined population (Wellmer 1998; Davis 1998; Schreuder et al. 2004; Neuendorf et al. 2005; Downing 2010). *Monitoring* is a widely used term, but its meaning can be broad and vague. In this handbook, monitoring is referred to as sampling or collecting data (e.g., visual observations or continuous field readings) over a period of time. Monitoring activities usually follow some sort of structured sampling schedule. All too frequently, sampling is an underappreciated function. It is important to remember that the success of a sampling program depends on the quality, integrity, and representativeness of the samples.

Problematic mine or natural drainage has been given many names including acid rock drainage (ARD), AMD, acidic drainage, and MIW. A discussion of each of these terms is provided in volume 1 of this Management Technologies for Metal Mining Influenced Water series, *Basics of Metal Mining Influenced Water* (McLemore 2008). The distinction between ARD and AMD sometimes has been misapplied, depending on whether drainage quality has been degraded by mining or is due, in part, to natural causes. In practice, the term *ARD* has been applied to all acidic drainage, mining-related or otherwise, and has generally replaced *AMD* in the metal mining drainage literature. Both terms refer to acidic, high mineral acidity, high sulfate (SO_4) waters from mines and natural sources where iron sulfide minerals have weathered in the presence of oxygen and water to form acidic or high metal concentration drainage. But not all adverse drainage from metal mines is acidic; some neutral pH, high metal concentration waters can be detrimental to the environment if not properly managed. In volume 1 of this series (McLemore

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TIMING OF ENVIRONMENTALLY RELATED SAMPLING IN THE MINE LIFE CYCLE

Ron L. Schmiermund, Economic and Environmental Geochemistry, Inc.

Timing of environmentally related sampling in the life cycle of a mine is singularly critical to a successful program, but it is an aspect of planning often ignored. Most obvious are baseline-monitoring programs that must take place in advance of planned disturbances. Failure to intelligently implement these programs very early in the exploration/prefeasibility process can result in a poor basis for permit applications and long-term monitoring.

Less obviously dependent on timing are waste rock characterization programs that require reliable drillhole information and can benefit greatly from geologic expertise. The expertise and acquisition of drillhole data co-exist most commonly during exploration and early feasibility phases. In addition, geo-environmental models (GEMs) are most easily and effectively applied and adapted at this time for the purposes of anticipating important variables. Exploration geologists, while available, will understand the implications of their site-specific findings with respect to the basic GEM, and may be able to adapt the drilling plan to provide additional environmentally relevant data. Timing and proactive intercession are critical.

As time passes, all samples deteriorate to varying degrees, but especially in highly sulfidic zones. Sometimes chemical changes and resulting losses of critical information occur in a period of hours or days. In addition, insightful information from project geologists is lost.

Also sensitive to timing is any interaction of environmental specialists with metallurgical testing programs. Such testing typically occurs at multiple times during the feasibility and developmental process, resulting in different and more relevant ore stockpiles and waste streams, each requiring characterization. Sampling of metallurgical process waste streams is best done promptly by persons with a vested interest in the required environmental testing so that relevant particulars of the often complex testing programs are not lost.

Timely interjection of environmental information into an emerging mine plan is the key to having a positive and cost-effective impact on the planning process. If a mine plan is already extensively developed, changes to address potential environmental impacts are likely to receive a cool reception or may simply be impossible to implement. Similarly, a late-stage discovery of significant environmental risks that must be avoided (e.g., leachable metals in critical construction material or abundant sulfides in highwall waste rock) can be very expensive to manage.

2008), the definition of MIW was expanded to include all waters affected by mining and metallurgical processing, which includes wastes from historical operations. This term resolves much of the confusion that exists from using AMD for cases where drainage is from mines but is not acidic, and MIW is the preferred term used to refer to all mining-related waters because acidic, neutral, and alkaline waters can all transport metals, anions, and other contaminants that can potentially impact the environment.

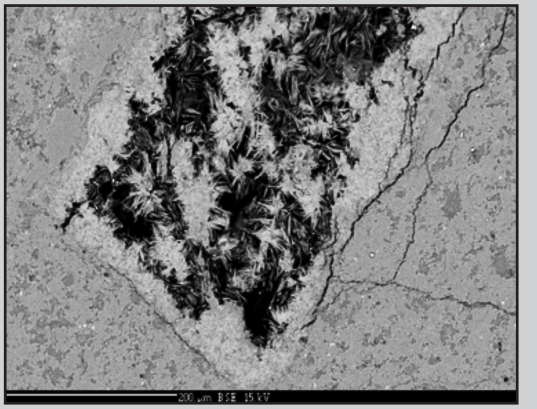
A significant miscommunication problem between scientists, engineers, regulators, and stakeholders (e.g., the public) is that there can be different definitions and interpretations for the same word. The legal definition can differ somewhat from the scientific definition or the common usage by mining companies, scientists of different disciplines, or the stakeholders. It is important that everyone speak the same language and use the same definitions. An example of one of these confusing terms is the word *clay*, which can refer to either particle size or a group of minerals (see Clay Size and Clay Minerals sidebar). Common scientific definitions are used wherever

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possible in this handbook. The introductory volume of this series (McLemore 2008) includes a comprehensive glossary that defines numerous technical terms relevant to and consistent with this handbook. It also introduces many concepts pertinent to mining.

CLAY SIZE AND CLAY MINERALS

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Photomicrograph of clay minerals (mostly sericite) replacing feldspar phenocryst in QSP (quartz-sericite-pyrite)-altered rhyolite (Amalia Tuff) from the Questa molybdenum mine, New Mexico. This sample is an unweathered drill core sample of the ore body in situ before mining.

Clay can be a confusing term that describes a specific group of minerals as well as a specific range in grain or particle size. Grain size distribution is an important physical characteristic of a soil and is different from the quantity of clay minerals. The gradation curve and the percentage of fines control the shear strength and compressibility of a soil or mine-rock pile. Clay minerals are hydrous aluminum phyllosilicates, typically with variable amounts of iron, magnesium, alkali elements, and other elements. Clay minerals are fundamentally built of tetrahedral and octahedral sheets. Geologists and soil scientists usually consider clay-size material to occur at a particle size of <2 μm (clays being finer than silts). Sedimentologists typically use <4 to 5 μm, and colloid chemists use <1 μm to define clay materials. Geotechnical engineers distinguish between silts and clays based on the plasticity properties of the soil, as measured by the Atterberg limits of the soil. Hydrothermal clay minerals can actually replace primary and secondary minerals within the rock fragments comprising soils or rock-pile material, and these replacement clays are not reflected in the grain size distribution and, therefore, do not affect the friction angle obtained from direct shear tests. The majority of the clay minerals are as replacements of other sand-size minerals (as illustrated in the photomicrograph). Therefore, the total amount of clay minerals is quite different from the total clay-size material, and this clay would not be expected to affect shear strength now or in the future.

GLOSSARIES RELATED TO MINING TERMINOLOGY

Basics of Metal Mining Influenced Water by V.T. McLemore, ed. 2008. Management Technologies for Metal Mining Influenced Water series, Vol. 1. Littleton, CO: SME.

Global Acid Rock Drainage Guide (GARD Guide) by the International Network for Acid Prevention (extensive online glossary): www.gardguide.com/index.php/Glossary

The Northern Miner's glossary of mining and mineral processing terms: www.northernminer.com/resources/tools/glossary.aspx

USEPA's Abandoned Mine Lands Program (online glossary): www.epa.gov/aml/gloss.htm

USGS's glossaries of scientific terms: http://water.usgs.gov/glossaries.html

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1.7 REFERENCES

Davis, B. 1998. What is a sample? What does it represent? *Aust. Inst. Geosci. Bull*. 22:39–34.

Downing, B. 2010. ARD sampling and sample preparation. http://technology.infomine.com/enviromine/ard/sampling/intro.html. Accessed 8/23/13.

Hartman, H.L. 1992. Introduction to mining. In *Mining Engineering Handbook*. Edited by H.L. Hartman. Littleton, CO: SME. pp. 3–42.

INAP (International Network for Acid Prevention). 2009. *Global Acid Rock Drainage Guide* (GARD Guide). www.gardguide.com/. Accessed 8/23/13.

Kleinmann, R.L.P. ed. 2000. *Prediction of Water Quality at Surface Coal Mines*. Morgantown, WV: West Virginia University, National Mine Land Reclamation Center. www.techtransfer.osmre.gov/nttmainsite/Library/hbmanual/predictH2O/predictH2O.pdf. Accessed 8/23/13.

McLemore, V.T. ed. 2008. *Basics of Metal Mining Influenced Water*. Management Technologies for Metal Mining Influenced Water series, Vol. 1. Littleton, CO: SME.

Neuendorf, K.K.E., Mehl, J.P., Jr., and Jackson, J.A. (eds.) 2005. *Glossary of Geology*, 5th ed. Alexandria, VA: American Geological Institute.

Popek, E.P. 2003. *Sampling and Analysis of Environmental Chemical Pollutants: A Complete Guide*. New York: Academic Press.

Schmiermund, R.L., and Drozd, M.A., eds. 1997. Acid mine drainage and other mining influenced waters (MIW). In *Mining Environmental Handbook*. Edited by J.J. Marcus. London: Imperial College Press. pp. 599–617.

Schreuder, H.T., Ernst, R., and Ramirez-Maldonado, H. 2004. *Statistical Techniques for Sampling and Monitoring Natural Resources*. General Technical Report-GTR-126. Fort Collins, CO: U.S. Department of Agriculture, Rocky Mountain Research Station. www.fs.fed.us/rm/pubs/rmrs_gtr126.pdf. Accessed 8/23/13.

Skousen, J., Rose, A., Geidel, G., Foreman, J., Evans, R., Hellier, W., and members of the Avoidance and Remediation Working Group of the Acid Drainage Technology Initiative. 1998. *A Handbook of Technologies for Avoidance and Remediation of Acid Mine Drainage*. Morgantown, WV: West Virginia University, National Mine Land Reclamation Center, http://anr.ext.wvu.edu/r/download/45301. Accessed 8/23/13.

TAEM and SENES (Terrestrial and Aquatic Environmental Managers, Ltd., and SENES Consultants Ltd.). 1997. *Guideline Document for Monitoring Acid Mine Drainage*. MEND Project 4.5.4. Ottawa, ON: Canada Centre for Mineral and Energy Technology.

Tremblay, G.A., and Hogan, C.M., eds. 2001a. *MEND Manual: Volume 2—Sampling and Analysis*. MEND Document 5.4.2b. Ottawa, ON: Natural Resources Canada. www.mend-nedem.org/reports/files/5.4.2b.pdf. Accessed 8/23/13.

Tremblay, G.A., and Hogan, C.M., eds. 2001b. *MEND Manual: Volume 6—Monitoring.* MEND document 5.4.2f. Ottawa, ON: Natural Resources Canada. www.mend-nedem.org/reports/files/5.4.2f.pdf. Accessed 8/23/13.

USBR (U.S. Bureau of Reclamation). 2001. *Water Measurement Manual–A Water Resources Technical Publication*. Washington, DC: U.S. Government Printing Office. www.usbr.gov/pmts/hydraulics_lab/pubs/manuals/WMM_3rd_2001.pdf. Accessed 8/23/13.

Wellmer, F.W. 1998. *Statistical Evaluations in Exploration for Mineral Deposits*. Berlin: Springer.

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CHAPTER 2

Sampling and Monitoring During the Mining Phases

Virginia T. McLemore, Kathleen S. Smith, and Carol C. Russell

2.1 INTRODUCTION

The mine life cycle can be divided into four main stages: exploration, mine development, operations, and closure/postclosure (Figure 2.1). The introduction handbook to this series (McLemore 2008) briefly describes the mine life cycle; and the following sections briefly explain each stage of the mine life cycle and its relationship to sampling and monitoring for closure.

Figure 2.1 also summarizes the data and information needs during the mine life cycle. During the premining and exploration phases of mining, it is beneficial to address climatological data, a water balance (also called water budget), and habitat disruption. Additionally, samples could be collected to determine potential for contaminant release (including, but not limited to, acid generation), and the receiving environment and receptors could be characterized to determine baseline conditions and possible contaminant pathways. It is advisable that geological units likely to be exposed during mining be fully characterized and evaluated for potential contaminant release.

2.2 EXPLORATION (PREMINING/UNDISTURBED)

The first stage of mining, the prospecting or exploration stage, can best be described as the search for a mineral deposit (Hartman 1992). A *mineral deposit* is any occurrence of a valuable commodity or mineral that is of sufficient size and grade (content) for potential economic development under past, present, or future favorable conditions. An *ore deposit* is a well-defined mineral deposit that has been tested and found to be of sufficient size, grade, and accessibility to be extracted and processed at a profit over a specific time. Most exploration programs do not evolve into an operating mine because the deposit size is too small, the grade is too low, or the environmental risks are too high to be profitable at a given time. Therefore, the economics include an assessment of the quality of the mineral deposit and potential environmental costs, which will dictate the need for sampling and monitoring at this stage (see Figure 2.2).

Mineral exploration can typically require 10 years or more of study before a decision is made to develop or drop a potential mineral deposit. Several feasibility studies can be conducted to determine if the deposit can be mined economically at a specific time. Companies constantly reevaluate formerly discovered projects as economics change with time. Exploration can include literature searches; geological mapping; drilling; geochemical characterization of rock, soil, water, or biological media; geophysical and remote sensing surveys and aerial photography; among other studies (most of these techniques are briefly described in Appendix 3). A tremendous amount of

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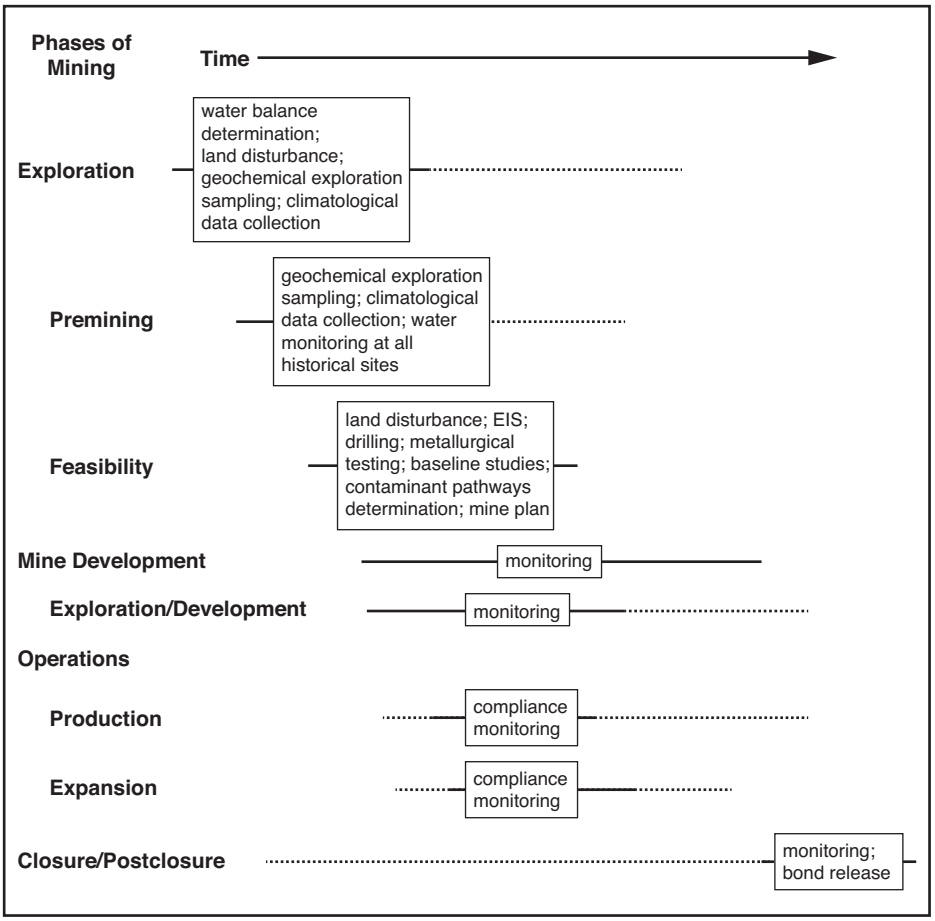


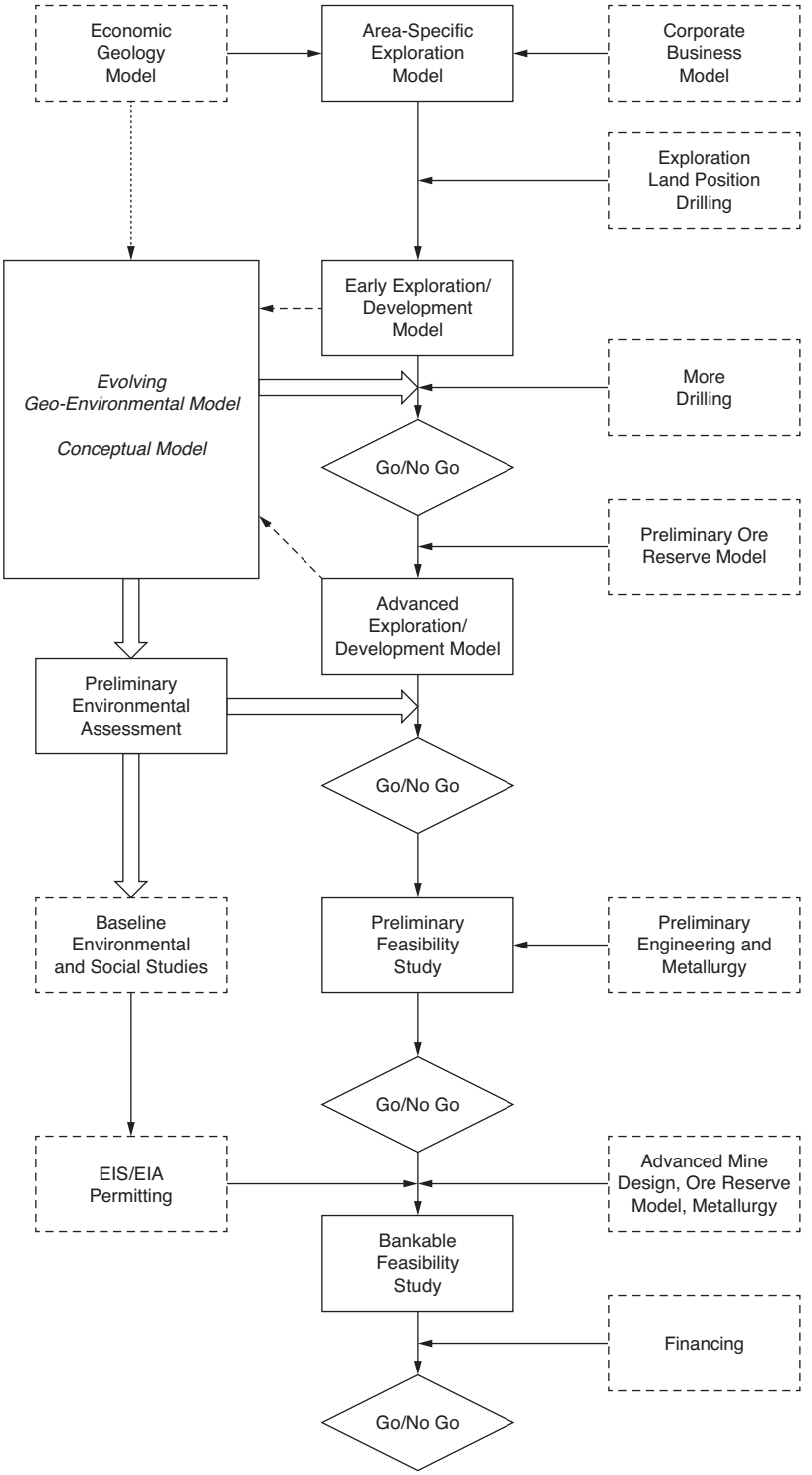
FIGURE 2.1 Data and information needs during the various phases of mining. Operations can include advance exploration and continuous planning for closure. Closure can occur at any stage.

data collected during exploration can be useful later in identifying, predicting, and mitigating mining influenced water (MIW).

Exploration has the potential to disturb the baseline conditions, and typically these disturbed areas need to be reclaimed. The environmental impacts of exploration can range from habitat disruption to surface-water and groundwater impacts from improper management of fuel spills. Exploration commonly requires trenching or drilling to retrieve rock samples for assay or other chemical analyses or metallurgical testing. Exploration roads, drill pads, and trenches have the potential to damage water and drainage quality from the erosion of sediment alone. These trenches and drill sites and their access roads should be sampled prior to disturbance and reclaimed after exploration, unless further development of the deposit is planned. Geochemical sampling, road construction, and drilling, which typically occur during the exploration stage, can continue into the mine development stage and throughout the mine life cycle.

Acidic drainage (AD) can result where outcrops of sulfide-bearing rock are disturbed. One common misconception is that ores near the surface contain no sulfide minerals because they have been oxidized or weathered. Whether sulfide minerals exist at the surface depends on local climate and its effects on factors such as

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Source: Adapted from Schmiermund and Ranville 2006.

FIGURE 2.2 Flowchart summarizing the major steps during project development and integration of geologic, social, economic, and environmental inputs in feasibility studies to determine if a project can proceed to the next stage in the mine life cycle

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ACID DRAINAGE FOUND DURING CONSTRUCTION OF TAILINGS DAM AT THE KENSINGTON MINE IN ALASKA RESULTS IN ADDITIONAL MONITORING

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The Kensington gold mine is approximately 72 km (45 miles) north-northwest of Juneau, Alaska, and began production in June 2010 by Coeur Alaska. The gold is hosted by multiple mesothermal, quartz-carbonate-pyrite swarms and veins within the Cretaceous Jualin diorite. The mine is expected to produce 108,000 to 114,000 ounces of gold in 2013 at a grade of 0.27 ounces/short ton using underground mining techniques. Kensington's proven and probable reserves were 1.0 million contained ounces of gold at year-end 2012 (Coeur Mining, Inc., n.d.). The Kensington deposit is in carbonate rocks, and, therefore, the waste rock piles and tailings piles are not net-acid generating, because the carbonate rock will neutralize any acid that is produced. However, during construction of the tailings dam, pyrite-bearing graphitic phyllite was exposed to air and generated acid drainage near the site (Golden 2008; Alaska Department of Environmental Conservation 2009). Modifications to the existing reclamation plan were required. Reclamation of the acid drainage was followed by plans requiring special handling of any excavated pyrite-bearing rock and rigorous sampling and monitoring plans. This example illustrates

- The importance of characterizing all geologic materials that will be encountered during construction;
- That despite all geologic characterization studies and mine plans, unexpected problems can occur during the construction and operation stages that could require changes to the original mine plan, including additional sampling and monitoring; and
- The quick response required to remediate the problem.

- Weathering rates,
- Mineral type and composition,
- Length of exposure to oxygen (time since uplift or exposure),
- Location of rocks above the local water table, and
- Quantity and rate of water flow through the rocks.

Generally, higher accessibility of sulfide minerals to moisture and oxygen promotes oxidation, which is why sulfide-bearing rocks tend to weather more slowly in dry climates compared with wet climates. Sulfides are not present in all mineral deposits, but the possibility of acid rock drainage (ARD) should be considered during both shallow and subsurface exploration. Any material with sulfide minerals should be sampled. Furthermore, country rocks that could be affected during construction of mine facilities should be sampled and characterized to determine if sulfides are present. For example, during construction of the tailings dam at the Kensington mine in Alaska, pyrite-bearing graphitic phyllite was exposed that generated ARD, even though the Kensington deposit is in carbonate rocks and therefore non-acid generating (see Acid Drainage sidebar).

In the past, prospectors typically panned in streams and sampled at the surface to identify potential areas for continued exploration. Once a likely area was identified, the prospector would typically dig pits, shafts, or adits to test the mineral occurrence and determine the grade and

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extent of the deposit. Commonly, the prospector proceeded directly into mining the deposit as long as minerals were visible. Many such formerly discovered prospect sites have AD because pyrite, the nearly ubiquitous indicator of most types of metallic ore bodies, was disturbed at the surface and broken into chunks that exposed more pyrite to oxygen and water. This exposed pyrite then weathers to form AD.

Traditional and modern exploration technologies, such as satellite and other remote-sensing imagery, geologic maps, geochemical and geophysical studies, and geologic reports, can be used in environmental studies (see summaries of these techniques in Appendix 3). Traditional exploration samples from stripping, drilling, trenching, bulk sampling, and even panning can provide useful environmental insights into potential water- and air-quality problems. Extra chemical analyses of sediment and soil samples and biological materials collected during the exploration stage can be used for establishing baseline environmental monitoring data. Geophysical techniques, which explorationists use routinely to predict the depth and breadth of mineral deposits, can be used to characterize the probable extent of natural AD sources prior to mining (see summaries of various techniques in Appendix 3). Geologic maps, the foundation of mineral exploration and mining, can provide fundamental information useful in interpreting baseline environmental implications of geochemical analyses.

Environmental evaluations in mining are useful not just for site characterization, but for determining if a deposit is economically feasible. The economic feasibility determination must consider baseline or ambient conditions and the costs of closure. Where mineral deposits associated with sulfide minerals occur near the surface, it is likely that current soil productivity, natural drainage quality, and biological conditions are affected or impaired by minerals associated with that deposit. Furthermore, these unique environmental conditions can support rare or endangered species that have colonized these unusual habitats. Defining these conditions before mining begins can be critical in evaluating the economic and environmental liabilities of mine development. See the Objectives of Feasibility Studies sidebar.

Another economic assessment consideration includes the proximity to wilderness areas, national parks, wildlife refuges, recreation sites, or other valuable or fragile environments. The presence of threatened or endangered species, or their habitats, can make or break a project. Knowing these features in advance of or during exploration commonly determines the "go or no-go" decision. Some of these features should be sampled if the project moves forward. A significant gold deposit in Montana near the border of Yellowstone National Park drew unprecedented opposition from citizens concerned about how mining might affect the park. Their opposition was perhaps of key importance in the company's decision to abandon its plans for this deposit (Russell 2006; see New World Mining sidebar). Similar public opposition to the Pebble mine in Alaska, which is located in a major salmon habitat, led Anglo American to pull out as a partner in the venture.

Regulations governing exploration programs vary from jurisdiction to jurisdiction. Some exploration projects are so extensive that operational and reclamation plans must be developed prior to drilling. Permitting is required for exploration in most areas in the United States and elsewhere, and reclamation of disturbances caused by exploration is also required in most areas.

2.2.1 Sampling and Monitoring During the Exploration Stage

Information and data collected during exploration can be applied to later stages of the mine life cycle. By slightly expanding data collection during this stage, exploration sampling and monitoring becomes essential for the foundation of later mining stages, especially mine planning, development, and closure. As the first to set foot on potential mine sites, exploration geologists

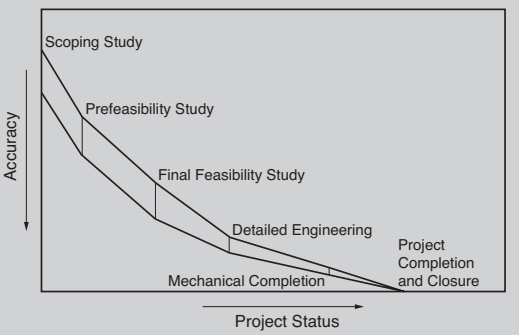
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OBJECTIVES OF PREFEASIBILITY AND FEASIBILITY STUDIES

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A *mine prefeasibility study* or a *preliminary feasibility study* is a preliminary comprehensive study of the viability of a mining project where a mining method and effective method of mineral processing has been established (CIM 2005). A mine feasibility study is a more detailed evaluation of a mine project to determine if that project has the likelihood of becoming an economic, operating mine. Feasibility studies are generally performed during exploration and mine development but can also be performed during any stage of the mine life cycle. Besides determining the location, accessibility, geology, climate, topography, resources and reserves, processing, transportation, market and price expectations, and infrastructure, various risks should be identified and evaluated, including if the property is under company control or outside company control, is the community generally favorable for the mining operation, what are the environmental risks, political climate, and so on. Guidelines for prefeasibility and feasibility studies can be found in the Canadian National Instrument (NI) 43-101 requirements or the Australasian Code (JORC 2012).

Different types of feasibility studies are required during the exploration stage to determine if the project could be an economic mine and determine whether more money should be spent on the project. Typically, several feasibility studies are required and can have different names, such as scoping studies (order of magnitude), prefeasibility studies, final feasibility study, detailed feasibility study, and so forth, depending on company policy. Most exploration projects do not become operating mines, because the feasibility study at a specific time determined that the project would not be economic at that time. Additional information, change in company, or change in economic conditions can result in a decision change and new feasibility studies are generally required. The accuracy of each feasibility study improves as the project becomes closer to becoming an operating mine (see figure), because more knowledge is obtained as the company spends more money. The early feasibility studies provide estimates of the likelihood of the property becoming an operating mine, and later feasibility studies will be more accurate. The results of the feasibility studies should be incorporated into the final mine plans.



Source: Adapted from McCarthy 2010.

Probable accuracy of each phase during an exploration project

have a chance to develop insights and fill many future data needs, provided they sample, analyze, and preserve data and information with a secondary objective toward the later mining stages and closure.

Explorationists analyze more ground than is mined and can produce a regional geochemical and environmental picture that can be applied to environmental planning and monitoring. For example, by noting areas prone to flooding, collapse, or landslides, explorationists can lead mine planners away from such dangerous or unstable features. Recent studies near Lake City, Colorado, show that head scarps of major landslides lie in hydrothermally altered rock that is unstable

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NEW WORLD MINING HALTED BECAUSE OF PROXIMITY TO YELLOWSTONE NATIONAL PARK, MONTANA

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The New World mine was proposed in 1990 in a previously mined area near Cooke City, Montana, just northeast of Yellowstone National Park. The controversy took on national and worldwide significance when in 1995, a delegation from the World Heritage Commission visited the park to investigate the proposed mine and placed Yellowstone on the list of World Heritage Sites in Danger. A *New York Times* reporter stated that "this [author] page is convinced that the proposed New World mine is a disaster-in-waiting that could ruin one of America's leading ecosystems" (Semple 1995). The mine plan was good and included procedures to clean up all of the abandoned mine wastes in the area. However, the end result was the purchase of the mine by the U.S. government with the U.S. Forest Service (USFS) taking on the responsibility of acid rock drainage and remediation of the previously mined areas. Remediation by the USFS is ongoing. See USFS Web site (www.maximtechnologies.com/newworld/index.htm) for more information.

because of rapid weathering caused by pyrite oxidation (Diehl and Schuster 1996). This alteration is related to nearby mineral deposits of gold and sulfide veins. Similar landslide scarps are found in other areas of extreme alteration, such as at the Questa molybdenum mine in New Mexico (Figure 2.3; Vincent 2008; McLemore 2009) and the upper Alamosa River Basin in Colorado (Bove 1994). Exploration work integrated with soil chemistry and physiographic analyses could be used to identify unstable areas to avoid during construction of waste rock piles and other mine facilities.

By recording observations of media other than rocks, such as plant life, soil characteristics, and water chemistry, the explorationists lend insights into native environmental conditions, and set the stage for monitoring programs and features that can minimize impacts to the environment. However, cursory visual observation that is incomplete or done by persons who are not properly trained can lead to skewed or incorrect interpretations and should be avoided. Hydrologic features, such as wetlands, seeps, and iron bogs, can indicate where MIW will surface over the mine life cycle. Subsidence and historical mining structures tend to affect how subsequent mines are planned and developed, and, because explorationists tend to discover such features through ordinary ground studies, their observations can be useful for mine planners.

Explorationists tend to concentrate on analyzing potentially valuable elements, such as gold, copper, and zinc, and analyses of *pathfinder elements* to assess large areas and focus on smaller drill targets. Even today, not all exploration geologists will use complete trace element analyses. If, in addition to these limited analyses, a broader suite of chemical parameters are analyzed, data thus produced can be useful for studies as diverse as geochemical modeling, weathering, and slope stability, if the data meet data quality objectives. Complete chemical analyses of water, soil, sediment, rock, and vegetation are commonly necessary to determine the background or baseline geochemistry and biochemistry of a site. If exploration and environmental sample results are integrated, there is a better chance of identifying problems that mining will encounter. For example, at the Kendall mine in Montana, thallium was found to be a problem long after mining had begun (W. McCollough, personal communication, June 2003). Early identification could have allowed incorporation of management practices to mitigate or minimize potential problems. The difference in cost between complete chemical analyses and limited analyses of only pathfinder elements can be insignificant compared with the potential consequences of inadequate documentation of

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Courtesy of Virginia T. McLemore.

FIGURE 2.3 Landslides and debris flows in the Questa alteration scar above the Questa molybdenum open pit mine, Taos County, New Mexico

premining conditions. However, it is important to realize that these up-front costs are significant to limited exploration budgets. Also, it is important to keep in mind that analytical limits of detection adequate for determining premining environmental conditions can be lower than those needed for exploration. Whenever possible, it is good practice to retain all data and samples, even if they give negative results for exploration purposes. Properly archived data and samples could be essential during reclamation and closure because they can provide the only premining information. Data needs to be archived and backed up continuously, and samples should be stored such that they can be readily retrieved for additional analysis. Even data from formerly discovered projects can be important in establishing baseline or premining conditions in other areas and aid to a better understanding of geological processes that lead to adverse MIW conditions (see Schmiermund et al. 2006).

Efforts should begin early in the mine life cycle to record climatic conditions in areas of exploration interest, because these data are required for defining a water balance for the mine site and regulatory purposes, including defining baseline and/or background conditions. Parameters of interest include

- Annual and seasonal precipitation rates and intensity,
- Daily temperature range,
- Relative humidity,
- Predominant wind direction,
- Wind speed, and
- Evaporation rates.

Seasonal data are necessary, and supplementary climatological data can be required to adequately characterize the site. Weather stations can seem like a waste of exploration dollars, especially the collection of that data, but the data thus developed serve as a basis for water balance calculations that will be needed throughout the mine life cycle. Moreover, on-site climatic data

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CLIMATE DATA AT THE SUMMITVILLE MINE, COLORADO

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Summitville is a classic example in the United States where the water balance and precipitation calculations were based on data from the nearest town, Alamosa, 75 km (46.6 miles) away and more than 2,000 m (6,562 ft) lower in elevation. Very cold temperatures were unanticipated when the liner for the cyanide heap leach was laid in place. Failure of the liner occurred almost immediately, releasing acid rock drainage and cyanide (Gray et al. 1994). "The polluted water was supposed to be contained in a heap leach pond, but the state's oversight of the operation was lax. Unusually heavy snow and runoff overwhelmed the pond's critical systems, sending cyanide-laced liquid into tributaries of the Rio Grande, killing all fish downstream for 17 miles [27 km]" (USEPA 2011a). In addition, no one expected to have snow depths of more than 8 m (26 ft) the first winter the government took over the site. The unknown effects of the water balance and weather are factors contributing to the mine's failure. See King (1995) for more information.

can be far more accurate, and thus more applicable for mine design, than data collected from the nearest weather monitoring stations, which can be distant in location or climatic conditions. For example, at Summitville, Colorado, where climatological data came from a site 75 km (47 mi) away, the site-wide hydrologic balance underestimated the precipitation, which led to critical shortcomings in the water balance and the site's water management needs (Pendleton et al. 1995). Many climatic stations can be required at some sites to adequately characterize different microclimates because of elevation and topographic changes. Such is the case at the Questa molybdenum mine, New Mexico, where elevations range from 2,377 to 3,048 m (7,800 to 10,000 ft), and slope aspects vary from north- to south-facing, which places some slopes directly in the sun where evaporation/sublimation is the greatest, while other slopes are in the shade where snowpack is present much later in the season (McLemore 2009). See the Climate Data sidebar.

Surface and subsurface rock samples can provide essential mining and environmental information, and closure/postclosure needs should be considered in the choice of collection methods. Surface-rock samples are collected with either manual tools or a backhoe. Rocks from below the surface can be collected with a variety of drilling methods, including churn, hammer, reverse circulation, and diamond drilling. The former three drilling methods produce rock chips or powders, whereas the diamond-drill method produces a continuous core of the rocks it traverses. Diamond-drill cores provide a more complete record of the original stratigraphy of overburden, waste rock, and mineralized rock than samples from rotary methods, and are considered the most useful for collection of detailed geologic data. Selected exploration drillholes can be converted to monitoring wells to provide samples for baseline, operational, and postclosure conditions or other monitoring needs.

Prefeasibility and feasibility studies are employed at various stages of the exploration project to determine if the project could result in an ore deposit that could be extracted economically at known and projected costs with a profit. Further details of types and uses of prefeasibility and feasibility studies and evaluating risk are presented by Lord et al. (2001), Dowd (2002), Schmiermund and Ranville (2006), and others who have described the process, which is summarized in Figure 2.2. Part of any prefeasibility or feasibility study should be the identification of geologic conditions that could impact surface water and groundwater. One such method is the U.S. Geological Survey's (USGS's) geo-environmental models (GEMs) (Plumlee and Nash 1995); some examples of their use are summarized in Appendix 6. See the GEMs sidebar.

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GEO-ENVIRONMENTAL MODELS OR GEMS

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Geo-environmental models (GEMs) provide one method of developing site conceptual models and identifying potential water quality impacts based on characteristics of the mineral deposit. A GEM is a compilation of geologic, geochemical, geophysical, hydrologic, and engineering information pertaining to environmental behavior of geologically similar mineral deposits prior to mining and resulting from mining, mineral processing, smelting, and refining (Plumlee and Nash 1995). Geologic data are compiled, including deposit type, related deposit types, deposit size, host rocks, surrounding geologic terrain, 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 (see Appendix 6; Plumlee and Nash 1995). Thus, sites can be initially assessed by determining site characteristics and comparing them with previously determined relationships between similar characteristics and their potential for adverse impacts on water quality. GEMs have more applications in the early stages of the mine life cycle.

2.3 MINE DEVELOPMENT

Once an economic mineral deposit is discovered, its limits defined, and the feasibility studies indicate that an economic deposit exists, then permits must be obtained so that mining can proceed to the second stage of the mine life cycle, mine development. Baseline environmental conditions characterized during the exploration stage should be extended into the development phase in greater detail and any information gaps addressed during the earliest part of development, prior to ground disturbances. This is because disturbances during development and mining can typically be environmentally indistinguishable from naturally degraded conditions in near-surface mineral deposits. It is important to incorporate appropriate natural conditions into the site plan to set realistic reclamation goals. Permitting, operational, and preliminary mine-closure plans are generally developed at this stage. Mine development of sites typically requires additional infill drilling to refine the three-dimensional features of the deposit to sufficiently develop plans for excavating the deposit. This phase, which is commonly conducted by a contractor, consultant, or a junior mining company, also includes construction of mine access and mine-haul roads, rail lines, and terminals (if they are required); installation of electrical, gas, and other utilities; and construction of mine and process buildings, support structures, and, in some areas, housing facilities. In remote or undeveloped areas, facilities construction can include housing and social infrastructure, such as schools, churches, recreation sites, and shopping centers. Excavation requires heavy machinery and the use of petroleum and other industrial products for operation and maintenance. If the mine site is not pristine prior to exploration, chemical scans can be useful to establish premining baseline conditions. All excavated sites should be examined and characterized to determine if any acid-generating material was encountered and to determine long-term stability of slopes.

Before engineered mine-waste and process-waste repositories, roads, buildings, or other structures are constructed on a mine site, condemnation drilling should occur; that is, closely spaced drillholes are bored to ascertain that viable minerals and historical mine workings do not lie beneath the proposed structure. Information collected through such drilling can be applied to geochemical and hydrological balance studies.

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The mine development stage can also include construction of metallurgical processing facilities, such as mills, pipelines, conveyance facilities, engineered tailings impoundments, containment facility liners, leach pads, and solution collection facilities. Metallurgical testing and development are locally conducted on-site at metallurgical pilot plants. For operations requiring cyanide or acid leaching, mills, leach pads, processing ponds, and their related infrastructure, such as cyanide detoxification facilities, are constructed during the development stage, and leach pad construction can continue into the operational stage, especially at large, multiyear operations.

Metallurgical testing provides critical data for evaluation of ore and has environmental applications at mining and processing sites. Metallurgical testing is conducted prior to the mine development stage but is generally at a smaller scale that provides data for preliminary economic assessments or feasibility studies. Depending on the type of processes ultimately selected, metallurgical testing will generate ore composites, leach residues, process solutions, or tailings that can be made available for geochemical testing to evaluate the potential for ore, leach residue, and tailings to generate MIW/ARD in the pit walls and stockpiles, on leach pads, and in tailings storage facilities. Metallurgical testing solutions provide important information on the concentration of metals and anions in process solution, which is particularly important for process facilities with a positive water balance requiring excess water to be treated and discharged. Also during this metallurgical testing, contaminants arising from the solutions and slurries that come from mineral processing should be evaluated to determine if they contain elevated levels of contaminants. For example, some of these slurries could have elevated levels of arsenic that are released during mineral processing. Metallurgical testing produces assays and geochemical analyses similar to many that are run during exploration, such as detailed multi-element analyses. These analyses describe the rocks and minerals at a very detailed mineralogical and physical scale to enable effective recovery of the valuable commodity. During this phase of testing, trace-element composition of minerals is examined, and from the environmental perspective, this testing can be important for understanding of site-wide geochemical characteristics that can be useful in ore and waste rock management. For instance, cadmium is commonly present as a trace element in sphalerite (ZnS), but sphalerite in some deposits is not of sufficient quantity to warrant recovery during milling. In such cases, if waste leaching or weathering testing shows that the material containing sphalerite could cause an environmental impact, the waste storage facility could be engineered so that it could be specially handled by placing it in a specific, more isolated area of the waste rock facility instead of with other gangue minerals (noneconomic minerals, such as quartz or calcite that are part of an ore deposit) to avoid release of cadmium. Additional environmental protections can also be put in place to minimize the distribution of cadmium and zinc into the environment. Such detailed mineralogical observations are typically made during metallurgical testing. See the Why Mineralogy Is Important sidebar.

Material strength properties, which are typically determined during mine development, can provide useful environmental and closure information. Whereas mining engineers examine rocks to determine the maximum height or depth of mine benches, the rock properties can also be used to predict the conditions of potential failure of mine features during operations and after closure. For example, if a mine pit fills with water after mine closure, pit walls can become unstable. Slope failures after mining can be as much a problem for mining engineers as are slope failures during mining. Areas of potential collapse or subsidence can be indicated as well, because these collapses can affect water quality, containment, and water flow. In areas of historical mining, past underground workings that are accessible need to be mapped and characterized for potential stability and drainage issues.

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WHY MINERALOGY IS IMPORTANT IN ACID MINE DRAINAGE STUDIES

Stephen J. Sutley, U.S. Geological Survey

A remedial investigation is being undertaken at Holden mine in the northern Cascade Range in north-central Washington under the supervision of the U.S. Forest Service, the U.S. Environmental Protection Agency, and the Washington State Department of Ecology to determine the presence, magnitude, nature, and extent of potential environmental concerns associated with this historical mining endeavor. Between 1938 and 1957, more than 10 million short tons of copper, zinc, silver, and gold ore were extracted from this deposit.

Mineralogical studies, mostly involving X-ray diffraction techniques, have proven critical in the characterization and interpretation of prevailing site conditions. Some of the major issues addressed and resolved by mineralogical studies include (1) verification of the primary sulfide minerals (pyrite, pyrrhotite, chalcopyrite, etc.) ultimately responsible for the site's acidic, metalliferous discharge; (2) identification of various soluble secondary sulfate minerals (including iron, copper, and zinc phases) known to have a major impact on the seasonal variations in the site's water chemistry (as a result of the soluble sulfates cyclic dissolution and hydraulic flushing); and (3) determination of the mineral residences for metals detected in the site's surface and subsurface soils (permitting an estimation of the bioavailability of metal toxicants).

These, and other studies, will enable government agencies, consultants, and other concerned groups to evaluate the effectiveness of proposed remedial measures and determine the most practicable treatment alternatives as they apply to the Holden mine cleanup.

2.3.1 Sampling and Monitoring During the Mine Development Stage

Mine development is the transition stage between exploration and mine operations. Hence, many issues considered during exploration also need to be addressed during this stage of mining. If collection of on-site climatological data did not begin during exploration, it is essential that this information be collected early in the mine development stage, even if the local regulation does not require them. In order to establish successful revegetation at the time of mine closure, observations of native vegetation, soil depth, slope and aspect, and other preexisting conditions must be documented. This information is also useful during the operations stage for planning placement of waste materials; it is more economical to move waste only once. During the early stages of development, it is important to consider reclamation. An example of this practice is when Nevada Placer Dome hired the Duckwater/Shoshone tribe to collect seeds and grow plants needed for revegetation (B. Upton, personal communication, September 2003).

During the mine development stage, metallurgists evaluate detailed mineralogy. Concurrently, others are developing a water balance for the site and defining baseline environmental conditions. It can be extremely useful at this stage to set aside an area for site weathering tests and initiate predictive tests, such as acid–base accounting, meteoric water mobility, static and kinetic tests, and other long-term weathering tests, conducted under natural local conditions. Some of these tests could begin earlier in the mine life cycle. These data are useful in the prediction of acid-generation and water management requirements at the site. For example, the acid-generation characteristics of the waste rock at the Zortman–Landusky mine in Montana changed over time (Miller and Hertel 1997) because of the buildup of ferric iron, a strong oxidant, in the sulfidic waste-rock piles and the heap pile. This unanticipated change in mineralogy contributed to the

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eventual bankruptcy of the parent company (Russell 2006). Air monitoring can also begin during this stage, or earlier, to develop baseline conditions prior to ground disturbance.

Permitting, which generally takes place during the mine development stage, can require significant environmental data to accommodate the permitting process. The mine development stage is a good time to design and implement compliance-monitoring programs. Lysimeters can be installed at this time to measure depth to water and to aid in evaluating infiltration rates. Pilot studies, such as test heap-leach pads and site waste-rock weathering columns or test pads can be constructed at this time, and monitoring can begin. It is good practice to continue these site weathering and closure pilot studies throughout the remainder of the mine life cycle to provide information for closure. For example, when research on pilot heap closures was undertaken at the Golden Sunlight mine in Montana with the U.S. Environmental Protection Agency's (USEPA's) Office of Research and Development and MSE Technology Applications, Inc., the operator (Placer Dome) found pilot-testing results to be very useful and is now undertaking similar tests at their operations in Nevada (B. Upton, personal communication, September 2003).

Transport and pathways of waters and potential contaminants need to be identified during this stage. Detailed geological maps, including structural information, should be refined and updated. Potential biological receptors can be evaluated and indicator organisms established.

Disturbance from road building and other structures should be sampled and characterized for use during closure. It is important to keep in mind that closure can occur any time during mine development if feasibility studies determine that continued development or mining cannot be economical. If closure occurs, all disturbed ground will have to be reclaimed.

2.4 OPERATIONS

The operations stage of mining includes the process whereby ores and waste rock are removed from the ground, milled and/or leached, and concentrated into a salable product, such as a metal sulfide concentrate for smelting or high-grade gold or silver bullion for refining. Sampling of the ore and waste materials is necessary in order to know what potential contaminants exist in the rock mass, and monitoring of groundwater or surface water can be necessary for environmental compliance. Air pollution monitoring also could be required. Excess water produced by the operations may require sampling during treatment and discharge. Much of this excess water is reused elsewhere in the mine and process operations.

2.4.1 Excavation

The initial and usually ongoing phase of the operations stage is excavation, which includes the extraction of ores and waste rock by drilling, blasting, and mucking (removing the broken material from the mine). During excavation, as rock is removed from the ground, excess water may need to be pumped or drained from the area of underground and surface workings in order to accommodate miners and equipment; sampling of this water is important for closure. At open pit mines requiring dewatering, water is typically pumped from wells adjacent to the pit, a process that lowers the water table below the bottom of the pit, and from sumps within the pit, if necessary. In underground mines, excess water is usually pumped from sumps that are constructed in the lower reaches of the excavation and piped upward through vertical mine shafts to the surface. At many older underground mines located in steep terrain, such as in Colorado, underground mining was made possible through construction of an adit (horizontal shaft) or tunnel that drained groundwater from one or more mines to a nearby stream. Such drainage tunnels, which were constructed several kilometers (miles) underground, could dewater many square kilometers (miles) of surrounding rock and workings, and thereby reduced the need for expensive pumping

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of water from mine workings. However, drainage-tunnel water is commonly of poor quality and could require treatment. Today, much of this water is recycled and used in operations.

Whenever the water table is lowered to allow mining below it or when it fluctuates (such as occurs seasonally or during storm events), there is a chance that water quality will degrade because of oxidation of sulfide minerals. When this degradation happens, acid water or any MIW can form wherever there are sulfide minerals that had resided below the water table. Monitoring these waters during mining, both inside and outside the mine permit area, will be important in planning for mine closure. Dewatering that occurs during the operation stage may have to continue after excavation ceases in order to control environmental impacts. If a mine floods, resulting MIW can require treatment or other special management. See volume 3 of this series (Castendyk and Eary 2009) for more details on chemistry and dynamics of pit lakes at mine sites.

2.4.2 Milling, Concentrating, and Leaching

After ores are excavated from the ground, they are generally placed in engineered rock piles (i.e., dumps) or reduced to a nominal size to allow ore minerals or leachable metals to be segregated from waste rock or gangue minerals or to promote rapid leaching. Crushing and milling generate a rock mass with a surface area that is many times greater than the original rock mass. Once exposed to air, finer-grained crushed and milled rock can generate acid if it contains acid-generating sulfide minerals, such as pyrite. Sulfide ores are typically milled to a fine-grain size (generally <100 μm, although this varies). This allows particles of ore minerals to be segregated from gangue minerals, or from other ore minerals, via flotation, gravity, electromagnetic methods, or other processing means, or they may be directly leached. Other types of ores, including many gold and copper-oxide ores, do not require further size reduction (dump leaching), or they need only be reduced to larger chunks (typically 2.5–12.7 cm [1–5 in.]) so they can be heap leached after being placed in a lined facility and treated with a leaching solution to remove the economic metals. Dump-leach materials are processed with no further size reduction. Size reduction is also used to promote rapid leaching. Some gold ores are milled to a fine-grain size to expose gold-bearing minerals to leaching solutions or roasted/autoclaved to oxidize the carbon or sulfidic minerals. Tailings are finely ground waste from milling followed by metal extraction and are managed by dewatering and storage, primarily in lined facilities.

Mineral separation techniques have changed over time, and the sampler must be aware of historical and modern technologies, because different technologies can lead to potentially different environmental consequences (W.R. Jones 2007). For example, stamp mills were commonly used at gold mines during the late 1800s and early 1900s. These mills ran gold ore through a number of levels, with cam-driven hammers pounding the ore into finer and finer particles. Gold was separated from the finest particles using mercury amalgam. Waste materials from stamp mills commonly contain fine-grained sulfide minerals that can result in acidic metal-rich drainage, and mercury contamination can be found in the vicinity of some historical stamp mills.

Leaching is a common technique in which valuable, leachable metals are removed from the gangue, typically along with other less desirable metals. The metals are then extracted from the high-metal concentration pregnant solution by other hydrometallurgical processes (such as electrowinning, zinc cementation, and carbon adsorption). Leaching chemicals, such as cyanide and sulfuric acid, can be potential environmental contaminants. See Uravan Mill sidebar.

2.4.3 Smelting and Refining

Although not always the case, metal smelters and refineries today are large, specialized facilities that are not profitable enough to operate at most individual mine sites. Consequently, many are located in areas away from mining districts and service multiple mines; many of these areas are in

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URAVAN MILL, COLORADO

Carol C. Russell, U.S. Environmental Protection Agency

The 1.8-km^2 (450-acre) Uravan uranium mill began as a radium-recovery plant in 1915. Its owners later converted it for vanadium extraction. From the 1940s to 1984, the plant utilized sulfuric acid in the uranium and vanadium processing facility, thus the origin of the town's name, Uravan. Operations at the site left a large volume of acidic and alkaline wastes, contaminating air, soil, and groundwater near the plant and the San Miguel River. Contaminants included radioactive products such as raffinates, raffinate crystals, and mill tailings containing uranium and radium. Other chemicals in the tailings and groundwater were heavy metals such as lead, arsenic, cadmium, and vanadium. The most difficult aspect of remediation of this mining waste and acid rock drainage site was that the town of Uravan was in the middle of the site. The final remediation solution required moving everyone, and all buildings, from the town permanently (USEPA 2011b).

larger cities and towns. Smelting, which involves heating in the presence of flux, is employed to melt the mineral concentrates and segregate metals from their combined elements (e.g., oxygen or sulfur). Refining is a more advanced process to segregate one metal from another and produce a higher-grade, refined metal or metallic compound that can be sold for use in metal applications. Refining of the recovered metal can involve pyrometallurgical, chemical, and electrochemical processes that must be properly managed to prevent negative environmental impacts from wastes or emissions. Smelting and refining typically produces a waste slag (mostly melted minerals and other materials that form a rough, glass-like substance when cooled) that can require special handling. These processes also produce flue gases or smoke-stack emissions that need to be sampled and monitored. Sulfur dioxide, mercury vapor, and smelter dusts can be produced, which should be properly managed to prevent air- and drainage-quality impacts. Sulfuric acid is commonly produced and sold as a by-product of sulfide smelting and roaster operations.

2.4.4 Mine Expansion

Many mines undergo one or more expansions during their operating life as economic conditions change because of factors such as changes in metal prices, development of new technologies, and discovery of new ore reserves. The challenge is to keep the existing mine operating efficiently while the permitting process to expand mining moves forward. Ultimately, the ease in permitting expansions can depend on the ownership of land, past compliance record, attitudes of the general public, and the policies of regulating agencies.

2.4.5 Standby/Inactive Mines

Many mines will be forced to temporarily cease production for a variety of reasons, mostly related to economic conditions. Monitoring is highly recommended during temporary closure. At this time, few people are on-site to visually monitor potential problems, and the local community generally is not as focused on the mining operation. The possibility of oversight by regulatory agencies increases because this can be the mine's time of highest vulnerability. Mine sites can be inactive for many years, depending on government regulations and economic conditions. It is important that funding for continuation of sampling and monitoring is maintained during these standby periods. Another situation that can occur is when environmental targets become more restrictive under the new permit, and mine operators have to be ready with analytical results to show that all past waters and wastes will meet the new permit levels.

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2.4.6 Sampling and Monitoring During the Operations Stage

The operational units of mining and metallurgical processing can include the mine, overburden and waste-rock storage facilities, development waste materials, ore stockpiles, tailings, crushing facilities, concentration facilities, leaching facilities, tailings storage, and other operational facilities. Each of these units can require environmental monitoring, depending on their potential contaminants, process requirements, and their handling and closure characteristics. Potential environmental effects of mineral production include the following:

- Impacts to wildlife and fisheries
- Habitat loss
- Changes in local water balance
- Sedimentation
- Introduction of potential contaminants in tailings storage facilities or leaching-solution impoundments
- Formation of potentially unstable dams and highwalls
- Construction of tailings ponds or leaching pads and ponds
- Potential acid generation from ore
- Waste rock
- Pit walls and tailings
- Metal leaching by acid, neutral, and alkaline drainage
- Cyanide-solution contamination at gold and silver metallurgical recovery operations
- Wind-borne dust
- Gas and dust emissions from metallurgical operations
- Introduction of contaminants from blasting operations

Most of these impacts are routinely addressed or resolved as part of the facilities design during development and production stages.

An important aspect of the operations stage is compliance monitoring. One key to compliance monitoring is to have complete chemical analyses with analytical detection limits and other QA/QC (quality assurance/quality control) documentation. Complete analytical data can be used to verify predictions made during exploration and mine development, and can provide a basis for modifying plans during the operations stage (Kuipers et al. 2005, 2006). For example, selective handling of ore and waste material can become necessary during the operations stage to manage pyrite-bearing material and prevent or minimize acid mine drainage. Modeling of groundwater flow can require chemical data that were obtained during the exploration or mine development stages. Similarly, chemical analyses of bench samples of blastholes during open pit production can be useful in characterizing ore stockpiles and mine waste facilities for operation control as well as for reclamation. It is good practice to set site-specific threshold levels that trigger additional sampling or monitoring activities to ensure that compliance levels are not exceeded. Although many mines are not required to monitor nitrogen compounds or organics in water, it is good practice to include ammonia and nitrate analysis to monitor the effects of blasting, and to include TCE (trichloroethylene) analyses to monitor residues from degreasing of equipment in shop areas.

Sampling and monitoring during operations is also done to meet objectives during closure. Closure plans should be continuously reviewed, and results of sampling and monitoring should

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be compared to what was predicted for closure and appropriate changes should be made to the closure plan.

It can be important to characterize all materials, including backfill materials and dewatering fluids, using chemical, physical, and mineralogical techniques, and to maintain detailed records of sampling and analyses. At the Pinto Valley mine in Arizona, a tailings impoundment failed when waste rock was placed on tailings (Kiefer 1998). Supplementary geotechnical characterization and monitoring of the physical properties of the tailings and impoundment could have prevented this failure.

The water balance determined during the mine development stage needs to be continuously updated during the operations stage as new data become available (O'Kane and Waters 2003; Marcoline et al. 2003). USEPA found that a common thread among past Comprehensive Environmental Response, Compensation, and Liability Act (commonly called Superfund) mine sites is that they did not have an accurate water balance (Russell 2006).

Expansions and temporary closures can occur during mine operation. Frequent reexamination of the sampling and monitoring programs and revision of sampling plans are ways to lower the impacts of these circumstances. It is good practice to maintain monitoring programs during temporary mine closures and to maintain adequate staff and funding to be able to address unforeseen problems.

2.5 CLOSURE/POSTCLOSURE

Today, numerous mines and metallurgical facilities in many countries have preliminary mine-closure plans in place prior to development or operations. It is recommended that closure plans begin at the start of mining, if not during the exploration phase (H. Jones 2011), and that plans are updated on a regular basis. *Closure* generally is the period following excavation and operations during which activities must be conducted to complete mineral extraction and return lands to their intended postmining use. For some portions of the exploration and operation, closure can be done immediately. For example, a waste rock pile or heap leach pad that is no longer being used can be closed. During exploration activities, drill pads and excavation pits can be closed. Wherever possible, closure activities should be planned concurrently with exploration and operations. This overcomes the cost of moving material more than once and is, thereby, cost-effective because operations handle some of the closure costs, which can also offer other economic benefits, such as taxation, in some jurisdictions. Closure plans usually require that mine and process structures be removed, land be graded and revegetated, openings be sealed or safeguarded, and lands be generally returned to a beneficial use other than mining. *Postclosure* is that period during which local environmental equilibration takes place (e.g., groundwater and surface-water conditions return to steady state and vegetation reestablishes). During both the closure and postclosure stages, monitoring can be required, and continuing monitoring after closure is proactive.

Closure can involve recontouring of pit walls and waste-rock storage facilities, covering of reactive tailings and waste-rock storage facilities, decommissioning of roads, dismantling of buildings, re-seeding/planting of disturbed areas, ongoing monitoring, and possible water quality treatment and discharge. Geomorphological stability in the long term is a prime objective of postclosure. For example, Table 2.1 summarizes the differences between natural hills and artificial rock piles and tailings (H. Jones 2011). Some of the potential problems include seepage of contaminated solutions into groundwater and surface water from MIW, wildlife and fisheries habitat loss, re-vegetation failure, wind-borne dust, and slope and tailings impoundment failure. Plugging of shafts and adits needs to be carefully evaluated because plugging can cause changes in the groundwater flow and possibly contribute to contamination of the groundwater. Closure

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TABLE 2.1 Comparison between natural hills and waste rock piles/process tailings

Element	Mine Waste-Rock Piles and Process Tailings	Natural Hills
Construction material	Loose, uncompacted run-of-mine waste or run-of-mill waste	Bedrock core with weathered and or eroded regolith
Top surface	Varying size, large to small; surface typically bladed flat	Varying size, large to small; domed
Slope angle	Uniform, typically at angle of repose	Concave to convex, typically less than angle of repose
Slope configuration	Large rock piles and tailings constructed with regular steps; smaller rock piles, single slopes	Smooth
Drainage	Poorly drained	Well-developed, clearly defined drainages
Vegetation	Uniform age, introduced by the company	Mature and varied for climate and topography

Source: Adapted from H. Jones 2011.

plans typically establish a postmining use of the mine site, except for uranium mill tailings. Some sites may require long-term sampling and monitoring, whereas other sites will require no monitoring. Uranium mill tailings in the United States are controlled by specific federal regulations that provide for 1,000 years of monitoring and control by the federal government; these areas will not be released for future use of any kind. Biological media and water are the major media sampled during the final stages of the mine. Mine closure plan updating should be considered in all phases of mining.

2.5.1 Sampling and Monitoring During the Closure/Postclosure Stage

Closure plans typically establish a postmining use of the mine site commensurate with the desires of the landowner and adjacent local land uses. Some sites can require long-term maintenance and monitoring. Important factors in the closure/postclosure stage are to maintain monitoring and not to discontinue it too soon. For example, Placer Dome plans to monitor Las Cristinas mine in Venezuela for potential ARD for at least 20 years after mine closure because there are extensive underground and surface pathways for ARD, and any problems would have to be mitigated quickly (Dahlberg 1999). Problematic conditions can arise during, or years after, closure. At the San Luis mine in southern Colorado, a metal-rich seep developed adjacent to a backfilled pit during closure. Even though operations had ceased, there were personnel and equipment on-site to handle the problem, and mitigation was able to begin quickly. Continued monitoring can lend insight into source-control possibilities in lieu of ongoing treatment of MIW.

Continued characterization of solid materials moved during contouring and reclamation can avert unexpected results during closure. Also, predictive tests need to be reevaluated to assess their continued reliability. Some postmining uses, such as property sales or sale of non-acidic waste rock for construction purposes, could generate income for continued monitoring of the site.

2.6 HISTORICAL, INACTIVE, AND ABANDONED MINES

Historical, inactive, and abandoned mines, also known as legacy mines, offer the greatest challenge to the mining and environmental industries. These mines are historical, typically old, have not been mined for many years, and some of them do not have an owner and/or the original miner is unknown (i.e., true abandoned mines). Information on these sites, in many cases, is limited or

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nonexistent, and many would not comply with today's high mining standards of operation. Many of more than 500,000 legacy mine sites in the western United States pose no safety or environmental risk, but some of the mines could be challenging to remediate.

Historical miners had different operational philosophies than do miners of today. These miners had to make a profit quickly; often they would selectively mine only the high-grade ore (i.e., mine the high-grade ore without taking the lower-grade material). Exploration methods commonly involved construction of pits, shafts, and/or adits that have not been utilized in production of any ore. Historical miners typically located ore processing facilities (mills, tailings, stockpiles, etc.) near the production or haul shaft. Processing spoils were dumped near the processing unit, in many places into arroyos, streams, rivers, lakes, or other drainages. There were no laws or other guidelines available to prevent contamination of nearby drainages. Monitoring programs were nonexistent; sampling was conducted only to determine zones that could be economically mined. Metal mines were developed by underground and surface mining methods that were as inexpensive as possible, and safety and stability were not top priorities. Historical mines had as few ventilation shafts as possible in order to save money, resulting in poor air circulation. As little groundwater as possible was pumped from the mine. Mine surveying and mapping was minimal to nonexistent. Upon depletion of the ore, many mines were abandoned; mine waste piles and tailings piles were not engineered and were left as they were created without caps or vegetative cover. Surface and excess equipment was typically sold for scrap. It is important to understand the exploration, development, and production history of any mine before attempting to reclaim these sites (see case studies in Appendix 5).

Drainage quality is adversely affected in many past mining districts, but because there are typically no baseline data on conditions prior to mining, the cause and extent of the drainage quality is suspect. It is difficult to determine natural drainage conditions from those caused by mining. Studies determining background conditions in mining districts by USGS, such as the Questa and Red River districts in New Mexico (Nordstrom 2007) and Animas Basin in Colorado (Church et al. 2007b, 2007c), can be used as analog examples (see Appendix 5). The use of GEMs can provide some general information on basic deposit types (Appendix 6). What are considered poor mining practices today, such as dumping mine wastes into drainages, were considered a common, economic mining practice in the past, and these sites can still affect drainage quality.

One of the most difficult tasks in remediating historical mine sites is determining and characterizing the baseline conditions or natural background that existed prior to mining (Nordstrom 2007; Church et al. 2007a). Baseline conditions are necessary to establish meaningful remediation goals and monitoring programs. Methods used for this evaluation include the following:

- Integration of historical information
- GEMs
- Data from unmined, mineralized areas
- Analyses of water from monitoring wells (Figures 2.4 and 2.5)
- Leaching studies
- Statistical analyses
- Isotopic studies
- Identification of background by subtracting mining influences
- Computer modeling

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Courtesy of Virginia T. McLemore.

FIGURE 2.4 Monitoring well near the Copper Flat pit lake (porphyry copper deposit), Hillsboro District, New Mexico

2.7 SUMMARY

The mine life cycle can be divided into four main stages: exploration, mine development, operations, and closure/postclosure (Figure 2.1). Information and data collected during exploration can be applied to later stages of the mine life cycle. More ground is sampled during the exploration stage than is mined and can produce a regional geochemical and environmental picture that can be applied to environmental planning and monitoring. Permitting and mining can proceed to the second stage of the mine life cycle, mine development, once an economic mineral deposit is discovered, its limits defined, and the prefeasibility/feasibility studies indicate that an economic deposit exists.

Mine development is the transition stage between exploration and mine operation. Hence, many issues considered during exploration also need to be addressed, sampled, and monitored during this stage of mining. Permitting, which generally takes place during the mine development stage, can require significant environmental data to accommodate the permitting process.

The operations stage of mining follows and includes the process whereby ores and waste rock are removed from the ground, milled and/or leached, and concentrated into a salable product, such as a metal sulfide concentrate or high-grade gold and silver bullion. The operational units of mining and metallurgical processing can include the mine, overburden and waste-rock storage facilities, development waste materials, ore stockpiles, tailings storage facilities, crushing facilities, concentration facilities, leaching facilities, and other operational facilities. Each of these units can require sampling and monitoring, depending on potential contaminants and their handling and closure characteristics.

The end of the mine life cycle is closure/postclosure, and closure plans typically establish a postmining use of the mine site commensurate with the desires of the landowner and adjacent local land uses. Some sites can require long-term maintenance and monitoring. Historical, inactive, and abandoned mines, also known as legacy mines, offer the greatest challenge to the mining and environmental industries for reclamation.

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Courtesy of Virginia T. McLemore.

FIGURE 2.5 Monitoring field, Ambrosia Lake uranium subdistrict, Grants District, New Mexico

2.8 REFERENCES

Alaska Department of Environmental Conservation. 2009. Inspection report. http://dnr.alaska.gov/mlw/mining/largemine/kensington/pdf/dec/101509ir.pdf. Accessed 8/23/13.

Bove, D.J. 1994. *Preliminary Report on the Geology of Hydrothermally Altered Areas Within the Upper Alamosa River Basin, Colorado*. U.S. Geological Survey Open-File Report OF 94-224. http://pubs.usgs.gov/of/1994/0224/report.pdf. Accessed 8/23/13.

Castendyk, D.N., and Eary, L.E., eds. 2009. *Mine Pit Lakes: Characteristics, Predictive Modeling, and Sustainability*. Management Technologies for Metal Mining Influenced Water series, Vol. 3. Littleton, CO: SME.

Church, S.E., Fey, D.L., and Unruh, D.M. 2007a. Trace elements and lead isotopes in modern streambed and terrace sediment—Determination of current and premining geochemical baselines. In *Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado*. Edited by S.E. Church, P. von Guerard, and S.E. Finger. U.S. Geological Survey Professional Paper 1651. http://pubs.usgs.gov/pp/1651/. Accessed 8/23/13.

Church, S.E., Kirschner, F.K., Choate, L.M., Lamothe, P.J., Budahn, J.R., and Brown, Z.A. 2007b. *Determination of Premining Geochemical Background, Blue Creek Drainage, Stevens County, Washington*. U.S. Geological Survey Scientific Investigations Report 2007-5262. http://pubs.usgs.gov/sir/2007/5262/. Accessed 8/23/13.

Church, S.E., von Guerard, P., and Finger, S.E., eds. 2007c. *Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado*. U.S. Geological Survey Professional Paper 1651. http://pubs.usgs.gov/pp/1651/. Accessed 8/23/13.

CIM. 2005. CIM definition standards. web.cim.org/UserFiles/File/CIM_Definition_Standards_for_Mineral_Resources_and_Mineral_Reserves.pdf. Accessed 8/23/13.

Coeur Mining, Inc. n.d. Kensington, Alaska, 100% gold operation. www.coeur.com/operations/kensington-alaska#.UbYnp6BUHTo. Accessed 8/23/13.

Dahlberg, K. 1999. 14 steps to sustainability. Mining Report Card, Placer Dome, Inc. Washington, DC: Mineral Policy Center. www.earthworksaction.org/files/publications/14stepstosustainability.pdf. Accessed 8/23/13.

Diehl, S.F., and Schuster, R.L. 1996. Preliminary geologic map and alteration mineralogy of the main scarp of the Slumgullion landslide. In *The Slumgullion Earth Flow: A Large-Scale Natural Laboratory*. Edited by D.J. Varnes and W.Z. Savage. U.S. Geological Survey Bulletin 2130. http://pubs.usgs.gov/bul/b2130/. Accessed 8/23/13.

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Dowd, P.A. 2002. The assessment and analysis of financial, technical, and environmental risk in mineral resource evaluation. In *Deposit and Geoenvironmental Models for Resource Exploitation and Environmental Security*. Edited by A.G. Fabbri, G. Gaal, and R.B. McCammon. Netherlands: Kluwer Publishers. pp. 187–221.

Golden, K. 2008. Acid drainage found at Kensington mine: Juneau Empire, September 30, 2008. www.juneauempire.com/stories/093008/loc_338558528.shtml. Accessed 8/23/13.

Gray, J.E., Coolbaugh, M.F., Plumlee, G.S., and Atkinson, W.W. 1994. Environmental geology of the Summitville mine, Colorado. *Econ. Geol*. 89:2006–2014.

Hartman, H.L. 1992. Introduction to mining. In *Mining Engineering Handbook*. Edited by H.L. Hartman. Littleton, CO: SME. pp. 3–42.

Jones, H. 2011. Mine closure: Plan for the end from the beginning. *Min. Eng*. 63(5):107–109.

Jones, W.R. 2007. History of mining and milling practices and production in San Juan County, Colorado, 1871–1991. In *Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado*. Edited by S.E. Church, P. von Guerard, and S.E. Finger. U.S. Geological Survey Professional Paper 1651. http://pubs.usgs.gov/pp/1651/. Accessed 8/23/13.

JORC (Joint Ore Reserves Committee). 2012. *Australasian Code for Reporting of Exploration Results, Mineral Resources and Ore Reserves (JORC Code)*. www.jorc.org/docs/jorc_code2012%284%29.pdf. Accessed 8/23/13.

Kiefer, M. 1998. Cleaning up the Creek: A mining company mucks out its own mess at Pinto Creek. *Phoenix News Times*, May 7.

King, T.V.V., ed. 1995. *Environmental Considerations of Active and Abandoned Mine Lands, Lessons from Summitville, Colorado*. U.S. Geological Survey Bulletin 2220. http://pubs.usgs.gov/bul/b2220/b2220.pdf. Accessed 8/23/13.

Kuipers, J., Maest, A., MacHardy, K., and Lawson, G. 2005. Evaluation of the National Environmental Policy Act (NEPA) process for estimating water quality impacts at hardrock mine sites. SME Preprint 05-89. Littleton, CO: SME.

Kuipers, J.R., Maest, A.S., MacHardy, K.A., and Lawson, G. 2006. *Comparison of Predicted and Actual Water Quality at Hardrock Mines: The Reliability of Predictions in Environmental Impact Statements. Kuipers and Associates and Buka Environmental*. http://pebblescience.org/pdfs/ComparisonsReportFinal.pdf. Accessed 8/23/13.

Lord, D., Ethridge, M., Wilsom, M., Hall, G., and Uttley, P. 2001. *Measuring Exploration Success: An Alternative to the Discovery-Cost-per-Ounce Method of Quantifying Exploration Effectiveness*. Society of Economic Geologists, Newsletter No. 45, April.

Marcoline, J.R., Beckie, R.D., Smith, L., and Nicol, C.F. 2003. Mine waste rock hydrogeology: The effect of surface configuration on internal water flow. In *Proceedings, Sixth International Conference on Acid Rock Drainage*, Cairns, Queensland, Australia, July 14–17. Victoria, Australia, Australasian Institute of Mining and Metallurgy. pp. 911–918.

McCarthy, P.L. 2010. Objectives of feasibility studies. AMC Consultants internal paper. www.scribd.com/doc/91679597/Objectives-Feasibility-Studies. Accessed 8/23/13.

McLemore, V.T., ed. 2008. *Basics of Metal Mining Influenced Water*. Management Technologies for Metal Mining Influenced Water series, Vol. 1. Littleton, CO: SME. www.smenet.org/store/mining-books.cfm/Basics-of-Metal-Mining-Influenced-Water/259-8. Accessed 8/23/13.

McLemore, V.T. 2009. *Geologic Setting and Mining History of the Questa Mine, Taos County, New Mexico*. Open-File Report 515. Socorro, NM: New Mexico Bureau of Geology and Mineral Resources. http://geoinfo.nmt.edu/publications/openfile/details.cfml?Volume=515. Accessed 8/23/13.

Miller, R.A., and Hertel, T.M. 1997. Mine rock characterization: Zortman and Landusky mines, Little Rocky Mountains, Phillips County, North-Central Montana. In *Proceedings of the Fourth International Conference on Acid Rock Drainage*, Vancouver, BC, Canada, May 31–June 6. pp. 515–532.

NI 43-101. 2011. Rules and policies. In *Standards of Disclosure for Mineral Projects*. 34 OSCB 7086. www.ccpg.ca/profprac/en/Documents/ni_20110624_43-101_mineral-projects.pdf. Accessed 8/23/13.

Nordstrom, D.K. 2007. *Questa Baseline and Pre-Mining Ground-Water Quality Investigation. 25. Summary of Results and Baseline and Pre-Mining Ground-Water Geochemistry, Red River Valley, Taos County, New Mexico, 2001–2005*. U.S. Geological Survey Professional Paper 1728. http://pubs.usgs.gov/pp/1728/pdf/p1728_508.pdf. Accessed 8/23/13.

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O'Kane, M., and Waters, P. 2003. Dry cover trails at Mt. Whaleback—A summary of overburden storage area cover system performance. In *Proceedings, Sixth International Conference on Acid Rock Drainage*, Cairns, Queensland, Australia, July 14–17. Victoria, Australia: Australasian Institute of Mining and Metallurgy. pp. 147–153.

Pendleton, J.A., Posey, H.H., and Long, M.B. 1995. Characterizing Summitville and its impacts: Summarizing the scene. In *Summitville Forum Proceedings*. Edited by H.H. Posey, J.A. Pendleton, and D. Van Zyl. Colorado Geological Survey Special Publication 38. pp. 1–12.

Plumlee, G.S., and Nash, J.T. 1995. Geoenvironmental models of mineral deposits—Fundamentals and applications. In *Preliminary Compilation of Descriptive Geoenvironmental Mineral Deposit Models*. U.S. Geological Survey Open-File Report 95-0831. http://pubs.usgs.gov/of/1995/ofr-95-0831/. Accessed 8/23/13.

Russell, C. 2006. Mine drainage challenges from A to Z in the United States. In *Proceedings, 7th International Conference on Acid Rock Drainage (ICARD 7)*. Lexington, KY: American Society of Mining and Reclamation. pp. 1785–1802. www.asmr.us/Publications/Conference%20Proceedings/2006/1785-Russell-CO.pdf. Accessed 8/23/13.

Schmiermund, R.L., and Ranville, J. 2006. Application of geo-environmental and biotic ligand models in the life cycle of mine development—A flowchart for integration. In *Proceedings, 7th International Conference on Acid Rock Drainage (ICARD 7)*. Lexington, KY: American Society of Mining and Reclamation. pp. 1920–1925. www.imwa.info/docs/imwa_2006/1920-Schmiermund-CO-3.pdf. Accessed 8/23/13.

Schmiermund, R.L., Lazo, M.C., and Parnow, C.C. 2006. Application of geo-environmental models to accelerated EIA and permitting processes for an Andean porphyry Cu-Au deposit. In *Proceedings, 7th International Conference on Acid Rock Drainage (ICARD 7)*. Lexington, KY: American Society of Mining and Reclamation. pp. 1903–1911. www.imwa.info/docs/imwa_2006/1903-Schmiermund-CO-1.pdf. Accessed 8/23/13.

Semple, R.B., Jr. 1995. Canceling the New World mine. *The New York Times*, 10 December.

USEPA (U.S. Environmental Protection Agency). 2011a. Summitville mine, Del Norte, Colorado, Superfund Program (EPA ID: COD983778432). www2.epa.gov/region8/summitville-mine. Accessed 8/23/13.

USEPA (U.S. Environmental Protection Agency). 2011b. Uravan Uranium Project (Union Carbide), Uravan, Colorado, Superfund Program (EPA ID: COD007063274). www2.epa.gov/region8/uravan-uranium-project-union-carbide. Accessed 8/23/13.

Vincent, K.R. 2008. *Questa Baseline and Pre-Mining Ground-Water Quality Investigation. 17. Geomorphology of the Red River Valley, Taos County, New Mexico, and Influence on Ground-Water Flow in the Shallow Alluvial Aquifer*. U.S. Geological Survey Scientific Investigations Report 2006–5156. http://pubs.usgs.gov/sir/2006/5156/. Accessed 8/23/13.

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CHAPTER 3

Sampling Considerations in the Mining Environment

Kathleen S. Smith, Virginia T. McLemore, and Carol C. Russell

(with contributions by Andrew S. Todd, James J. Gusek, Lisa Bithell Kirk, Karl L. Ford, and Susan J. Caplan)

3.1 INTRODUCTION

The intent of this chapter is to highlight some information and issues that relate to sampling at mining and processing sites, and to discuss some relevant information from recent research studies. Internet sources related to this chapter are listed in Section 3.13. Appendices 2 and 3, which summarize many types of sample-collection and analytical methods, are supplementary and complementary to the information herein. In addition, Appendix 1 lists sources for online resources related to sampling, and Appendix 5 consists of several case studies that illustrate different aspects of sampling and monitoring. Sampling and monitoring of pit lakes are discussed in volume 3 of the Management Technologies for Metal Mining Influenced Water series, *Mine Pit Lakes: Characteristics, Predictive Modeling, and Sustainability* (Castendyk and Eary 2009); considerations related to mitigation of mining influenced water (MIW) are covered in volume 2, *Mitigation of Metal Mining Influenced Water* (Gusek and Figueroa 2009); prediction methods are included in volume 5, *Techniques for Predicting Metal Mining Influenced Water* (Williams and Diehl 2014); and geochemical modeling is covered in volume 4, *Geochemical Modeling for Mine Site Characterization and Remediation* (Nordstrom et al., forthcoming). The general media types of water, solids, biological (aquatic), and air are included in this chapter. Table 3.1 lists many of the types of data and sampling media that can be considered for inclusion in sampling and monitoring programs.

Metals present unique sampling issues in that they are naturally occurring and have variable content in solids and variable concentrations in MIW from different geographic areas. In addition, some metals are essential nutrients but can be toxic at higher concentrations in a variety of media. Also, the form of a metal can control its mobility, bioavailability, and toxicity, and metal-ion mixtures can have additive or synergistic toxicological effects. Because metals generally occur in mixtures at mining and processing sites, there can be several pathways and receptors of interest due to the potentially dissimilar behaviors of different metals. These factors make sampling for metals at mining sites a challenge and underscore the importance of having a fundamental understanding of the physicochemical and biogeochemical processes that control metal behavior. Nordstrom (2011) provides an overview of hydrogeochemical processes that control metal mobility in mined and mineralized systems.

At the outset of a sampling program, it is essential to define the reasons for sampling, the questions to be answered, the desired analytes and sampling media, and the desired degree of

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TABLE 3.1 Data and sampling media for sampling and monitoring programs

Physical	
Topography and altitude	Stream/river flow data
Natural drainage vs. human-made drainage	Slope aspects
Geological	
Structural geology (faults, fractures, joints, lineaments)	Geomorphology
Seismic data/earthquake prediction	Deposit type
Stratigraphy and lithology	Mining methods
Particle size analysis	Mineralogy
Aquifer Characteristics	
Permeability/porosity	Groundwater flow direction and rates
Hydraulic conductivity	
Climatological	
Temperature	Growing season
Evapotranspiration	Days of sunlight
Rainfall/snowfall	Floods (10 yr, 100 yr, etc.)
Freeze-thaw	Storm events and types
Wind direction and speed	Probable maximum precipitation event
Solid	
Overburden	Tailings
Country rock	Soils/topsoil
Ore	Subsurface samples (drill core, cuttings, other)
Sub-ore or low-grade material stockpiles	Sediments (lake, stream, river, overbank)
Sludge	Spent leach ore/material
Waste (piles, other)	
Liquid	
Surface water Storm water (impacted and non-impacted) Stream/river Lakes and ponds Processed wastewater Other wastewater	Human-made discharge points (adits, tunnels, pit walls, etc.) Rainwater/snow melt Brine Residuals from passive treatment Process solutions
Groundwater	Pit lakes
Leachate	Seeps
Surface runoff	Springs
Air	
Gases (oxygen flux, cyanide, mercury)	Aerosols
Particulates (dust)	Fugitive dust
Biological	
Humans	Habitats
Animals Aquatic (macroinvertebrates, vertebrates, fish) Terrestrial	Plants Aquatic (algae) Terrestrial
Microorganisms (bacteria) Aquatic Terrestrial	

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confidence in the answer(s). Numerous textbooks, compilations, handbooks, and articles address the types, quantity, and quality of data (e.g., Keith 1991; Horowitz et al. 1994; USEPA 1984, 2000, 2001a, 2001d, 2003b, 2006c, 2007b, 2007c, variously dated; Tremblay and Hogan 2001a, 2001b; Zhang 2007; Rice et al. 2012; ICMM 2008; INAP 2009; GRI 2010; USGS variously dated; BCMOE 2012). The U.S. Environmental Protection Agency (USEPA) provides links (listed in Section 3.13) to several methods for environmental sampling. Callender (2003) reviews sources and distribution of heavy metals in a variety of media.

USEPA has also developed an agency-wide Quality System that includes a series of guidance documents (see Section 3.13; USEPA 2002a). USEPA's data quality objectives (DQOs) guidance document (USEPA 2006b) describes the DQO process, which is USEPA's recommended planning procedure when environmental data are used to select between two alternatives or to derive an estimate of contamination. USEPA's Quality System is discussed in more detail in Chapter 4 of this handbook.

There has been an evolution in the approach for characterization of hazardous waste sites, which may also be applied to environmental sampling and monitoring of mining and processing sites. Traditional sampling and analysis programs have been relatively inflexible, and data quality has been focused on analytical error, with little concern for other factors (e.g., sampling techniques) that can influence data quality. To address some of these issues, Crumbling et al. (2001) introduced the Triad approach, which is an integrated work strategy to manage decision uncertainty that allows consideration of all parameters that influence data quality. Triad is a federal and state interagency partnership (see Section 3.13). The Triad approach relies on thorough, systematic planning to set clear project goals. The three elements of the Triad approach are (1) systematic (or strategic) project planning, (2) dynamic work strategies, and (3) real-time measurement technologies. This approach emphasizes the need for a multidisciplinary team of experts for planning and decision making. Dynamic work strategies allow work planning documents to be written in a flexible way so that the course and decisions of the project can adapt in real time as new information becomes available. The Triad approach allows for a variety of analytical techniques. If possible, initial analytical measurements should be made in the field (real-time) so that they can inform the dynamic work strategies while the crew is still in the field. Some techniques that can support real-time measurements include field kits, field analytical instruments, in situ sensing systems, field leaching tests, and some geophysical techniques. The Triad approach reinforces the distinction between data quality and analytical quality, and considers sampling quality to be just as important to data quality as is analytical quality. Data quality can be achieved by initially using results from more rapid, often less expensive, analytical methods to provide high-density data that can assess sample representativeness. The level of tolerable error depends on the objectives of the investigation. Although the Triad approach has yet to be adopted for mining sites, screening level approaches have been utilized to assess potential environmental considerations at mining sites. For example, Smith et al. (2002) discuss tools for rapid screening and characterization of waste piles, and Jordan (2009) outlines an approach by which geological information can be used in the environmental assessment of mining areas. Schmidt et al. (2012) and Schmidt (Appendix 5, this handbook) discuss the links between geology and potential impacts on aquatic ecosystems. Once sample representativeness is assessed, more precise and accurate analytical techniques can be used if necessary.

Geo-environmental mineral deposit models (GEMs; also discussed in Chapter 2 and in Appendix 6) are a geology-based classification system for identifying environmental challenges associated with specific types of mineral deposits and can provide an initial prediction of water quality at a given mine site based on the type of mineral deposit (e.g., Plumlee et al. 1994, 1995,

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1999; Appendix 6). In addition to being useful in developing a site conceptual model, GEMs are helpful as a guide in forecasting and prioritizing potential environmental considerations and challenges at a particular site, in assessing potential remediation options, and as a reality check for evaluating numerical modeling results. Information about GEMs is provided by du Bray (1995), Plumlee and Nash (1995), Plumlee (1999), Plumlee et al. (1999), Wanty et al. (1999), Seal and Foley (2002), Kwong (2003), Seal and Hammarstrom (2003), Schmiermund et al. (2006), Schmiermund and Ranville (2006), and Seal et al. (2009).

3.1.1 Background, Baseline, and Premining Conditions

A topic of potential confusion that deserves mention is the difference between the terms *background* and *baseline*. These words are often used interchangeably, but many researchers make a distinction between them. Therefore, when discussing background or baseline conditions, it is critical to define the terms in the context that they are being applied. Lee and Helsel (2005) define the term *environmental background* as being descriptive of the natural tendency of an environmental system, or material, in the absence of human influences. In contrast, they define an *environmental baseline* as a summary of existing conditions over some time frame for some environmental system or material of interest. They also state that baselines can, and typically do, include influences of human activities such as land use or mining and processing. Galuszka (2007) defines *baseline* as a measure of a given sample in a specific location and time. The term *natural background* has been widely used to refer to background levels that are not influenced by human activities. Runnells et al. (1992) suggests that *natural background* conditions be the remediation goal at environmentally impacted mining sites. USEPA policies require cleanup to *natural background conditions* (see USEPA 1997), where natural background is defined as concentration due only to nonanthropogenic sources. Reimann and Garrett (2005) introduced the term *ambient background* to describe "the unmeasurably perturbed and no longer pristine natural background." The European Chemicals Agency (ECA 2008) provides guidance on determining the background and baseline for metals. Because background usually represents conditions prior to any human influence, background conditions often cannot be accurately determined and are commonly determined based on conditions at analog sites that have not been subjected to human-induced changes, or calculated based on modeling or projections to past conditions. These types of projections involve establishing the spatial and temporal distribution of elements of interest, and determining their behavior in response to different physicochemical and biological conditions.

Premining conditions are those that existed prior to mining, regardless of other human influences. Estimation of premining conditions has been a topic of recent interest and research. For example, considerable work is being done to determine premining conditions at the Pebble mine in Alaska (Alaska DNR 2013). Mine sites are generally located in areas with naturally elevated metal concentrations in water, and metal contents of sediment, soil, vegetation, and other biota. Therefore, it is important to conduct careful sampling and monitoring before mining to document premining conditions at proposed mine sites (Alpers and Nordstrom 2000; McLemore et al. 2007). It also is important to develop scientifically defensible approaches and methods to estimate premining conditions at existing mine sites where no premining conditions were documented (e.g., Church et al. 2007a, 2007b). The U.S. Geological Survey (USGS), in cooperation with the New Mexico Environment Department and supported by Chevron Minerals Co. (formerly Molycorp, Inc.), conducted the *Questa Baseline and Pre-Mining Ground-Water Quality Investigation* (Nordstrom 2008) to estimate premining groundwater quality at the Questa molybdenum mine in northern New Mexico. USGS identified several geochemical trends and found

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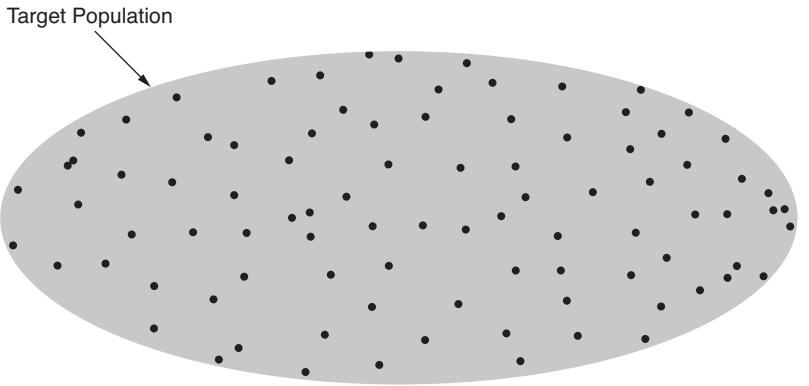


FIGURE 3.1 Illustration showing the relationship between a target population (gray oval) and a Multi Increment sample, represented by the black dots

that these trends, combined with mineral-solubility controls and site-specific lithology and mineralogy, provide constraints on premining groundwater quality (summarized in Nordstrom 2008; see USGS 2011). USGS conducted detailed studies in the Upper Animas River Basin, Colorado, which is an area with a legacy of MIW from historical metal production (see USGS 2013b). Characterization, monitoring, and process-oriented studies provide insights for effective remediation strategies and estimation of premining conditions (summarized in Church et al. 2007c; see case studies by Church and Williams in Appendix 5). Verplanck et al. (2009) provide an overview of controls on water quality in several areas draining different porphyry mineralized areas, and Runkel et al. (2007) present a simulation-based approach for estimating premining water quality.

3.2 SAMPLING OVERVIEW

Sampling is the process by which inference is made to the whole by examining a part. The sampling process should be defined by the DQOs (USEPA 2006b) and the sampling and monitoring program (see Figure 4.1 and discussion of international guidelines in Section 4.3, both in Chapter 4), which incorporates the project sampling plan, quality assurance plans (USEPA 2001b), and standard operating procedures (USEPA 2007c). The target population is that which is being sampled and is defined based on the objectives of the investigation. It is critical that the target population be clearly identified in detail at the beginning of the investigation, be understandable to the users, and that all of a given target population be equally available for sampling. Examples of a target population might include historical fluvial mill tailings within the 100-year flood plain of a stream, or fish along a particular stream reach, or groundwater contamination downgradient from an environmentally impacted mine site, or the top inch of soil in a particular area. It is likely that there will be multiple target populations for a given investigation.

A *sample* is that portion of the target population that is actually studied and used to characterize the target population. For the purposes of this discussion, a sample consists of several increments (subsamples) collected from the target population (Figure 3.1). Multi Increment (MI) sampling (EnviroStat, n.d.) is a sampling methodology that can be used to collect a representative sample and is discussed in more detail in Section 3.4.8.1.

The sample needs to be representative of the target population. The term *representative* commonly is loosely defined in qualitative terms. Determining *sample representativeness* involves careful planning and formulation of a proper sampling design. The representativeness of samples can be addressed by using either a probabilistic approach (e.g., Rollinson 1993; Koch and Link

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WHAT DOES *REPRESENTATIVE SAMPLE* MEAN?

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The term *representative sample* has been used for many applications. It is actually an operationally defined term that depends on the objectives of the investigation and the definition of the target population. A representative sample is both unbiased and precise within limits specified in the objectives. For a given investigation, the meaning of representative sample must be defined so that the ultimate decision-making needs of the investigation can be met. The concept of a representative sample exists throughout the life cycle of an investigation, from field sampling, to maintaining sample integrity, to sample preparation through staged comminution, to laboratory subsampling, to sample analysis. Determining sample representativeness involves careful planning and formulating a proper sampling design.

For field sampling, sample preparation, and laboratory subsampling of particulate materials, it all comes down to the sampling error. "There is always some degree of sampling error unless the entire population is analyzed" (Ramsey and Hewitt 2005). Pierre Gy (1982, 1998) studied the technical and statistical aspects of sampling error applied to mineral processing, and Pitard (1993) further refined Gy's sampling theory. Sampling error is related to the heterogeneity (both compositional and distributional) of the target population. Two primary sources of precision error are (1) the grouping and segregation error (GSE), which is related to distributional heterogeneity and number of subsamples collected to compose a sample; and (2) the fundamental sampling error (FSE), which is related to the compositional heterogeneity, particle size, and the mass of the sample. The major source of bias error can be minimized by using correct sampling practices and devices (see Pitard 1993). As a rule of thumb, Pitard (1993) recommends collecting at least 30 increments (subsamples) to constitute a sample in order to reduce the GSE relative to the FSE (in practice, 50–100 increments are usually required). Hence, when correct sampling practices are employed and sufficient increments are collected, then the majority of the total sampling error is due to the FSE.

The FSE can be estimated prior to sampling and can be reduced by increasing the sample mass or decreasing the particle diameter. The FSE can be estimated using the following equation (when the sample size is much smaller than the target population being sampled):

$$\sigma^2_{FSE} = (K\, d^3)/M_s$$

where M_s is the sample mass (g), d is the maximum particle diameter (cm), and K is an empirical constant for a given material that is related to the particle shape, range of particle sizes, particle mineralogical composition, and particle liberation.

To obtain representative samples, it is essential to be aware of the properties of heterogeneous materials and the guidance provided by sampling theory. Investigators need to proactively design sampling and analysis programs with clearly defined data quality objectives to achieve data of known quality and integrity (Wait and Ramsey 2007).

2002; Schreuder et al. 2004; USEPA 2006a) or a sampling theory approach (see Representative Sample sidebar and Sections 3.4.8 and 3.4.9). If using a probabilistic approach, a software tool called Visual Sample Plan (VSP) developed by the Pacific Northwest National Laboratory (Richland, Washington) can help develop a sampling plan that supports the DQO process (see Section 3.13 and Nuffer et al. 2009). VSP is founded on statistically based sampling designs. Decision Error Feasibility Trials (DEFT; USEPA 2001a) is another computer model that supports the DQO process (see Section 3.13). DEFT assists with the seven-step DQO process (USEPA

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2006b) and is used to develop statistical sampling design plans that allow users to evaluate the financial feasibility of incorporating selected DQO constraints into a statistical sampling design before developing a final plan.

A sampling theory approach considers the effect of both sampling and analytical errors on the representativeness of samples (Crumbling et al. 2001; Gy 1982, 1998; Maney and Wait 2005; Pitard 1993; Ramsey and Hewitt 2005; Wait and Ramsey 2007). The major source of uncertainty in environmental data sets is due to the heterogeneity of environmental materials. Use of sampling theory can help minimize sampling errors that are due to the heterogeneity of the target population (see Sections 3.4.8 and 3.4.9).

Sampling methods are the procedures of selecting those sampling units that provide estimates of the whole with associated margins of uncertainty. Poor sampling can introduce sample bias as well as lack of precision. Uncertainty arises from examination of only a part and not the whole. For example, selecting a specific sample size fraction in characterizing sulfidic waste rock piles becomes important and a bias can be introduced because the acid-generating and acid-neutralizing minerals are not evenly distributed in the various size fractions. Similarly, surface sampling of environmentally impacted pit lakes can provide an incorrect estimate of contaminant loads if the pit lake is stratified with respect to dissolved constituents. In such cases, replicate sampling or increasing sample volume or mass will not correct the bias.

In summary, it is essential to carefully define the target populations during the sampling planning phase. There are four main groups of sampling media (solid, liquid, biological, and air) that are generally considered by a sampling and monitoring program. The types of media sampled will be determined by the anticipated sources, pathways, and receptors potentially impacted by mining and processing activities, as well as by other factors such as climate and regulatory requirements. It is important to consider and address the types of sample variation (e.g., spatial and temporal). It is helpful to consider use of indicator species, either aqueous or biological, to provide an early warning system for potential environmental impacts.

3.3 WATER SAMPLING CONSIDERATIONS

The purpose of collecting water and process-solution samples at mine and processing sites can be to assess water quality, provide data for contaminant loading calculations, use in toxicological testing, locate contaminant source(s), provide input for modeling efforts, establish baseline conditions, manage water treatment processes, or meet regulatory requirements. A general summary of water quality characteristics is discussed by Hem (1985), a compilation of the biogeochemistry of major world rivers is given by Degens et al. (1991), and acid rock drainage (ARD) sampling is described by Downing (2011). Some Web sites that discuss general characteristics of water are listed in Section 3.13. The National Water Quality Monitoring Council also maintains a Web site and offers training and information relevant to the sampling and monitoring of water.

MIW can have many possible characteristics (Nordstrom and Alpers 1999; see Appendix 3 for discussion of water constituents and field measurements). Schmiermund and Drozd (1997) list five common possible characteristics of MIW, which include low pH, high sulfate (SO_4^{2-}), high iron and aluminum, high non-iron metals, and high turbidity. Importantly, not all MIWs need to have all of these characteristics (e.g., not all MIW is acidic), and each characteristic often requires different sampling and monitoring techniques. When evaluating an existing MIW, care should be taken to fully characterize the system, both absolutely and in terms of an appropriate baseline or background (Schmiermund and Drozd 1997; Smith 2011). For example, MIW from a cyanidation processing operation discharge may be much different than from a MIW source that contains high-pH values and metal species present as anions instead of cations. Focusing

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characterization activities solely on regulated constituents often results in incomplete or incorrect characterization, which could lead to costly oversights and insufficient data for remediation planning.

Major advances have been made in developing *clean* sampling procedures and extremely sensitive analytical techniques for trace elements. However, sampling procedures that address and capture the natural variability of constituents in water need some further development; sampling procedures can frequently be the major source of error in the overall process. It is critical to use current, relevant, documented sampling protocols and to record how, when, and where samples were collected, preserved, and analyzed. Textbook preservation procedures and holding times may not always be applicable to MIW. Numerous protocols describe sampling, preservation, and analytical procedures (e.g., USGS, variously dated). Great care must be taken to ensure that water quality sampling procedures, sampling devices, and preservatives do not contaminate the sample, react with the sample, or interfere with the analytical method, and all such information needs to be recorded in the sampling plan documents. In addition, documentation of calibration should be included for all field instruments. When stream sediment is also being sampled, the water sample needs to be collected first to avoid contamination from the disturbed sediment. Experienced personnel should be chosen to collect water quality samples. Collection of quality assurance/quality control (QA/QC) samples, such as replicates and blanks, is very useful to determine if any contamination has occurred and whether variation in constituents between samples is due to contamination or to environmental variation. It is important to document such things as frequency of sampling; whether samples were composited; where in the stream samples were collected; whether samples were collected using a pump, a wire sampler, a composite sampler, or by a grab method; the existence of QA/QC samples; and whether samples were filtered (and filter size and type). It also is recommended that ambient weather conditions before and during the sampling event be recorded. For example, does the sampling event occur during a prolonged drought at the site or perhaps hours/days after the site received significant rainfall (see Section 3.3.6.1)?

Wire samplers are often used in metallurgical plants to collect shift composite samples. In this sampling procedure, a wire is passed through a constant stream of solution and a portion of the sample is diverted to flow down the wire to drip into a sampling bottle.

3.3.1 Filtration

Filtration and filtration techniques can affect the concentration of constituents of interest in the sample, so filtration should be specifically addressed in the sampling plan and procedures, and be consistent with the overall objectives of the investigation. Filtration through a 0.45-μm pore-size membrane is a widely accepted, regulation-driven method to separate particulates from water to obtain the *dissolved* fraction. However, this pore size is arbitrary and does not always remove all of the particulates from water. Examples of some particulates that may not be filtered include colloids (suspended solids, sometimes referred to as nanoparticles) and some microorganisms. Ranville and Schmiermund (1999) have presented a discussion and overview of colloids. Notably, the use of acid or other additives for preservation and the digestion of filtrates using acid or other chemicals likely dissolves at least some of the particulate material that passes through the filter.

Sampling of slurries, such as tailings, in metallurgical plants may require the use of a primary cross-stream sampler followed by one or more rotary Vezin samplers. The slurries are typically pressure filtered to separate the solids from the solution. Fine filtration can follow this preliminary separation, and gravity filtration in a funnel of small volumes of slurries is also a common practice for extraction of solution for metallurgical process control measurements.

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Filters and filtration devices can be sources of contamination and need to be carefully precleaned when trace constituents are of interest. Also, there can be artifacts as a result of filtration (e.g., Danielsson 1982; Horowitz et al. 1992, 1996). For example, Horowitz et al. (1996) have illustrated that filter clogging changed the effective pore size of filters during filtration. When possible, it is good practice to filter samples in the field if filtration is necessary. Filtration of groundwater samples is discussed in Section 3.3.7.

3.3.2 Unstable Constituents and Field Measurements

Reduction-oxidation (redox) potential determination should not be considered to be a routine determination due to the difficulties inherent in its theoretical concept and its practical measurement (Nordstrom and Wilde 2005). Redox and Eh can be measured by several methods (see Appendix 3 for more detailed information). Eh determination using a platinum (or other noble metal) electrode is valid only when redox species are electroactive and present in the solution at concentrations greater than about 10^{-5} molal (Nordstrom and Wilde 2005). Generally, a single Eh measurement does not represent the entire system; in many MIWs, iron controls the response of the electrode because of its elevated concentration. Eh measurements can also be used to determine sulfide in sulfate-reducing systems (Nordstrom and Wilde 2005). Most redox species can be directly measured, which can be preferable to Eh measurements (see Baedecker and Cozzarelli 1992).

A number of parameters are traditionally measured in the field or processing plant rather than in the laboratory. These include temperature, specific conductance, pH, dissolved oxygen (DO), turbidity, and flow rate. Also, acidity/alkalinity samples need to be kept cool and be analyzed as soon as possible. Some measurements, such as turbidity, are designed to be measured in the field. When it is uncertain whether a particular constituent or parameter is stable over time, it is better to try to measure that constituent in the field. For example, in MIW resulting from ARD, the pH value should always be measured in the field because pH can change with time due to precipitation of hydrous iron oxides, evolution of carbon dioxide, and other issues. Field techniques are discussed in more detail in Appendix 3, and a review of field methods is given in Ficklin and Mosier (1999). Additionally, many types of speciation measurements must be performed in the field because the species being measured are not stable. When considering speciation sampling, it is cost-effective to first analyze for total concentration of the constituent to determine if the constituent is present at a concentration level that is sufficient for speciation analysis. Various field test kits are available for a number of constituents. These test kits can be very useful in determining unstable constituents or aqueous species and are discussed in more detail in Appendix 3.

3.3.3 Sensors and Passive Sampling Devices

Recent advances in sensor technology allow for greater capabilities in water quality monitoring. The ability to collect continuous, rather than discrete, data provides the foundation for many potential applications in sampling and monitoring that allow for better understanding of constituent variability. For example, continuous and remote monitoring are potential tools for determining total maximum daily loads (TMDLs), which are generally developed and monitored using discrete water samples collected on a fixed schedule; Walton-Day et al. (2012) discuss some of the hydrogeochemical complexities involved in developing TMDLs. Roig (2007) presents performance evaluation and validation information for several alternative screening methods (i.e., physicochemical devices, biosensors, bioassays), Glasgow et al. (2004) review advancements in sensor technology, Chapin and Todd (2012) describe an in situ sampling device that can improve sampling resolution, Wanekaya et al. (2008) provide an overview of several types of biosensors,

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and Horsburgh et al. (2010) discuss the use of surrogates to monitor constituents using sensor technology.

Passive sampling devices (e.g., diffusion gradients in thin films, stabilized liquid membrane devices) offer another alternative to discrete sampling. In passive sampling, the analyte concentration is integrated over the sampling period; passive samplers do not require an energy source and can be deployed in many types of environments (e.g., Brumbaugh et al. 2002; Balistrieri et al. 2007). However, passive sampling does not provide information about constituent variability. Vrana et al. (2005) review passive sampling techniques for environmental applications. Madrid and Zayas (2007) point out that one of the most important shortcomings of the use of sensors and passive sampling devices is validation under field conditions. See Appendix 3 for further discussion of various sampling devices.

3.3.4 Water Balance

It is essential to understand the water balance (also called water budget) in a given study area (see Argo Tunnel sidebar). Russell (2006) points out that USEPA has found a common thread among recently active Superfund mine sites: they did not have an accurate water balance. A water balance is the rate of change in water stored in an area (e.g., a watershed) balanced by the rate at which water flows into and out of the area. Water balances are fundamental to the conceptualization of hydrologic systems at all scales (Healy and Scanlon 2010.) Developing a water balance includes collection of hydrological (e.g., flow or discharge measurements, groundwater flow parameters) and climatological (e.g., precipitation, temperature, evapotranspiration) information. Some factors that can play an important role in a site-wide water balance include seasonal flow variability, runoff variability, evapotranspiration, lag discharge (e.g., storage in waste rock piles), mine dewatering, water application for dust control, process discharge retention in tailings facilities, and consumption. Healy et al. (2007) present an overview of water balances and provided several case studies. Water sources and losses need to be identified and, at least partially, quantified.

3.3.5 Groundwater/Surface-Water Interactions

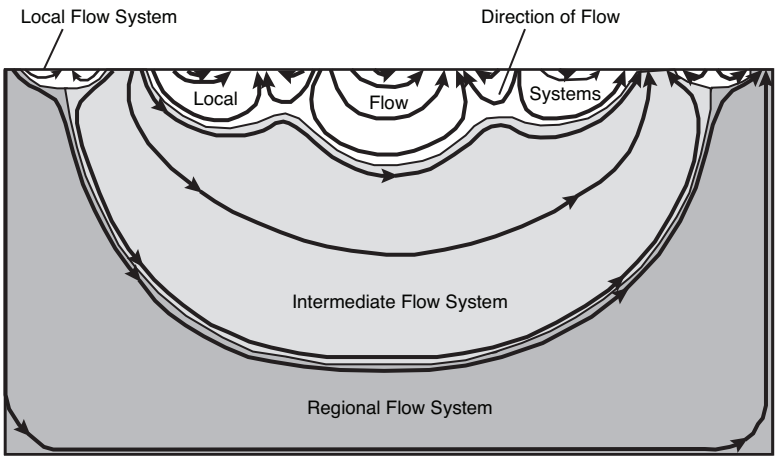
Understanding interactions between groundwater and surface water is an important, often overlooked consideration in water management. Winter (1999) discusses the influence of surface-water bodies (e.g., streams, lakes, wetlands) on groundwater flow systems. Winter et al. (1998) and Idowu (2007) provide an overview of groundwater/surface-water interactions. They point out that groundwater flow systems can be local, intermediate, and regional in scale. Intermediate and regional flow systems have longer flow paths and contact time with subsurface materials, and these waters tend to contain dissolved constituents that reflect the composition of the subsurface materials (e.g., Wanty et al. 2009a, 2009b). Local flow systems can be very dynamic and have interactions with surface-water systems (see Figure 3.2; Toth 1963; Winter et al. 1998). Interactions at the surface-water/groundwater interface (the *hyporheic zone*) can play an important role in the concentration and load of constituents in surface water and can have significant environmental influences on biogeochemical processes (Harvey and Fuller 1998; Bencala 2000, 2005; Fuller and Harvey 2000). Gandy et al. (2007) provide a review of information about attenuation of potential contaminants in the hyporheic zone. Bencala et al. (2011) point out that streams gain and lose water over a range of spatial and temporal scales, and that it is important to consider the influence of scale on contaminant and nutrient fluxes. It is important to keep these surface-water/groundwater interactions in mind when designing a sampling plan and locating sampling sites (e.g., Harvey and Bencala 1993). The hyporheic zone is discussed in more detail in Appendix 3.

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QUALITY AND CERTAINTY OF DATA—ARGO TUNNEL

Carol C. Russell, U.S. Environmental Protection Agency

An example of the need for an accurate water balance for an acid rock drainage (ARD) evaluation is clear when considering the Argo Tunnel, a 6.69-km (4.2-mile) tunnel completed in 1910 that passes from Idaho Springs (the portal) to underneath Central City in Colorado. The Argo Tunnel provided dewatering and ore haulage-ways draining more than 300 mines and is the largest single source of ARD and metals contamination to Clear Creek. As part of a Superfund remediation project, construction of a 700-gpm conventional sodium hydroxide treatment facility was completed in 1998. Although the plant has been successful in meeting the effluent quality goals, it has been at much higher costs than estimated due, in part, to inaccurate flow measurements of the ARD. Initial flows were measured at the portal, but the flows being captured several meters within the tunnel area are twice that of the initially measured flow (USEPA 2007d). Follow-up analysis of the flows found that fracture flow from the weathered surface near the tunnel exit and seasonal variations in flow are responsible for the differences in flow measurements. The plant was designed as a dual train system with one filter press. Rather than being able to run one train while the second was being maintained, both trains must function all of the time, necessitating more oversight and much more costly maintenance. Moreover, an additional filter press purchase incurred rush charges, and more filtration sludge than expected has led to increased disposal costs. Nevertheless, the plant continues to remove more than 453 kg (1,000 lb) of metal each day from the ARD before discharging to Clear Creek (CDPHE 2011). A document published by USEPA titled *Performing Quality Flow Measurements at Mine Sites* (USEPA 2001c) can assist in more accurate measurement of ARD from mines.



Source: Adapted from Toth 1963, as cited in Winter et al. 1998.

FIGURE 3.2 Different scales of groundwater flow systems: local, intermediate, and regional

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3.3.6 Surface-Water Samples

Dissolved metals occur in many chemical forms, including free ions and inorganic and organic complexes. The chemical speciation of metals influences their bioavailability to and toxicological effects on aquatic organisms. Constituents in surface water draining mined and mineralized areas tend to be geochemically, spatially, and temporally variable, which presents challenges in collecting water quality samples. Important considerations in spatial variability include location of water body in the catchment, the underlying lithology, presence of weathering ore deposits or waste, process discharges, climate, and geochemical processes that can transform and redistribute the analytes. Important temporal considerations include seasonal, episodic, and daily changes. This section addresses some of the important considerations in collecting surface-water samples from mined and mineralized areas.

3.3.6.1 Temporal Considerations

Temporal variation of metal concentrations can be significant in streams draining mined as well as mineralized, unmined areas, and for streams impacted by excess and treated process water from mine dewatering, MIW, and metallurgical processing. For example, Nagorski et al. (2003) conducted a year-long study of four Montana streams and found that

1. Monthly changes were dominated by snow melt and precipitation dynamics, which tend to follow an annual cycle;
2. Daily-scale changes were dominated by episodic events, such as thunderstorms, that resulted in short-term solute and particulate concentrations similar in magnitude to those during the early spring flush; and
3. Diel (i.e., 24-hour) cycles for several constituents and some geochemical parameters were similar in magnitude to much longer (monthly) time scales.

Nimick et al. (2003) reported substantial diel cycles in dissolved metal concentrations (e.g., arsenic, cadmium, manganese, nickel, and zinc) during low-flow conditions along 12 neutral- and alkaline-pH streams draining historical mining areas. In their review of biogeochemical diel cycles in streams, Nimick et al. (2011) stated that the amplitude of diel changes for some water constituents can be as large as changes occurring on annual time scales (see Diel Biogeochemical Processes sidebar). In a study of the seasonality of diel cycles, Nimick et al. (2005) stated that "Diel cycles of dissolved metal concentrations should be assumed to occur at any time of year in any stream with dissolved metals and neutral to alkaline pH." Diel cycles have also been documented in acidic streams and appear to be related to photoreduction of iron oxyhydroxides (e.g., McKnight et al. 1988; Sullivan and Drever 2001; Gammons et al. 2005; Nimick et al. 2011).

Diel variations have serious implications for the collection of water quality data, evaluation of historical water quality data, and collection of baseline water quality data. It is important to consider these short-term variations when designing water quality sampling plans, and it cannot be assumed that a single water sample accurately represents water quality conditions for a given stream. Sampling techniques that effectively address diel variations currently are a topic of research and development. Existing USEPA acute freshwater aquatic-life standards for metals refer to 24-hour average concentrations. As such, it might be useful to consider a sampling program that involves collecting samples throughout the day and night to obtain actual 24-hour average concentrations (see Nimick et al. 2011). Also, consistently collecting periodic samples at a particular site at a specific time of day can minimize the influence of diel variations (Nagorski et al. 2003). However, this approach is problematic for comparisons between sites unless all sites are sampled at the same time under similar hydrologic conditions. An example of misinterpretation

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DIEL BIOGEOCHEMICAL PROCESSES IN STREAMS

David A. Nimick, U.S. Geological Survey

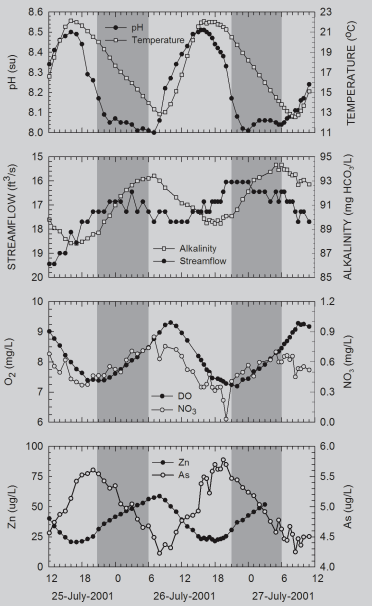
The earth's orbit produces the day–night solar photocycle that is part of our daily lives. Many physical, biological, and chemical processes in streams respond to this diel, or 24-hour, cycle and produce persistent cyclic changes in a host of physical and chemical characteristics. The amplitude of some of these diel cycles can be as large as changes observed on annual time scales. The figure presents one example of the multiple diel cycles that can occur in a stream. Changes in streamflow caused by daily changes in the rates of melting snowpack or riparian evapotranspiration, for example, often follow a diel pattern and can be accompanied by changes in water chemistry. Water temperature also increases and decreases on a daily cycle. The most important and ubiquitous diel biogeochemical processes in streams are photosynthesis and respiration by aquatic organisms. This daily life cycle directly affects the dissolved oxygen concentration, pH, carbonate chemistry, and nutrient content of stream water. Because of the diel pH cycle produced by aquatic life and the diel temperature cycle produced by daily solar warming, the concentration of many trace elements also changes on a daily basis because of pH- and temperature-dependent reactions involving photochemistry, adsorption and desorption, and mineral precipitation and dissolution. The possible magnitudes of these changes are summarized in the table below.

Although recognized for more than 150 years, diel biogeochemical cycling in streams was not studied extensively before the 1990s, perhaps because of the expense and inconvenience of the intensive temporal sampling required. Thus, many monitoring and research plans for streams have been developed without sufficient understanding of the potential impact and importance of diel cycling. A recent review by Nimick et al. (2011) summarizes what is known about diel biogeochemical cycling in streams and the changes in aqueous chemistry that are associated with these processes.

Magnitude of diel cycles for dissolved trace elements in near-neutral to alkaline streams. The source of trace elements in these streams is drainage from historical mines, natural weathering of mineral deposits, or geothermal springs.

Trace Element	Maximum Daily Increase, %	Number of Diel Samplings
As	54	>25
Cd	330	12
Cu (pH = 6.8–7)	140	3
Cu (pH > 7)	<10	12
Methyl Hg	93	2
Mn	306	20
Ni	167	1
Rare earth elements	830	2
Se	<10	1
Zn	800	>35

Source: Modified from Nimick et al. 2011.



Source: Data from Klein et al. 2003.

Diel variation in physical and chemical characteristics at a U.S. Geological Survey gaging station, Prickly Pear Creek, Montana, July 2001. All constituents shown are dissolved.

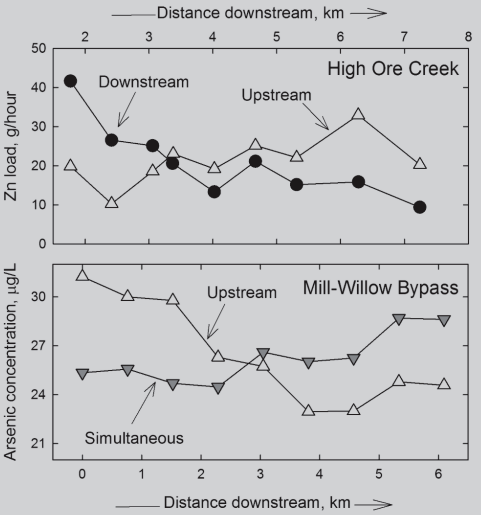
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SYNOPTIC SAMPLING: A MOVING TARGET?

Christopher H. Gammons, Montana Tech of The University of Montana

Synoptic sampling is often used to quantify changes in solute concentration and load within a watershed. An analysis of spatial changes in solute load reveals likely sources of contamination and informs whether a given solute is conservative or reactive. However, this type of analysis is only valid if short-term (e.g., hourly) temporal changes in solute concentration and load are negligible. To minimize changes in concentration due to snowmelt or rain, synoptic studies are often performed in mid- or late-summer, during baseflow conditions. However, this is also the time of year when diel (24-hour) changes in solute concentration are most pronounced. Failure to consider the effects of diel cycling in a synoptic survey could lead to incorrect conclusions about solute behavior, as shown in the following two examples.

The first example (top figure) is from High Ore Creek, a small, alkaline stream draining abandoned mine lands in Montana. The second example (bottom figure) is from the Mill-Willow Bypass, another small stream in Montana with elevated arsenic concentration. In the High Ore Creek study, one sampler began the day at the bottom of the drainage and worked upstream, collecting samples and streamflow measurements each hour. A second sampler did the same tasks in reverse, working downstream. In the Mill-Willow Bypass study, a pair of samplers spent a day working upstream to collect a set of samples. The next day, nine samplers were dropped off at the same stations and each collected a sample at the same time of day (11:00 a.m.).



The implications of the different synoptic trends in the examples shown above illustrate the importance of diel cycles. In the case of High Ore Creek, the data set collected by the downstream sampler would indicate continuous attenuation of Zn load along the lower 6 km (3.7 miles) of the stream. In contrast, the upstream sampler would come to the opposite conclusion (i.e., that there are additional sources of Zn load in the stream). Likewise, in the Mill-Willow Bypass example, the upstream data might suggest that arsenic is attenuating in this stream, whereas the simultaneous data lead one to the opposite conclusion. The reason for the different spatial trends could be very perplexing unless the researcher was familiar with diel cycling. In both streams, concentrations of Zn^{2+} decreased during the day at all sampling stations and increased at night, whereas arsenic, an anionic solute, followed the opposite cycle. In both cases, the assumption of negligible short-term temporal changes in solute concentration was incorrect. To assess the magnitude of this problem, it is recommended that a detailed synoptic study should include one or more sets of 24-hour samples. In some cases, the effects of diel cycles will be minimal or absent, which is good to know.

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of data due to diel cycles is demonstrated by Gammons et al. (2007) (see Synoptic Sampling sidebar).

3.3.6.2 Suspended Sediment

An important consideration in collecting surface-water samples is whether suspended material is to be included as part of the sample or if suspended sediment is a focus of the investigation. Suspended sediment can be an important pathway for the transport of trace elements due to sorption reactions (Smith 1999). If suspended material is included in the sample, then use of proper sampling devices (e.g., an isokinetic, integrating sampler) is recommended (Edwards and Glysson 1999; Nolan et al. 2005; USGS, variously dated). Acoustic sampling devices are increasingly being used to determine suspended sediment loads when chemical data are not required. Horowitz (2008) discusses issues involved in suspended sediment sampling.

3.3.6.3 Discharge Measurements

Stream-water and treated-water flow/discharge measurements can be invaluable when comparing solute concentrations between different sampling events because concentration variations and flow variations are generally linked. Discharge measurements are also a typical requirement for permitting. In mined and mineralized areas, it is common to see relationships between solute concentrations and flow. Predominant causes of changes in hydrologic conditions include evapotranspiration, snow melt, thunderstorms, discharge from treated water, and loss or gain of flow due to infiltration through the stream bed. In their evaluation of historical surface-water quality data for the Red River Valley, New Mexico, Maest et al. (2004) found that the time of year has a major effect on water quality due to changes in hydrologic conditions. They also reported that episodic events, such as thunderstorms, cause flushing of sulfide oxidation products from altered areas, and that alkalinity values decrease during storm events, which indicates that there can be short-term changes in buffering capacity. Johnson and Thornton (1987) and Jambor et al. (2000) also reported elevated metal concentrations in streams draining mined areas due to flushing of stored acid and metals from soils and mining wastes by melting snow and rainfall events. McKnight and Bencala (1990) and Boyer et al. (1997) reported flushing of dissolved organic carbon (DOC) and other constituents from soil during spring snow melt. Therefore, when collecting stream-water samples, it is important to know where stream-water samples fall on the hydrograph. It is good practice to obtain samples from a given stream under a variety of hydrologic conditions.

Streamflow information is critical to computing the load of water constituents, predicting floods, and designing engineering structures and mitigation strategies at mine and processing sites. Load is the product of concentration and stream discharge and hence represents the quantity of an analyte. Depending on the question to be answered or the application, analytes can be reported as concentrations or as loads. Regulatory criteria and toxicological data are generally reported as concentrations. Loads can be used to address questions that involve accumulated mass or volume and treatment efficiency. TMDLs, which are the amount of contaminant that an impaired water body can receive and still meet water quality standards (Walton-Day et al. 2012), are an example of the application of loads.

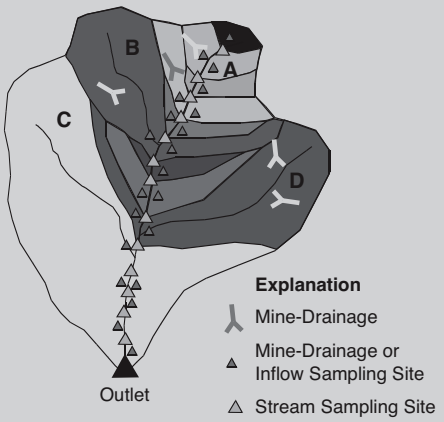
Many techniques can be used to obtain stream-water flow or discharge measurements (e.g., Rantz et al. 1982). USEPA (2001c) provides guidance on performing flow measurements at mine sites. Turnipseed and Sauer (2010) and Kennedy (1984) provide detailed descriptions of discharge measurements, equipment, and calculations. Hydroacoustic meters (e.g., acoustic Doppler current profilers [ADCPs] and acoustic Doppler velocimeters) represent a relatively new approach for determining streamflow. The ADCP is rapidly replacing traditional mechanical

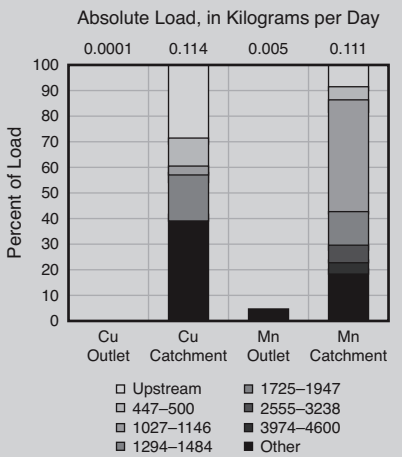
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WHERE'S THE LOAD?

Briant A. Kimball, U.S. Geological Survey

The total maximum daily load (TMDL) process requires an accurate accounting of loading along the course of a stream or river. When a load is determined at the outlet of a catchment (see figure), the loading of individual sources within the catchment is integrated into one sample; no spatial detail is available to compare and quantify loads from the various sources. An approach that combines two methods, synoptic sampling and tracer-dilution discharge, has been used to provide the kind of spatial detail that helps to compare loads for a TMDL process (Walton-Day et al. 2012).





Synoptic sampling within a catchment is accomplished by walking the stream and identifying inflows to the stream. Stream sampling sites are located upstream and downstream from these inflows, or from representative inflows if there are numerous inflows. Stream sites are also located upstream and downstream from areas of likely groundwater or dispersed inflow. In any case, sufficient stream-sampling sites are selected so that spatial detail on the order of 100–300 m (328–984 ft) is achieved. Thus, as suggested in this figure, detailed synoptic sampling divides the catchment into *slices* that each account for a portion of the total load. The same spatial detail is available for measurement of discharge using the continuous tracer injection method described herein (see Appendix 3). With the combined concentration and discharge data, loads can be calculated at each of the stream sites along the catchment study reach (Kimball et al. 2002, 2007). Each slice of the catchment can be compared in terms of constituent loading rather than concentration, providing the frame of reference that is needed for a TMDL evaluation.

Results from a catchment that is affected by several areas of mine drainage are illustrated in the bar graph and compared to the load that would have been measured at the outlet of the catchment. The absolute quantity of the loading, in kilograms per day, is given at the top of each bar. The portion of the load that can be attributed to specific sections, or slices, of the catchment is indicated by the different shades. Those stream segments that contribute the most copper load are not always the same as those that contributed the greatest load of manganese. The load that would have been observed only by a sample at the outlet was negligible for copper and only slightly greater for manganese. This shows the substantial difference in accounting by taking an outlet perspective rather than a detailed catchment assessment. The difference can be attributed to attenuation of loads along the course of the stream as metals are removed by physical and biogeochemical processes. However, almost all of the load that is removed by attenuation is subsequently flushed by snow-melt runoff and still contributes to the loading on an annual basis. This seasonal variation of load from the catchment should be quantified by sampling over time at the outlet, and the slices of the total should be quantified by a detailed catchment approach.

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current meters for streamflow measurements (Muste et al. 2007; Oberg and Mueller 2007). Treated water discharges are generally monitored using magnetic flowmeters.

Use of tracers in surface water is an effective approach to determine gain or loss of water quantity. An approach using a tracer-dilution method to quantify stream-water discharge and synoptic sampling to quantify solute concentrations has been developed to determine metal loading in streams located in mined and mineralized areas (see Where's the Load sidebar, Appendix 3, and case study by Walton-Day et al. in Appendix 5; Kilpatrick and Cobb 1985; Kimball 1997; Kimball et al. 2007, 2010). Using this approach, it is possible to identify and quantify the principal inflows of metals, the presence of nonpoint source, dispersed inflows of metals, and the amount of natural attenuation of metals in a stream. Tracer-dilution methods can also be used together with other tools, such as isotopic measurements, to identify sources and attenuation processes (e.g., Borrok et al. 2009).

3.3.6.4 Modeling Considerations

Geochemical, toxicological, and hydrogeochemical modeling requirements need to be considered when preparing a sampling and analysis plan. Modeling hydrogeochemical processes using a variety of numerical models is an expanding area of research and development. Both numerical and water mass-balance models can be used to refine conceptual models prior to mine startup, evaluate treatment and mitigation options, manage water on the site during operation, and guide selection of appropriate reclamation options during mine operation and closure. Voss and Letient (2006) and Eary et al. (2009) have demonstrated how existing numerical models can be reconfigured and used as decision support tools at mine sites.

For geochemical modeling, it is necessary to have complete dissolved water analyses including major, minor, and trace elements, and both anions and cations. Additional important determinations include pH, temperature, iron speciation, and direct measurement of species for elements of concern. Also, specific conductance, alkalinity/acidity, DO, redox conditions, and total dissolved solids can provide useful information. Current analytical techniques offer simultaneous analysis of a number of chemical elements; therefore, it is relatively simple to obtain complete water analyses. Also, complete water analyses can serve as a guide to unanticipated environmental concerns or to potential by-products that may be recovered. Nordstrom and Munoz (2006) discuss information needs for various applications of geochemical modeling. For transport modeling, it is necessary to have water-flow or discharge measurements. For inverse modeling, it is necessary to have mineralogical information about the geologic material in contact with the water. Caruso et al. (2008) discuss recent advances in metals fate and transport modeling. Geochemical modeling is the topic of another handbook in this series (see Nordstrom et al., forthcoming) and is also discussed in Alpers and Nordstrom (1999) and Nordstrom (2004). Butler (Appendix 5 of this handbook) provides a case study related to sampling and geochemical modeling.

Toxicological considerations are also an important aspect of water quality studies. Stillings et al. (Appendix 5, this handbook) provide a case study about selenium cycling and bioaccumulation at Idaho phosphate mines. Toxicological modeling of water quality is beginning to have a presence in federal regulations. For example, USEPA incorporated the biotic ligand model (BLM) into its recently revised aquatic life ambient freshwater quality criteria for copper (USEPA 2007a). The BLM is a computational model that predicts metal toxicity to biota in aqueous systems for one metal/organism pair at a time (Di Toro et al. 2001; Santore et al. 2001; Appendix 3). Required input for the BLM includes temperature, pH, DOC, alkalinity, and dissolved concentrations for calcium, magnesium, sodium, potassium, sulfate, and chloride. Although DOC samples are rarely collected, DOC is a required input parameter for the BLM and can have a significant influence

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on predicted toxicity of some metals such as copper (e.g., Smith et al. 2006, 2009). Therefore, it is important to include DOC in surface-water sampling plans. A research version of the BLM (see Section 3.13) includes computations for copper, silver, cadmium, and zinc. In addition to determining site-specific water quality criteria, the BLM can be used for additional applications such as determining the time of year when the water chemistry is most toxic, which is often difficult to determine without a rigorous and expensive toxicity-testing program.

3.3.7 Groundwater Samples

There are many reasons for sampling groundwater, such as tracking a contaminant plume, determining the existence or extent of contamination, compliance monitoring, or performing water-supply measurements. Groundwater sampling can be conducted during any of the phases of mining depending on the objectives of the investigation. Preserving the integrity of groundwater samples is not an easy task. LeGrand and Rosen (1998, 2000) called for a widespread change in mindset for groundwater contamination investigations. They listed three problematic philosophies and procedures used in groundwater contamination studies, which include (1) almost exclusive reliance on quantitative, mathematical, precision-oriented data; (2) an incomplete or belated conceptual model of hydrogeologic characteristics; and (3) insufficient use of existing databases and other available information. They encouraged developing an early *prior conceptual model explanation* that is largely derived from existing information to conduct a cost–benefit evaluation of necessary groundwater protective actions.

General Web sites for online resources related to groundwater are listed in Section 3.13. Several methods for groundwater sampling are documented by ASTM International and USGS (variously dated). Some general groundwater references include Freeze and Cherry (1979), Barcelona et al. (1985), USEPA (1991), Chapelle (1993), Fetter (2000), Alley et al. (2002), Appelo and Postma (2005), Nielsen (2006), Nielsen and Nielsen (2007), Younger (2007), and ASTM (2007).

Significant advances in groundwater sampling include addressing colloid formation and sampling (e.g., McCarthy and Zachara 1989; Backhus et al. 1993; Gschwend and Reynolds 1987; Kearl et al. 1992), low-flow sampling, and aquifer heterogeneity with emphasis on small-scale differences. The potential of hydrochemical communication between aquifers is another important consideration. As with other sampling media, it is critical to determine the sampling objectives and data applications prior to locating and designing groundwater wells and sampling groundwater. An important question for groundwater sampling is if it is necessary to focus on a particular portion of the formation because information about small-scale spatial differences in groundwater quality requires planning and specialized sampling techniques. Locating wells with respect to site geology and hydrology are also important considerations. Surface geophysical techniques can provide information to aid in properly locating well sites. Some geophysical techniques can be used to map subsurface plumes and locate sources of acidity (see discussion of geophysical methods in Appendix 3).

Filtration is a controversial issue for groundwater samples (e.g., Puls and Barcelona 1989; Puls et al. 1990). Puls and Powell (1992) have reported that colloids as large as 2 μm can be mobile in porous media. Federal and state regulations vary on this issue, and some analytical procedures require that a sample be filtered. If filtration is used, it is preferable to filter in the field using in-line filtration, especially for unstable samples (e.g., anoxic groundwater). It is good practice to collect unfiltered samples in addition to filtered samples.

Tracers and isotopes in groundwater are generally used to characterize groundwater flow and to identify potential contaminant pathways (e.g., Wolkersdorfer 2002; Manning et al. 2008;

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Walton-Day and Poeter 2009; see Appendix 3). An overview of tracers is given by Healy and Scanlon (2010).

3.3.7.1 Representative Groundwater Samples

It is very difficult to obtain a groundwater sample that is representative of the formation water. Several researchers have addressed the difficulty of obtaining representative groundwater samples (e.g., Parker 1994; Puls et al. 1992). Nielsen and Nielsen (2007) list the following factors that influence the representative nature of groundwater samples:

- Formation and well hydraulics
- Groundwater flow paths
- Flow-through wells and well screens
- Hydraulics within a well between sampling events
- Sampling point placement and well design, installation, and maintenance
- Placement of sampling point with respect to contamination source(s)
- Placement of sampling point intake in the zone of highest hydraulic conductivity
- Drilling and installation methods
- Suitability of design with regard to material selection, diameter, depth, screen length, and screen slot size
- Construction methods including placement of annular seal materials and filter pack materials
- Method, timing, and duration of sampling point development
- Long-term maintenance of the sampling point (e.g., compromises in well integrity)

3.3.7.2 Well Design and Purging Methods

Proper well design is an important consideration for groundwater sampling. The objectives of the investigation should dictate the well's design. For example, it is generally preferable that the screen interval only encompass the sampling zone of interest so that mixing of different water sources does not occur in the well bore (e.g., Church and Granato 1996). Nested wells can be used to allow sampling of multiple depths at the same location.

Purging and collection methods have a significant impact on the sample quality and integrity. Many traditional groundwater sampling procedures were designed for water supply investigations and are not suitable for contaminant-related investigations. Depending on the study objectives, it can be helpful to probe the water column before pumping to identify the presence and degree of stratification, and to geochemically characterize different zones. Traditional purging procedures require that three to five well-casing volumes be purged to remove stagnant water from the well prior to sampling. This purging is frequently done using high-speed pumping rates or with a bailer. This practice can be problematic for water quality sampling because rapid removal of water can lead to increased turbidity (producing particulates in the water sample; Kearl et al. 1994), disruption of well packing materials (producing contamination), and drawing water into the well that is not representative of the formation water adjacent to the well. Puls and Powell (1992) have reported that sample-collection practices that induce artificially high levels of turbidity have the greatest negative impact on sample quality. Filtration should never be considered as a remedy for improperly collected groundwater samples. Fracture porosity in the formation can also be very important because extensive pumping can draw water from a large volume of the formation. For

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example, McCarthy and Shevenell (1998) reported that pumping 10 L of water from a well in rock with fracture porosity could conceivably draw water from a volume of 100 m^3 of rock.

Recent reports suggest that it is important to keep subsurface disturbance and sampling handling to a minimum when collecting groundwater quality samples. Hence, it is important to use appropriate purging and sampling equipment when collecting groundwater samples. Low-flow (low-stress), minimum drawdown purging and sampling techniques are being recommended for most groundwater quality sampling (Puls and Barcelona 1996; USGS, variously dated; Ritchey 2002; Yeskis and Zavala 2002; ASTM 2005). Although time-consuming, these techniques reduce the need to dispose of large volumes of purged water, minimize sampling artifacts, reduce stress on the formation, and result in better consistency of groundwater samples (e.g., Sevee et al. 2000). The flow rate is site specific and this approach strives to cause minimal or no drawdown in the formation adjacent to the well. During the purging process, several indicator parameters (e.g., pH, temperature, specific conductance, turbidity, DO, redox potential) need to be monitored to determine when they stabilize (e.g., Wilkin et al. 2001; see USGS, variously dated, for parameter stabilization and calibration guidelines). Barcelona et al. (2005) have concluded that stabilization of field water-quality parameters is an effective indicator of when to collect samples. If possible, it is best to use dedicated downhole sampling devices.

3.4 SOLID SAMPLING CONSIDERATIONS

The purpose of collecting solid samples at mine and processing sites (other than for exploration purposes) is normally to quantify metal content at the sampling location. Additional reasons can include establishing baseline conditions, examining variation in lithologies, assessing the potential for acid generation, assessing the potential for contaminant release, locating contaminant source(s), providing input for modeling efforts, or meeting regulatory requirements. Samples collected for metallurgical purposes and assays can often be useful for environmental purposes.

3.4.1 Mining- and Metallurgy-Related Samples

When sampling to determine potential for acid generation or contaminant release, it is good practice to classify solids into geological categories (e.g., soil, overburden, waste rock, ore, sub-ore, tailings, grab, chip, composite; see Appendix 3 for discussion of various solid materials). Environment Canada (2009) outlines site-specific programs for predicting wastewater quality throughout the mine life cycle, and Miranda et al. (2005) discuss the importance of minimizing ARD and suggest that testing for ARD potential be done as a part of an environmental impact assessment in advance of mine development.

Runnells et al. (1997) provide a methodology for sampling mine waste rock and process tailings. They state, "Briefly, the method is based on the use of a statistical approach to determine, illustrate, and defend the adequacy of the sampling. [We do] not believe that there is a *correct* number of samples for characterizing a facility. That is, there is no general rule that can (or should) be followed, such as a given number of samples per ton of tailings, per acre of impoundment, or per foot of drillcore. Each facility is different, and the adequacy of sampling must be tailored to the facility." Price (2009) provides guidance about geological characterization at mine sites and the use of block models for environmental applications. He states, "The recommendation here and previously is that the final sampling frequency be determined site specifically based on the variability of analysis results for critical parameters, prediction objectives and required accuracy" (Price 2009). Hence, site-specific conditions are critical in determining what and how much to sample at mining sites (see the following three sidebars about mine rock sampling). McLemore provides a case study that addresses mine-rock sampling (Appendix 5, this handbook).

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WASTE ROCK SAMPLING

Patsy B. Moran, Arcadis U.S., Inc.

Waste rock geochemical characterization is primarily conducted to assess the potential for acid rock drainage and metal leaching as a result of excavation and subsequent exposure to oxygen and water. The objectives of geochemical characterization will change as mining projects advance from exploration to postclosure. Early-stage characterization is often carried out to assist with design of facilities and development of management plans. Operational programs frequently focus on meeting permit requirements, and postclosure characterization may be needed if drainage chemistry deviates from predictions. Regardless of the project phase, selection of representative samples plays a critical role in the effectiveness of any characterization program.

Although waste rock sample selection should be conducted on a site-specific basis due to inherent site and project variability, several factors should be considered for each project, including selection of

- Discrete samples from the primary rock types based on geologic or other operationally defined units,
- Geospatially distributed samples from throughout the area to be mined, and
- Enough samples to adequately characterize the range of acid–base characteristics for each rock and alteration type with focus on those most likely to be problematic.

Arguably, the most difficult of these items to address is determining how many samples will be needed to meet project objectives. Some important considerations include

- Heterogeneity of the rock types,
- Quantity of disturbed rock (e.g., tons, wall rock area),
- Desired level of confidence, and
- Project phase.

Several recent, publically available documents have detailed guidance for properly conducting waste rock sampling:

- *Prediction Manual for Drainage Chemistry from Sulphidic Geologic Materials* (Price 2009)
- The Web-based *Global Acid Rock Drainage Guide* (GARD Guide; INAP 2009)

Both documents also provide in-depth information to assist with development of waste-rock characterization programs and drainage-chemistry predictions.

The mine geologic model and block model are powerful tools that may be used for several purposes, including to identify target populations, assign environmental properties to various units, and discretize waste rock temporally and spatially that can inform material handling plans based on geochemical characteristics. Modern mine planning software packages (e.g., MineSight [Mintec, Tucson, Arizona], GEOVIA [Dassault Systèmes, Vancouver, British Columbia, Canada], Vulcan [Maptek Pty Ltd, Adelaide, Australia]) allow detailed three-dimensional definition of not only ore properties, but also geochemical data that can be applied to mine-waste management during operations and for waste rock placement as part of operation and closure plans. The excavation of the entire deposit, all ore and waste rock, can be simulated and waste-rock placement plans can be evaluated in advance of actual production to ascertain various waste-rock handling and placement strategies.

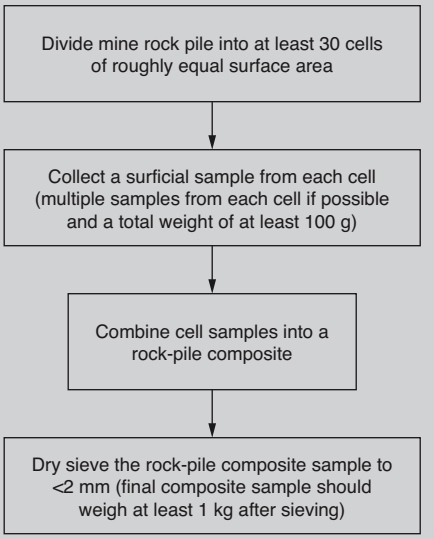
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SAMPLING SURFICIAL MATERIAL FROM MINING WASTE-ROCK PILES

Kathleen S. Smith, U.S. Geological Survey

Site characterization of mining wastes requires collection of representative samples of the mine-waste material. This is generally difficult because of the compositional and spatial heterogeneity of the waste material. A statistically based strategy was developed for sampling the surficial material of mine-waste material for use in screening and prioritizing historical waste piles (Smith et al. 2000).

The target population is defined as the upper 15 cm of a waste-rock pile. To minimize sampling errors, a composite sample of at least 30 increments (subsamples) was collected, which reduces the grouping and segregation error (GSE). To minimize the fundamental sampling error (FSE), the sample is sieved to a <2 mm size fraction. For an FSE below 2%, it is necessary to collect at least 1 kg for the final sieved composite sample. This combination of maximum particle size and 2% fundamental error keeps the total sample mass to a weight that can be easily carried in a backpack after sample collection. Samples are collected with a stainless-steel trowel, and each increment is successively placed in a plastic bucket. If samples are collected wet, they are air-dried and efforts made to break up clods prior to sieving.



In terms of average properties, one 30-increment composite sample collected using this sampling strategy contains as much information relative to average value as 30 individual grab samples at $\frac{1}{30}$ of the analytical cost. This sampling strategy has been used in conjunction with the U.S. Geological Survey field leach test (Hageman 2007) to characterize mine-waste material (e.g., Hageman and Briggs 2000) and to provide a comparison between the leaching characteristics of waste-rock piles and naturally altered areas (Smith et al. 2007). It can also be used for other target populations, such as lithologic units and soils, and has been used to collect soil, sediment, and ash samples from natural disasters (e.g., Plumlee 2009).

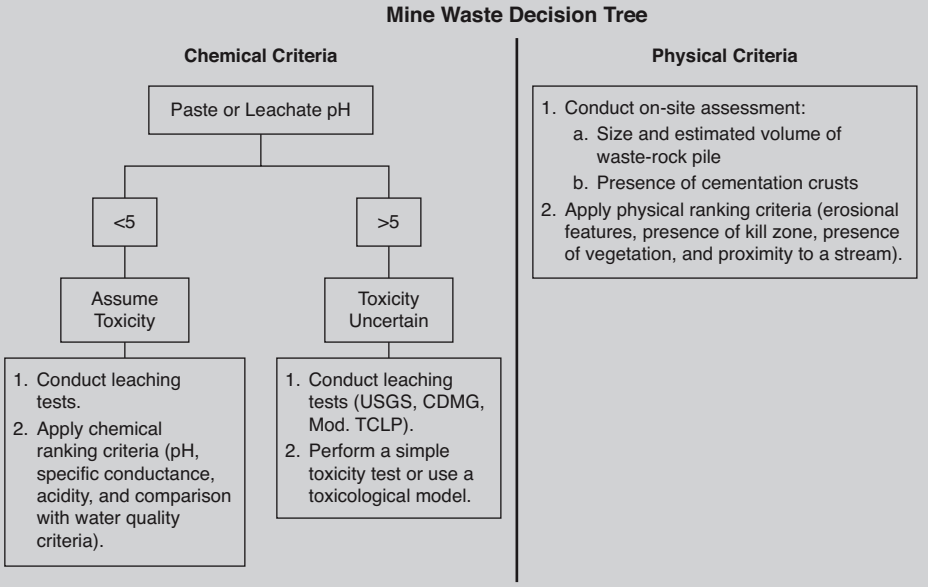
It is advisable to sample all lithologies and to determine the degree and range of alteration, acid-generating potential, mineralogy, and bulk chemistry (major, minor, and trace elements). Two big issues are common in sampling mine-waste facilities: (1) the typically very heterogeneous nature (both compositionally and grain size), which makes it difficult to obtain a representative sample, and (2) homogenization and subsampling of the material during sample preparation for analysis. These issues are discussed in more detail in Sections 3.4.8 and 3.4.9, and in some of the case studies in Appendix 5. Bulk chemistry is important for ore and waste materials, ore stockpiles, ultimate pit surfaces, and amendment materials blended with wastes because it allows identification of potentially problematic constituents and pinpoints which zone(s) of materials require further testing. Acid–base accounting (ABA) methods and kinetic testing are discussed in the prediction volume of this series (Williams and Diehl 2014) and will not be addressed here.

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A QUICK AND DIRTY ASSESSMENT

Thomas R. Wildeman, Colorado School of Mines

In Colorado there are thousands of orphaned mine-waste piles. The question is, how can one assess these piles in a cost-efficient manner to find out which are the most harmful to the environment? For more than a decade, scientists at the U.S. Geological Survey (USGS) and the Colorado School of Mines have been considering this problem and have developed a fairly simple scheme to assess potential impacts from a waste pile (Ranville et al. 2006; Wildeman et al. 2007). Two features are at the heart of the scheme. The first is the development of a simple method for obtaining a multi-increment sample that is based on sound sampling procedures and statistical rigor (Smith et al. 2000). The second feature is the development of the mine waste decision tree (MWDT), shown herein.



Source: Adapted from Ranville et al. 2006 and Wildeman et al. 2007.

The MWDT involves three interesting features. First, there is a separate chemical and physical assessment. The chemical criteria rate the availability of contaminants, and the physical criteria rate the ability to deliver the contaminants. The second feature is that if the paste pH of the composite sample taken from the pile is below 5, then the decision is to assume toxicity to the aquatic environment. This assumption is based on geochemical principles and on experiences of sampling and assessing hundreds of waste piles in the Rocky Mountain region. The third feature is three simple leaching tests that have been developed to assess the actual contaminants: The USGS field leach test (FLT; Hageman 2007), the Colorado Division of Minerals and Geology (CDMG) test, and a modified toxicity characteristic leaching procedure (Mod. TCLP) test. These tests are modified versions of standard regulatory tests, so they are primarily suitable for assessment projects. However, they are simple and inexpensive, and the FLT and CDMG tests can be performed on-site. The procedures for securing a composite sample and the leaching tests are robust enough that they can be used whenever a quick and dirty assessment needs to be done. They also have been used effectively to assess the rubble from the 9/11 disaster and the sediment left from the flooding caused by Hurricane Katrina.

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3.4.2 Soil Samples

Numerous references involve soil characterization and soil sampling (e.g., Mason 1992; USEPA 1995; Sparks 2003; Carter and Gregorich 2008). The Soil Science Society of America published a five-volume series titled Methods of Soil Analysis that covers physical and mineralogical methods, microbiological and biochemical properties, and chemical methods. USEPA considers soil to be an exposure pathway rather than a migration pathway because contact between contaminated soil and a receptor is initiated by the receptor (USEPA 1995). USEPA soil screening guidance (see Section 3.13) provides a framework for developing risk-based, soil screening levels (SSLs) for protection of human health. The SSLs do not represent national cleanup levels; they are intended to be used by site managers to eliminate areas, pathways, and/or chemicals of concern at National Priorities List sites.

Soil consists of heterogeneous mixtures of solid particles (inorganic and organic), microorganisms, air, and water. Because soil is so chemically, physically, and biologically complex, it is important to carefully define the target population and to ensure that it is consistent with the sampling objectives and methods. Soil samples need to be dried immediately after collection at ambient or low heat, and loosely packed into the sample container. For samples that contain jarosite, Desborough et al. (2010) report that the common practice of heating above approximately 100°C (212°F) during *drying* can change the structural nature of jarosite minerals. In situations where there are hydrous sulfate salts, it is preferable to freeze the samples or cores.

Soil sampling and analysis techniques can be applied to investigating the weathering, erosion, and stability of mine waste rock piles, and several of these techniques are described in Appendices 2 and 3. McLemore et al. (2009a) discuss results from a series of investigations at the Questa mine in New Mexico, where they employed a variety of physical, geochemical, petrographic, and mineralogical techniques to evaluate the stability of rock piles.

3.4.3 Sediment Samples

Many publications provide guidance for collection of sediment samples (e.g., Truckenbrodt and Einax 1995; Edwards and Glysson 1999) and suspended sediment (e.g., Nolan et al. 2005). Brandvold and McLemore (Appendix 5, this handbook) provide a case study that describes aspects of sediment sampling adjacent to mining sites. The approach for sampling sediment depends on the objectives and elements of concern. Studies of the distribution of trace elements in relation to size fraction generally show that most trace metals (e.g., copper, iron, manganese, and zinc) are concentrated in the finest fractions of the sediment. Typically the <150-μm size fraction is collected (by wet sieving) and analyzed. However, the possibility that some elements of interest can be preferentially bound to coarser, sandy material in the vicinity of a mine site also needs to be considered and investigated. Vertical profiles can provide useful information about metal residence and mobility (e.g., Church et al. 2007a). In collecting sediment samples for acid-volatile sulfide/simultaneously extracted metals (AVS/SEM; see Appendix 3) or other solid samples from reducing environments, care should be taken to protect samples from oxidation by enclosing them in sealed containers (under nitrogen if possible) or, better yet, to collect them under nitrogen followed by freezing. Equilibrium partitioning sediment benchmarks for metals are sediment-quality guidelines developed by USEPA that relate chemical concentrations in sediment to potential biological effects on benthic organisms (USEPA 2005; see Appendix 3 and case study in Appendix 5 by Seal et al.).

If sediment samples are not collected from reducing environments or being used to determine AVS/SEM, then wet samples need to be immediately air-dried, or dried at temperatures less than 40°C (104°F), to avoid changing the clay, iron, and sulfate mineralogy.

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3.4.4 Bulk Composition and Geoavailability

Bulk composition of solid material can be analyzed in the field by portable X-ray fluorescence (PXRF) or by standard wet chemical methods (see Appendix 3). The PXRF apparatus offers rapid in situ semiquantitative analysis; wet chemical analysis offers potentially greater accuracy but is a relatively slow and more expensive process. PXRF allows detailed mapping of contamination hot spots (based on bulk composition), and the perceived relative lack of accuracy is outweighed by the large number and wide range of samples that can be analyzed. Field leaching tests, such as the USGS field leach test (Hageman 2007), coupled with field test kits can be used to ascertain readily soluble (i.e., geoavailable; see Smith and Huyck 1999; Smith 2007) constituents at the field site (see Appendix 3 and Figure 4.4).

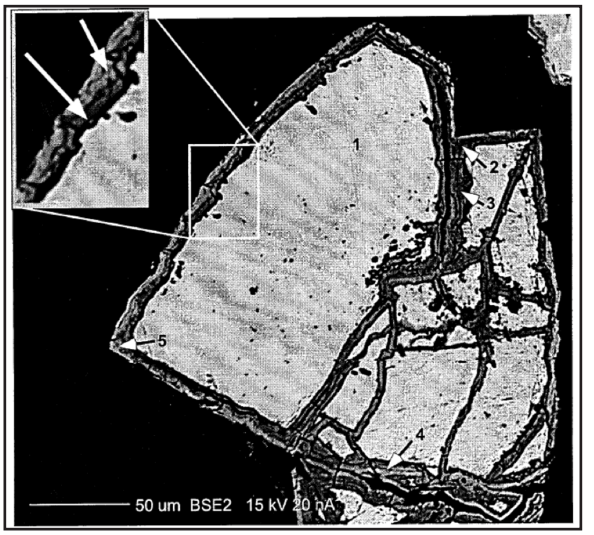
The transport and fate of trace metals are generally controlled by their partitioning between aqueous and solid phases. Understanding the partitioning mechanisms of these metals is critical for evaluating and predicting their mobility and bioavailability (Smith 2007). Organic constituents, iron and aluminum oxyhydroxides, and manganese oxides are generally the most important sinks for trace metals due to sorption reactions (Smith 1999). Generally, these phases occur as coatings on other mineral surfaces, which makes a molecular level of understanding difficult (e.g., Diehl et al. 2006).

The importance of mineral surfaces to reactivity and mobility of metals in the environment is increasingly better understood (Sparks 2005; Riviere and Myhra 2009). Fundamental understanding of minerals and their reactions in natural systems requires examination at the molecular and atomic scale. Hochella (1995) provides a review of some surface characterization techniques, and Fenter et al. (2002) provide an overview of the theory and applications of synchrotron radiation to the analysis of soil surfaces. Surface analytical techniques include a variety of spectroscopic methods that have been used in geological and environmental applications to examine the solid–water interface and surface speciation of elements. X-ray absorption spectroscopy (XAS; Kelly et al. 2008), including X-ray absorption near-edge structure (XANES; e.g., Foster et al. 1998; Fleet 2005; Walker et al. 2005) and extended X-ray absorption fine structure (EXAFS; e.g., Kim et al. 2004), is valuable because it can provide information about a particular element and its speciation and oxidation state, which is particularly useful for amorphous materials and metal-sorption studies (the techniques are discussed in more detail in Appendix 3). More specialized methods are being developed, and applications to earth materials will continue to grow.

3.4.5 Mineralogical and Petrographic Characterization

Mineralogical characterization can provide fundamental information regarding the potential for acid generation, neutralization, and contaminant release, yet it has historically been neglected. Mineralogy and texture are key factors that influence acid generation, neutralization, and metal release (e.g., Kwong 1993; Cabri and Vaughan 1998; Plumlee et al. 1999; Vaughan and Wogelius 2000; Munroe et al. 2000; Diehl et al. 2006; Shaw and Mills 2012). Kirk (Appendix 5) provides a case study that describes the use of historical mineralogical data combined with laboratory- and field-scale monitoring data to predict postmining water quality. Mineralogical studies are performed to determine primary and secondary mineralogy, alteration information, and identification of sulfide minerals, carbonate minerals, and other acid-neutralizing minerals (e.g., biotite; e.g., Neubert 2000; Hammarstrom and Smith 2002). Pitard (2010) recommends a thorough mineralogical or microscopic study at the early stage of a sampling project because it is important to understand how a given trace constituent behaves in the material to be sampled. In particular, textural information such as degree of particle/phase liberation, phase association, and particle size and morphology can provide important information about the potential behavior of

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Source: Munroe et al. 2000.

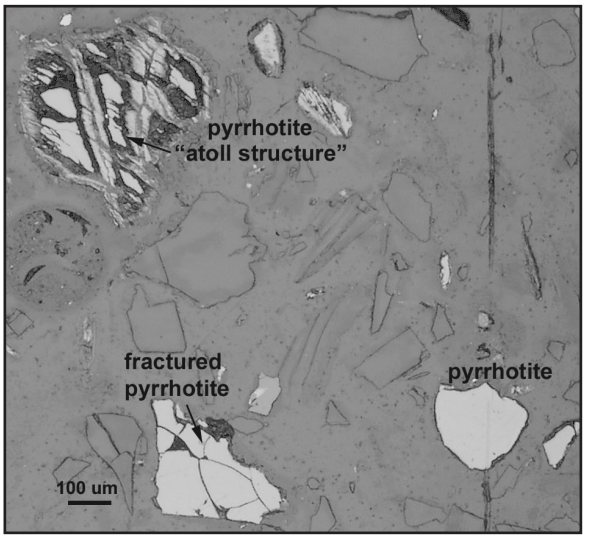
FIGURE 3.3 Backscattered electron microprobe image of an oxidized pyrite grain with Fe-hydroxide and Fe-sulfate alteration rinds of low and high atomic mean number (sample is from Laramide polymetallic veins). Brecciated parts of this grain are interpreted to have undergone hydrothermal fracturing and subsequent alteration along fractures and cementation by iron oxides.

constituents. A variety of techniques can be used for mineralogical, compositional, morphological, and textural characterization. Some traditional techniques include optical microscopy (e.g., Kerr 1977; Craig and Vaughan 1994; Nesse 2004), x-ray diffraction (XRD; e.g., Nuffield 1966; Bish and Post 1989), and electron microbeam methods (e.g., White 1985), including scanning electron microscopy (SEM) and electron-probe microanalysis (EPMA; e.g., Reed 1996; McGee and Klaus 2001; see Appendix 3).

Petrographic analysis provides information on the texture of the material and the relationship of the minerals to each other. Petrographic analysis of rocks has traditionally been performed with reflected and transmitted light microscopy using thin sections and point counting to provide a modal mineralogy, percent porosity, and grain size (van der Plas and Tobi 1965; Delvigne 1998; McLemore et al. 2009b). However, this method typically does not provide accurate and reproducible mineral proportions for sedimentary, volcanic, and soil-like material because lithic fragments, groundmass, and matrix are typically identified as separate phases, not as specific minerals. In addition, groundmass phases in volcanic rocks can be too fine grained to identify individually, and making thin sections of intact soil-like material is difficult because much of the material is not well cemented. Petrographic analysis is also useful to determine residual sulfide minerals and residence phases of trace elements in slags (e.g., Piatak et al. 2004; see case study by Piatak et al. in Appendix 5).

Optical microscopy, EPMA, and SEM can provide valuable information about the morphology and texture of the material. For example, microanalytical studies can determine the morphology of pyrite (whether it is chemically zoned, coarse grained, fine grained, or framboidal) and thereby identify forms that are more accessible to oxidation. They can also determine if pyrite is encased by gangue minerals, such as quartz or clay, and consequently less accessible to oxidation. Iron oxidation rims surrounding minerals can protect the mineral grains from subsequent oxidation (see Figure 3.3; Munroe 1999; Munroe et al. 1999, 2000). Spot analysis

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Courtesy of Sharon F. Diehl, U.S. Geological Survey.

FIGURE 3.4 Backscattered electron image of pyrrhotite grains in a tailings deposit, Elizabeth mine, Vermont. The pyrrhotite grains have undergone varying degrees of weathering, which could be due to trace-element content or differing lengths of exposure in the tailings. Atoll structure develops through the partial dissolution of the sulfide grain (Jambor 2003) and alteration to secondary iron minerals, which leaves residual islands of pyrrhotite.

and element-intensity maps by SEM or EPMA identify trace elements associated with the main mineral phase and characterize their residence (i.e., whether metal impurities are homogeneously distributed within the mineral or are associated with mineral inclusions). This information can be very useful in identifying the source of contaminants in water samples (e.g., Diehl et al. 2006, 2008). Incorporating mineralogical characterization into predictive testing and environmental impact assessments can help define potential environmental challenges, which allows for better ore stockpile and waste repository design as well as estimates of remediation and mitigation costs, if necessary.

Sulfide minerals such as pyrite, marcasite, and pyrrhotite, which are the prime generators of ARD, are commonly hosts to elements of concern such as arsenic, cobalt, copper, nickel, lead, selenium, or zinc. Trace metals change the physical properties of minerals, commonly increasing reactivity and solubility by lattice distortion during element substitution. Figure 3.4 is a backscattered electron image of pyrrhotite grains in a tailings deposit from the Elizabeth mine in Vermont. The important observations to note are that the pyrrhotite grains show varying degrees of weathering, ranging from fairly pristine grains to fractured grains, and that fractures allow ingress of oxygen and fluid that partially dissolve pyrrhotite grains, which results in alteration of the rims to marcasite and iron-oxide minerals. These sulfide minerals will continue to slowly weather, and they remain in the tailings deposit as a repository for future acid drainage (AD) and metal release.

It is important to quantify mineral phases that influence the composition of effluents from drainage of ore and waste materials. Not all sulfide minerals have the potential to produce acidic drainage, and iron-sulfide minerals may not produce acidic drainage in some materials. For example, galena, sphalerite, covellite, bornite, and chalcocite do not release acid when oxidized (Plumlee et al. 1999). Conversely, minerals other than sulfides can generate acidic drainage. For example, some forms of jarosite (e.g., Lapakko and Berndt 2003; Desborough et al. 2010) and

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soluble sulfate salts (e.g., Cravotta 1994; Jambor et al. 2000) release acidity when in contact with water. Identifying neutralizing minerals, such as carbonates and clay minerals, is an important step in assessing the neutralizing potential of the material (e.g., Jambor et al. 2002, 2007).

Determining quantitative mineral abundance by whole-rock XRD analysis is occasionally difficult because of the effects of mineral crystallinity (i.e., acidic waste materials generally consist of a high proportion of amorphous material), preferential orientation in the sample mount, differential absorption of X-rays, and overlapping peaks by different minerals, which all affect the diffractogram patterns and make their interpretation difficult. Some minerals, such as hematite and other iron oxides, have poor crystallinity and are not always detected by XRD. The Rietveld method attempts to overcome some of these issues (Bish and Howard 1988; Bish and Post 1993; Young 1993; Oerter et al. 2007). Oerter et al. (2007) describe a method for determining pyrite concentration by using a modified Rietveld method on heavy mineral separates. Clay minerals are best identified by XRD or EPMA (Moore and Reynolds 1989; Hall 2004).

Another approach that can be used to determine quantitative mineralogy is to calculate a normative mineralogy from the whole-rock chemical composition. A normative mineralogy is a set of idealized minerals that are calculated from a whole-rock chemical analysis; not all calculated minerals are always actually present in the sample (Neuendorf et al. 2005). Normative mineralogy is usually calculated for volcanic rocks that have glass and/or very small crystals, for which it is difficult to optically determine a modal mineralogy. A normative mineralogy is also calculated for altered or metamorphosed igneous rocks that no longer have the original igneous mineralogy. One of the most widely used normative calculations is the CIPW (Cross, Iddings, Pirsson, and Washington) norm. Another normative method involves the combination of XRD and whole-rock chemical analysis and attempts to provide better quantitative mineralogy (e.g., SedNorm; Cohen and Ward 1991; Ward et al. 1999). Combustion-infrared analysis for carbon and sulfur phases following various sample treatments is often useful in interpreting XRD patterns, due to greater sensitivity.

ModAn is a normative calculation that estimates modes "by applying Gaussian elimination and multiple linear regression techniques to simultaneous mass balance equations" (Paktunc 1998, 2001) and allows location-specific mineral compositions to be used. In a study at the Questa mine in New Mexico, representative mineral compositions for mineral samples were determined from EPMA and used in ModAn (McLemore et al. 2009b). A feature of ModAn that makes it appropriate for environmental studies is that it can model one of several sulfide minerals.

Automated SEM-based quantitative mineralogical analysis (e.g., MLA, QEMSCAN) is another method that quantifies mineralogical parameters such as modal abundance, mineral assemblages, texture (e.g., structural alignments), microfractures, microveins, and porosity in solid samples (Pirrie et al. 2004, 2009; Hoal et al. 2009a, 2009b). It is commonly used for metallurgical, mineral processing, and geometallurgical applications and shows promise for mineral-environmental applications. For example, it can be used as a complement to ABA and humidity cell testing. The method is based on an integrated system that includes a scanning electron microscope and proprietary software, and enables quantitative chemical analysis of materials and generation of high-resolution mineral or element maps and images (see Hagni 2008; Hoal et al. 2009b; Pirrie and Rollinson 2011; Smith et al. 2013; see Appendix 3 for additional information). This automated method produces statistically representative data sets due to the large number of high-speed determinations (approximately 150 per second).

Other techniques have been developed that can provide petrographic, chemical, and mineralogical information on ore, mine wastes, and other solid materials. For example, laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) can provide spatial information

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about metal concentrations in a solid matrix (e.g., Diehl et al. 2007, 2010). Data from LA-ICP-MS line scans can also highlight correlations between elements. For example, overlapping peaks of cobalt and nickel with iron may indicate that these two metals are coprecipitated within the same iron-sulfide mineral. Another example is an approach developed by Jambor and Blowes (1998), wherein XRD data are compared with petrographic observations to make mineralogical interpretations. Parbhakar et al. (2009) also developed a mineralogical ARD index that takes into account diameter/size, degree of alteration, and morphology of the acid-generating phase, content of neutralizing phases, and spatial relationship between acid-generating and acid-neutralizing phases.

Regional mapping of mineralogy can be accomplished using remote-sensing techniques coupled with field mapping (e.g., Bove et al. 2007a, 2007b; Rockwell 2011, 2013; Yager et al. 2013). Remote-sensing techniques can be very useful in prioritizing ARD cleanup in large areas by identifying iron- and aluminum-rich minerals that have specific pH-stability ranges (e.g., Swayze et al. 2000; Hauff et al. 2006). Although ground-truthing is generally necessary to verify remote-sensing data, the ability of these methods to assess large areas can be quite effective and can result in significant cost savings when compared with the costs of on-the-ground sample collection and characterization at a regional scale. However, these remote-sensing techniques only work in exposed areas because vegetation masks the measurements. Appendix 3 presents summaries of several remote-sensing techniques. A variety of regional- and national-scale geospatial data are available at the USGS Mineral Resources On-Line Spatial Data Web site (see Section 3.13).

3.4.6 Solid Extraction Techniques

Solid-extraction techniques are at the interface between solid characterization and water quality. Some extraction techniques relate to regulatory requirements (e.g., synthetic precipitation leaching procedure [SPLP], toxicity characteristic leaching procedure [TCLP], and meteoric water mobility procedure [MWMP]; see Appendix 3). The field leach test (Hageman 2007; see Appendix 3) was developed by USGS as a fast, simple, and cost-effective leach test to screen and prioritize mining wastes for their water reactivity. A variety of other short-term leaching techniques have been used to assess mine-waste materials (e.g., see Al-Abed et al. 2006). Selective-extraction techniques have been used to provide insights into element partitioning within operationally defined fractions in earth materials. Extraction methods attempt to fractionate the different pools of trace metals by using sequential extraction steps with chemical reagents of various strengths, reaction times, temperature, and pH conditions (e.g., Tessier et al. 1979; see Appendix 3). Bioaccessibility tests, which are extractions using surrogate biological fluids, have been used to evaluate uptake of metals under biological conditions (e.g., Ruby et al. 1993; Drexler and Brattin 2007). Toxicity field test kits and simple laboratory screening tests can be used to assess potential metal toxicity (e.g., Choate et al. 2008). See USEPA (variously dated) and Appendix 3 for additional information.

3.4.7 Microbial Sampling*

The kinetics of nitrogen, sulfur, metal cycling, and pyrite oxidation are often controlled by microbial activity. Microbes live in water as free-floating planktonic organisms or are attached to mineral surfaces within biofilms—within microniche environments defined by pore-scale carbon, gas, and moisture conditions. In highly productive environments, such ARD settings, thick mats of biofilm can develop that host multiple species of archaea and bacteria within a biogeochemical ecosystem (Druschel et al. 2004). Such a system may have spatial and trophic structure, which should be considered in the sampling plan where relevant. Therefore, the required sampling

* This section was written by James J. Gusek and Lisa Bithell Kirk.

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MICROBES IN MINE WATER AND WASTE—COLLECTING AND MANAGING LIVE SAMPLES

Lisa Bithell Kirk, Enviromin, Inc.

It's alive! Although they are not visible in hand specimens, diverse microbial communities live free floating in water and attached to mineral surfaces by biofilms, where they directly influence the sulfur, nitrogen, and metal geochemistry of mined materials. Enzymes produced by these microbes greatly accelerate biogeochemical reactions of interest to mine-waste management, both good and bad—especially sulfide oxidation and sulfate reduction, redox changes in solubility of many metals, nitrogen speciation, and carbonate precipitation. There is a growing comprehension of the influence of microbial biogeochemistry and the resulting biotechnological opportunities to harness these processes for environmental and metallurgical management. These matters point to a critical need for care in sampling to (1) avoid introducing bias by mismanaging microbial influence on biomineralization during storage and testing, and (2) obtain desired experimental control by correctly describing microbial influence where it is relevant. The demonstrated value of bioremediation applied in wetlands in remediation, use of biofilm-based reactors in water-treatment systems, and application of biologically based systems in hydrometallurgy and process engineering show a reverse trend in the application of biotechnology through the mine life cycle, and some believe that more comprehensive characterization of native microbial communities and integration of their functional ecology into the biogeochemical models of solute release and attenuation in mine-waste facilities may improve mine-waste management. To that end, microbiological considerations need to be addressed during sampling. Even if microbiology is not a focus of a mine-waste study, investigators should consider oven-drying the samples at low temperature to remove bias introduced by biological activity following collection or the potential for ongoing biomineralization processes during storage. Comparison of samples that have been treated differently (some live, some dead) is especially problematic and needs to be addressed carefully.

Samples collected for microbial analysis must be collected using sterile techniques, and the advice of a qualified microbiologist or biogeochemist should be sought to do this properly. Care must be taken to plan ahead to order sterile sampling supplies and prepare a detailed sampling, storage, shipping, and analysis plan. At times (e.g., when drilling), maintaining truly sterile conditions (e.g., with alcohol and an open flame) can be practically impossible, and sanitized conditions with suitable controls to evaluate the magnitude and extent of contamination must be an acceptable alternative. Where possible, drilling should be dry, with no use of additives involving carbon compounds. Collection of organisms through filtration is possible, although the density of planktonic organisms can be quite low and a large volume of water may need to be filtered to obtain the needed biomass for analysis. It can be more effective to collect suspended sediment and extract attached organisms from it as well. Sampling geologists or environmental scientists often require specialized training. Additional time is required for microbiological sampling to avoid contamination; this is especially true if collection of strict anaerobic organisms is desired because samples will need to be collected and preserved under nitrogen gas. Unique health and safety considerations arise from these sampling protocols, which should be considered in the industrial work environment. A good review of subsurface microbiological sampling issues is provided by Amy and Haldeman (1997).

Samples to be used for live cultivation (e.g., enumeration, rate reactors, enrichments, isolation experiments) should be collected using sterile protocol and stored under dark conditions at 4°C (39°F) until they can be shipped to the laboratory. Samples to be studied using molecular methods (e.g., analysis of genomic DNA, expression of enzymes using RNA) should be collected using sterile protocol appropriate to the desired analysis and flash frozen on-site using a dry ice and ethanol bath, and immediately transported on dry ice to a –80°C (–112°F) storage facility. Shipping can pose unusual challenges because freezing and thawing of nucleic acids is damaging; therefore, delays should be avoided wherever possible. International shipping of live samples for research and analysis purposes is also uniquely challenging and requires advanced planning.

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methods will depend on the characteristics of the studied environment and on the dynamics of the targeted microbial community, as well as the goals for analysis of the samples (see Microbes sidebar).

As the kinetics of pyrite oxidation are mostly controlled by microbial activity, sampling of rock for bacterial characterization or quantification may be included in a sampling and analysis plan. Specific sampling techniques will be governed by the situation (e.g., is the original rock sample submerged in liquids or dry) and the sampling technique (hand sampling or drill cuttings). In drill-cutting-derived samples, the drilling mud composition can bias the sample either positively or negatively for acidophilic microbes. Positive bias could be introduced through recirculating mud or cuttings that are developed nearer the surface of a given hole; negative bias may be introduced by using organic-based muds containing polymers that could support bacterial communities that can out-compete *Acidithiobacillus ferrooxidans*. Extraordinary care may be required in sterilizing drill bits and minimizing drill-cutting recirculation to obtain unbiased rock samples for microbial characterization. Microbiological sample preservation techniques are beyond the scope of this discussion, and it is assumed that a qualified microbiologist would be responsible for developing the sampling and analysis plan.

3.4.8 Sampling Theory and Errors

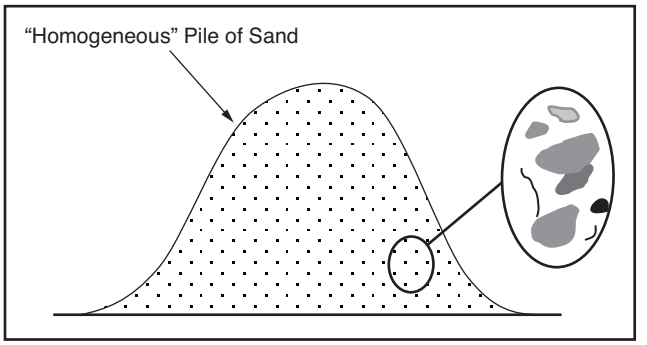
The final results of an analytical determination will never be any more reliable than the reliability of the sampling. In fact, sampling is the most important step in an analysis. Many modern analytical techniques have errors <5%, whereas poor sampling can result in errors ≥100%. If a sample was not properly collected or is not representative of the target population, then it doesn't matter how much QA/QC is performed by the laboratory or during other aspects of the investigation.

All materials to be sampled exhibit some degree of heterogeneity, which is important because overlooking, or unfamiliarity with, this heterogeneity can result in significant sampling errors, incorrect decisions, and wasted effort and expense. Sampling theory is applied as a preventive tool to find ways to minimize the negative effects of heterogeneity on sampling. The degree of heterogeneity depends on the scale of observation. This concept is illustrated by the pile of sand shown in Figure 3.5. From a distance, the sand appears to be homogeneous, but closer examination reveals the heterogeneity of the particles. Scale (both spatial and temporal) is a very important consideration. Variability at one scale may be negligible at a different scale; hence, the scale of sampling for an investigation must match the scale of the objectives of the investigation and of the ultimate application of the data (Smith 2007; see Understanding Scales sidebar).

Field sampling, sample preparation, and laboratory subsampling are generally the major sources of error in environmental investigations (Gy 1998; Ramsey and Hewitt 2005). Therefore, DQOs need to take into account sampling, sample preparation, and analytical errors when defining the sampling plan, scale, and desired uncertainty. The theories of Pierre Gy (1982, 1998) present practical sampling, comminution, and subsampling methods that can be applied for less expense because fewer samples are necessary with this sampling approach. Careful attention to these techniques of sampling correctness can result in samples that better represent the site and data that more truly represent the sample.

Sampling theory addresses two types of heterogeneity, compositional (constitutional) and distributional. Compositional heterogeneity refers to the variability in the composition of individual components of the material. For solid materials, it cannot be remedied by mixing; it can only be controlled by increasing the sample mass or physically changing the material (i.e., decreasing the particle size). The compositional heterogeneity error is greatest when the analyte is present as a few discrete particles of pure material (e.g., discrete gold particles, sometimes called the *nugget*

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Source: Adapted from Myers 1997.

FIGURE 3.5 Heterogeneity as a function of the scale of observation

effect in exploration). Distributional heterogeneity is the spatial component of the variability. It is related to the nonrandom spatial and/or temporal distribution of the components of the population.

Gy's sampling theory (Gy 1982, 1998; Pitard 1993; USEPA 1999) addresses seven types of errors:

1. **Fundamental sampling error (FSE):** This error is due to compositional heterogeneity in the chemical and physical composition of the material (includes particle size distribution). It can be reduced by decreasing the diameter of the largest particles (comminution) or by increasing the sample mass. In the field, increasing the sample mass is usually the only practical option (unless drilling results in particle size reduction).
2. **Grouping and segregation error (GSE):** A GSE is due to nonrandom distribution of particles (i.e., distributional heterogeneity). This error can be reduced by collecting and combining many randomly selected increments.
3. **Long-range heterogeneity error (shifts and trends):** This is a fluctuating, nonrandom spatial error.
4. **Periodic heterogeneity error (cycles; QE2):** This is a fluctuating temporal or spatial error.
5. **Increment delimitation error (IDE):** An IDE is due to the wrong choice of sampling equipment. It can be reduced by using well-designed sampling equipment.
6. **Increment extraction error (IEE):** This error is due to the inappropriate use of sampling equipment. It can be reduced by using well-designed sampling equipment and good sampling protocols.
7. **Preparation error (PE):** This error is due to loss, contamination, or alteration of a sample. It can be reduced by using appropriate sample handling, processing, and preservation, and by choosing appropriate analytical methods.

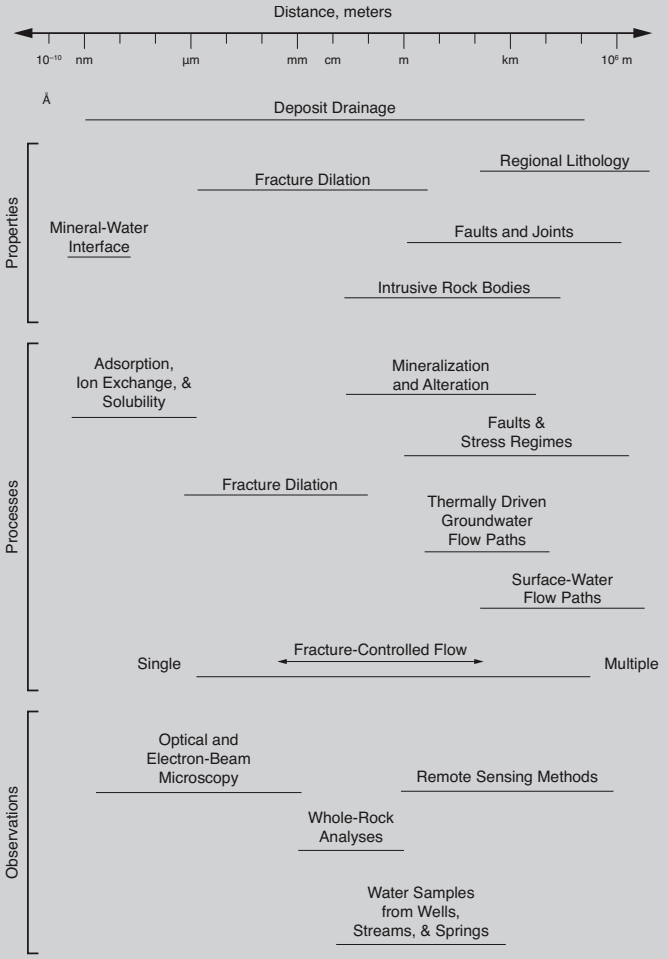
When a sample is collected, the amount of the FSE is due to the compositional heterogeneity (e.g., mineral composition, shape, liberation, and particle size) of the material being sampled. The FSE never can be eliminated, but it can be minimized and estimated. Procedures to minimize the FSE need to be included in the DQO process and the FSE should be estimated prior to sampling. It is important to keep the variance of the FSE within reasonable limits. The variance of the FSE can be estimated using the following formulas:

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UNDERSTANDING SCALES OF GEOLOGIC SYSTEMS

Richard B. Wanty, U.S. Geological Survey

In addition to using proper sampling protocols, it is important to have a comprehensive sampling strategy designed to meet the needs and goals of the project. Such strategies require a considerable amount of thought prior to collection of the first sample. Strategies are tempered by practical considerations such as budgets (of time and money), physical access to sampling locations, and availability of equipment, but they should be driven primarily by the question: What do I hope to learn about the study area?



A subsidiary question might be: If I collect a sample {of something}, what does it represent? To answer this question adequately, one must consider the spatial and temporal scales at which geologic and chemical systems vary, and plan sample location and timing accordingly.

In the adjacent figure, the number line across the top represents 16 orders of magnitude of distance, from 10^{-10} to 10^{6} m. A variety of properties and processes of geological systems are compiled, as well as some common techniques used in field campaigns, along with horizontal lines to represent the approximate limits of the spatial scales relevant to each item. By lining up the properties and processes that are relevant to the study with the observational methods in the bottom portion of the figure, a more efficient sampling strategy can be developed. In many cases, each observational method represents a particular specialist, so application of this figure can also aid with project staffing.

The figure could easily be modified to include other properties, processes, and observational methods, but the example given here is simply designed to illustrate the approach. Additionally, one might easily envision a similar figure in which the number line across the top represents temporal, rather than spatial, scale.

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$$\sigma^2_{FSE} = (K\, d^3) / M_S$$

$$\log \sigma^2_{FSE} = \log K + 3 \log d - \log M_S$$

where K is an empirical constant, d is the maximum particle diameter (in centimeters), and M_s is the mass of the sample (in grams). As shown in the previous equation, the value of $\log \sigma^2_{FSE}$ (the variance of the FSE) is proportional to $-\log M_S$. Pitard (1993) provides nomographs that relate the variance of the FSE to the maximum particle diameter and the sample mass. These nomographs can be used to optimize the sampling protocol.

From the time of collection to the time of analysis, a sample may undergo preparation involving several splitting or subsampling events, and each of these events gives rise to sampling error. For solids, the FSE can be minimized by increasing the sample mass or decreasing the particle size of the largest particles. Also, it is critical to use proper sampling equipment and protocols, which addresses the IDE, IEE, and PE. Once the correct sampling equipment and protocols are determined, it is possible to minimize the GSE by collecting multiple increments (i.e., subsamples). In general, a sample should be composed of at least 30 increments. In practice, especially for trace constituents, 50 to 100 increments should be used. Random sampling is also important, and it is essential that all of the target population be equally available for sampling.

Pitard (1993) and Myers (1997) provide several examples of the advantages and disadvantages of different designs of sampling equipment. Unfortunately, most of the sampling devices currently available for environmental sampling are incorrect (e.g., augers, thiefs; Myers 1997). Pitard (1993) points out that pipe samplers, such as augers and thiefs, are problematic because the bottom portion of the sample is under-represented due to the presence of the conical head. The U.S. Army Corps of Engineers Cold Regions Research and Engineering Laboratory has developed a Multi Increment sampling tool (CMIST) that is consistent with sampling theory (Walsh 2009). In addition, Pierre Gy recommends scoops and spatulas that are flat with parallel sides to avoid preferential sampling of coarse particles (Gy 1982, 1998; see Appendix 3).

3.4.8.1 Multi Increment Sampling

Multi Increment (MI) sampling (EnviroStat, n.d.) is a composite sampling approach based on sampling theory that reduces data variability and provides an estimate of mean concentrations within a target population (or decision unit; Ramsey and Hewitt 2005). An increment is the material extracted from the target population in a single operation of a sampling device. A sample is obtained by the combination of several increments, with the objective to minimize heterogeneity and to reduce the GSE. To properly represent the entire target population, material needs to be sampled from many locations within the target population (Wait and Ramsey 2007). MI sampling is similar to incremental sampling methodology and incremental-composite sampling, and involves collecting numerous (typically 30 to 100) increments of soil that are combined, processed, and subsampled according to specific protocols (see ITRC 2012; see also training resources listed in Section 3.13). MI sampling reduces analytical costs compared with collecting and analyzing multiple discrete samples. The State of Hawaii Office of Hazard Evaluation and Emergency Response discusses MI sampling in its online technical guidance manual (Hawaii Department of Health 2009). The State of Alaska has prepared draft guidance on MI sampling (Alaska 2009). In their guidance they point out that "MI may not be used to dilute contamination and therefore underestimate the need for cleanup. This may occur if the decision unit inappropriately incorporates large, uncontaminated areas in addition to real source areas" (Alaska 2009).

A potential disadvantage of MI sampling is that information about spatial variability within the target population is lost because the target population is represented by one MI sample.

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However, collection of triplicate MI samples allows for statistical calculation of the standard deviation, the relative standard deviation, and the 95% UCL (upper confidence limit) of the mean concentration for a given target population (or decision unit). Therefore, variability in spatial concentrations between different target populations can be evaluated, which requires careful definition of the target populations (or decision units) at the beginning of the investigation. The 95% UCL of the mean can be an important statistic because it is often used in risk assessments.

3.4.9 Sampling Strategies and Sampling Designs

During the seven-step DQO process (USEPA 2006b), it is necessary to define the goal of the investigation, which states how environmental data will be used in meeting objectives and solving the problem, identifies study questions, and defines alternative outcomes. This information dictates how the sampling strategy will be handled. For example, which of the following questions is more applicable:

- Is a particular contaminant present at the site? or
- What is the average concentration of a contaminant in a particular area?

These are very different questions that necessitate very different approaches to sampling.

Figure 3.6 illustrates reproducibility and bias in sampling where there is one sample collected for a given location or time. The top two graphs illustrate situations in which the mean sampling error and the sampling variance are near zero, which is the goal of proper sampling. The bottom two graphs illustrate problem situations in which the sampling is not reproducible. These problem sampling situations can lead to costly mistakes and flawed decisions.

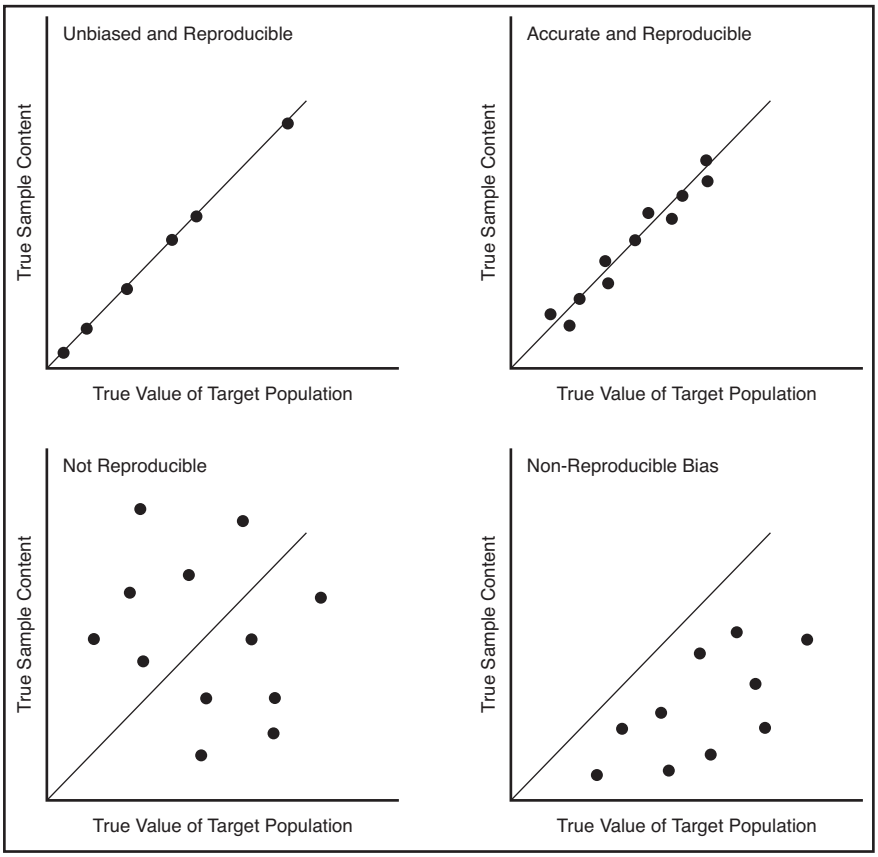
Correct sampling is the only way to eliminate sampling biases (Gy 1998). For correct sampling, all of the components of interest in the target population must be available and must have an equal probability of being randomly selected for inclusion in the sample. Therefore, both randomness and equiprobability must be considered when designing spatial sampling. Myers (1997) lists the following key components of a sampling strategy:

- Select the target population.
- Characterize the heterogeneities.
- Develop a sampling plan.
- Implement the sampling plan.
- Characterize spatial variability.
- Optimize within DQO constraints.

Cochran (1977), Gilbert (1987), and Gilbert and Pulsipher (2005) discuss the details of several sampling designs and their application for different sampling objectives. Sampling designs have different strengths and weaknesses; consequently, the chosen sampling design should be consistent with the site, media, and presence of trends or cycles. Commonly used sampling designs include strict random sampling, systematic random sampling, and stratified random sampling. There also are sampling methods that don't involve random selection, including convenience sampling, judgmental sampling, and purposive sampling.

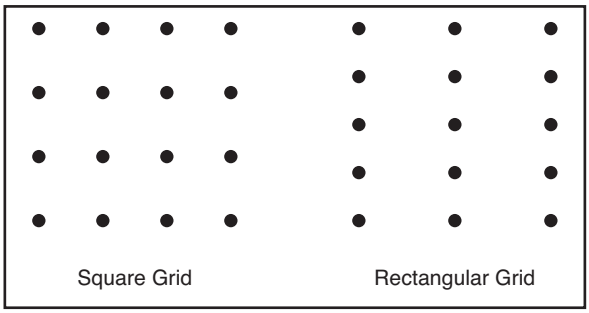
In systematic random sampling, samples are evenly distributed over the entire target population in an organized manner. Systematic grids can include square, rectangular, or radial grids (Figure 3.7). Systematic random sampling has a random start with subsequent samples taken at a set interval. If any periodicities or cycles exist, the sampling interval needs to be evaluated so that it does not result in biased results. Systematic random sampling has the lowest error of all random sampling protocols and is the easiest to implement.

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Source: Adapted from Pitard 1993.

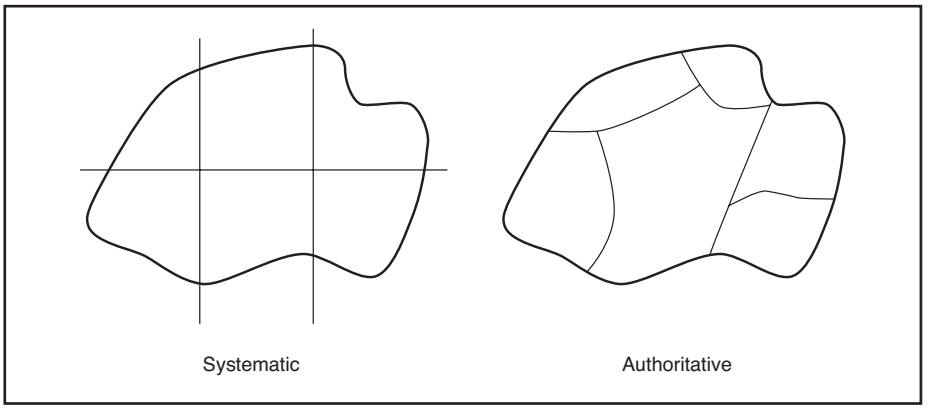
FIGURE 3.6 Reproducibility and bias in sampling



Source: Adapted from Myers 1997.

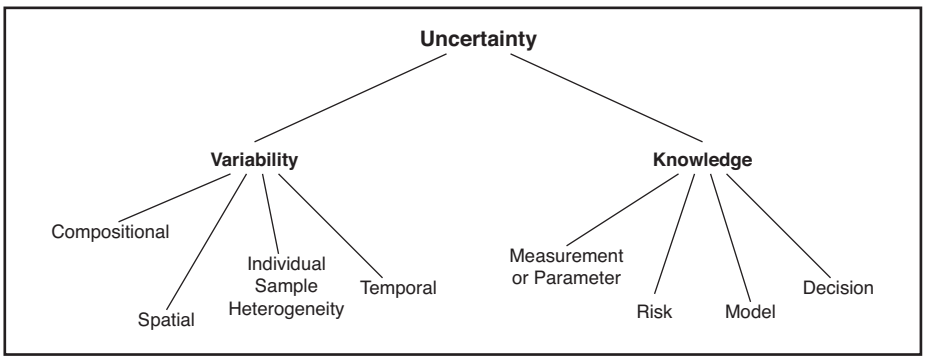
FIGURE 3.7 Different types of grids used in systematic random sampling

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Source: Adapted from Myers 1997.

FIGURE 3.8 Types of stratified random sampling



Source: Adapted from Haimes 1998.

FIGURE 3.9 Some sources of uncertainty

Stratified random sampling involves some grouping of the sample population and then randomly sampling within each group (Figure 3.8). It has both components of randomness and equiprobability. Stratified random sampling is often used when the target population can be split into non-overlapping subgroups that individually are less heterogeneous than the target population as a whole (but this is not a requirement). If there are no obvious subgroups, grid lines can be used to divide the target population (Figure 3.8). Random samples are taken from each subgroup and the results are combined to estimate parameters of the population. However, this approach does not lend itself to MI sampling because there is not enough mass or increments in each subgroup to represent it.

Figure 3.9 summarizes several sources of uncertainty. The desired degree of certainty for an investigation must be decided at the inception of the investigation. A low degree of certainty in the data can lead to erroneous interpretations and flawed decisions. Conventional thought is that a higher degree of certainty generally requires more sampling and higher costs. However, better sampling techniques that incorporate sampling theory can provide more representative data at reduced costs.

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3.5 DRILLHOLE/BOREHOLE SAMPLING CONSIDERATIONS*

3.5.1 A Reasonable Sampling Plan

A senior exploration geologist would typically develop a drillhole sampling plan, whether it is for exploration borings or production blastholes. The plan needs to consider drilling production because drillers may be compensated on depth penetrated. If drill bit advance is interrupted by sampling operations, then standby charges, which can be onerous, are certain to be incurred. Worse, an unhappy driller can compromise sample integrity in a number of ways that might only be discovered weeks after the fact, if at all.

Once the sampling plan is understood and accepted by the drill crews, a qualified geologist or engineer will periodically oversee the drilling program to examine borehole cuttings and disturbed material, and should be on call to resolve issues that can arise. Field inspection of materials is recommended for all components of the project, though especially for those components of the project in medium- to high-ARD risk zones or where potentially acid generating (PAG) rock or acid-producing rock (APR) materials have been identified. The geologist/engineer may examine the materials for

- Mineralogical content, in particular the presence of sulfides or siderite and other carbonates;
- Fizz rating;
- Sulfur odor, particularly during fizz testing;
- Paste pH; and
- Steam from completed drillholes.

If low paste-pH values (e.g., below 5.0; Skousen et al. 1987), a lack of fizz (Sobek et al. 1978), sulfurous odors, steam, and sulfide minerals are observed, then materials can be assumed to be PAG or APR and should be tested and/or managed appropriately. If some, but not all, of the signs of ARD are present, they should be noted in the drill logs and precautions taken to prevent ARD formation. The geologist or engineer overseeing the project may need to inform site personnel (e.g., the chief project/site geologist or geochemist) of these tests and their proper implementation to allow appropriate classification and handling of materials.

3.5.2 Borehole Sampling Considerations

If PAG/APR zones are identified during the preliminary exploration drilling program, then blast borehole sampling should be performed for ARD potential to ensure that these materials are accounted for and managed appropriately during active mining of the deposit. Until a less-prescriptive blasthole sampling program that can be understood and followed by drillers is developed by an experienced site geologist/geochemist, the following guidance is offered as a starting point. A minimum of three boreholes per blast should be sampled for environmental purposes, or as many as are necessary to obtain optimum coverage of the material to be blasted. Samples should be collected a minimum of every 1.5 to 2 m (5 to 6.6 ft), or a certain percentage of the samples collected for routine ore and waste control, if applicable. However, if significant lithologic changes are observed, samples may need to be collected at smaller intervals.

Samples should be immediately submitted for static ABA and paste pH analyses (e.g., 48-hour turnaround times or less) because once the holes are loaded with blasting agents and detonated, excavation to either the waste rock dump or ore stockpile(s) can shortly follow. If acid-producing mine production waste is to be properly managed, especially in zones that may or

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may not be acid producing, the destination of these materials needs to be known well in advance to avoid disruptions in haulage routes and fleet scheduling. Once a truck load of APR is dumped in a *clean* rock dump instead of in a planned APR repository, it is very difficult to undo because of contamination of the non-acid-producing material with the AD from the APR.

3.5.3 Sampling Methods

This section provides guidelines for appropriate sampling methods for rock and water that may be intercepted during drilling activities, which is not to be confused with water sampling from properly completed and developed monitoring wells. The purpose of sampling is to obtain representative samples of the material throughout the entire area that will be affected by project activities. The collection of representative samples is the primary intent of the sampling methods described in this section. Sample handling, logging, shipping, storage, and chain of custody are also described. Sampling should be performed by or under the oversight of a qualified scientist or engineer.

3.5.3.1 Rock Sampling

Rock and soil samples may be collected into resealable plastic bags or other clean, sealable sample receptacles (e.g., bottles or ore bags). Samples should be logged using standard site procedures or methods. Generally, a 4-L (1-gal) resealable plastic bag filled with material is sufficient volume for recommended laboratory tests; however, this volume may be adjusted by the analytical laboratory performing the work or the geologist/engineer requesting analyses. Microporous vapor-permeable bags can be useful for collection of development drilling or blasthole samples to permit drying of the samples in the bags without loss of fine solids. Drill samples containing particulate gold may require significantly larger samples (up to 15 kg [33 lb]) if gold assay is one of the sampling objectives.

Once collected, samples should be sealed to prevent oxidation or moisture loss if extent of oxidation and moisture content is to be determined. Typically, sample containers are labeled with information such as

- Date and time of collection,
- Borehole identification,
- Depth interval sampled,
- Geologic formation (if known or suspected),
- Alteration (if known),
- Individual conducting the sampling,
- Station number,
- Project identification, and
- Other pertinent information.

An appropriate number of duplicate samples should be collected to meet the project DQOs. Samples should be analyzed at a certified laboratory if the results are needed to meet permit or compliance requirements and always analyzed using appropriate QA/QC measures. Sample locations should be mapped and given a unique identifier. Locations and sample identification should be logged into field notes and entered into the geographic information system database, if applicable.

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3.5.3.1.1 Drilling Methods

Drilling for geochemistry samples for APR evaluation may be combined with geotechnical drilling. The following drilling methods are examples of acceptable alternatives for collection of rock or soil samples over the entire mine life. Whereas some methods are preferred because they provide more distinct and intact samples, this must be balanced with time and expense. The following methods are listed in order of preference for collection of geochemistry samples for APR evaluation:

- Conventional coring
- Roto-sonic coring
- Air rotary reverse circulation
- Air rotary

One drawback of the air rotary methods is that salting of the hole can occur. As the drill penetrates a rock layer, the rock chips are blown out of the hole by the air drilling process. In some cases, the pyrite-containing rock chips are heavier and tend not to be adequately exhumed from the drillhole. Subsequently, these chips fall back into the hole, biasing the amount of pyrite in the next sample. This can usually be detected by reviewing the test results. If the tests indicate an increasing amount of APR as the depth increases, then re-drilling and testing should be considered, or a review of the test results and rock samples may be required. A drawback of core drilling is that fines can be washed away before the core is retrieved from downhole, which can introduce significant bias into subsequent analytical procedures if critical minerals, such as sulfides or other trace metals, are associated preferentially with finer material.

Auger sampling is generally not recommended for geochemical sampling of rocks, as intermixing and contamination of samples can occur as materials are lifted by the auger screw (Kleinmann 2000). However, auger sampling of soils from shallow depths (i.e., less than 3 m [about 10 ft]) may be acceptable if soil stratigraphy or depth to bedrock is well understood. Samples can be collected from drill cuttings or the drill core. If difficulties in determining sample depth or the geologic strata are encountered because of incomplete or inefficient cuttings return or cuttings are significantly altered, then coring may be required. Samples should be collected dry when possible. In this handbook, it is assumed that the drilling fluids are derived from uncontaminated water sources; if the water source is suspect, then it should be sampled along with the rock core.

3.5.3.2 Water Sampling

Exploration drilling and mining operations can encounter groundwater. Drillers may note zones of excessive groundwater inflow in the drilling mud management or appearance of moisture changes in drill cuttings. It is virtually impossible to collect an unbiased sample of these waters, but water chemistry might be inferred by spot sampling the drilling mud, filtering out the suspended solids, and comparing the chemistry with a known baseline.

When drilling with air, drillers can pause at a zone that is *making water*, stop drill bit advance, and blow water out of the hole. Sampling and analyzing this water may provide clues to the chemistry of the rock mass being penetrated, and the results may suggest zones for future monitoring wells.

3.5.4 Sample Storage and Chain of Custody

Following logging and collection, any rock samples collected primarily for environmental purposes should be stored in a closed, water-resistant, noncontaminating receptacle (such as a cooler)

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and kept in accordance with the site-specific sampling plan protocol until transfer to the analytical laboratory. Routine ore and waste-control samples collected primarily for metallurgical purposes should be handled in a manner that optimizes assay turnaround requirements. Unlike water samples, which are cooled in transit to analysis laboratories, rock sample temperature will probably be governed by the ambient temperature of the ground mass, and use of an insulated cooler will likely preserve that condition at least temporarily. Rock samples should be shipped or transported to the analytical laboratory as necessary to ensure that holding times, if any that may be internally imposed, are not exceeded. For example, it might be poor policy to accumulate rock chip samples from a given blast round for ABA parameter analysis past the planned date/time that the round would be loaded and shot, and excavation would commence shortly thereafter. The holding times should consider typical laboratory turnaround times in order to receive the analytical data in time to make reasonable characterizations.

Samples should be stored and shipped following site-specific chain-of-custody procedures using guidance from ASTM D 4840-99 or the analytical laboratory. Doing this would help to ensure that the intended test methods and protocols are applied to the samples submitted. The chain-of-custody forms also provide a helpful paper trail for sample tracking.

3.6 BIOLOGICAL SAMPLING CONSIDERATIONS—AQUATIC*

Direct measurement of the biological condition of the aquatic environment in areas influenced by mining is a critical component in understanding the potential and realized ecological impacts of that human activity. If correctly employed, biological monitoring can help to answer many key questions about mining impacts, such as the following:

- What organisms would we expect to be present at a given site absent mining or processing?
- Are organisms that are expected to be present currently resident at a given site year-round or seasonally?
- How healthy is the aquatic ecosystem at a given site?
- Is poor water or sediment quality resulting from ARD a direct limiting factor for the aquatic ecosystem?
- What other factors (e.g., lack of suitable habitat) could be limiting factors for the aquatic ecosystem at a given site?

Collection of biological media is discussed in Appendix 3.

Aquatic communities present at a given location are a function of the biological, physical, and chemical conditions that exist at that site, all of which may vary over time. In order to answer questions about the potential impacts of mining and processing on surrounding aquatic resources, it is important to design studies in an effective manner. Several experimental approaches exist to evaluate an aquatic ecosystem's response to a stressor such as ARD (Power 2002). For example, to gauge the impact of a specific mine or process-related discharge on the receiving stream, comparison of the downstream aquatic community with an unimpacted upstream reach or reference site(s) may be appropriate. In order to evaluate the downstream extent of a mining impact, measurement of the biological community response to decreasing exposure severity with increasing distances downstream could be completed. In a temporal context, sampling the aquatic community before and after a mine remediation project could serve to document the benefits of that project. Where possible, identification and utilization of an analog watershed (i.e., a watershed that is lithologically similar, absent the mining disturbance) can be useful in determining the

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premining geochemical and ecological condition. Importantly, the characterization of a mine's impact or the development of a mine remediation goal relying solely on biological population/community data is not advised, and the collection of additional abiotic analytical data (see other sections of this handbook) is highly recommended to support these population/community data.

A typical first step in evaluating the *health* of an aquatic ecosystem is the characterization of its expected biological condition, absent any direct anthropogenic influence (e.g., mining). Typically, the expected biological condition is established through the quantification of biological community metrics within an appropriate *reference* site(s), which can be an unimpacted upstream reach, or from a site in a nearby watershed with similar physical characteristics (including similar lithology). Comparisons of resultant biological information obtained from the presumed unimpaired reference site with data from the test site can be used to assess degree of impairment. The establishment of good reference sites is paramount in this approach to ensure that sites would have comparable biological potential absent the anthropogenic stressor of interest (Stoddard et al. 2006). For example, a lack of fish at a well-selected reference site might suggest that a measured lack of fish at the test site is a consequence of poor habitat rather than poor water quality (Milhous 2007). As such, regardless of the apparent suitability of a reference site, some level of habitat characterization is advised in both reference and impacted reaches to ensure reliable interpretation of the results of their comparison. Habitat comparisons can range from qualitative to highly quantitative depending on one's needs.

Several biological assemblages (e.g., algae, benthic macroinvertebrates, fish) are suitable for characterizing ecological stress in aquatic systems. Overall study design and other practical considerations (e.g., budget, schedule, access) will ultimately define what biological communities can/should be sampled in a given water body. In general, employment of multiple lines of biological evidence is recommended to thoroughly characterize the nature and extent of impairment and/or risk (Besser et al. 2007; Farag et al. 2003). For example, in an ARD-impacted stream devoid of fish, electrofishing alone would not necessarily yield a likely cause of impairment; instead it would simply identify the impairment itself (i.e., lack of fish). However, in conjunction with analyses of exposure media for contaminants or with site-specific water and sediment toxicity testing conducted within a tightly controlled laboratory environment, it would be possible to establish if poor water or sediment quality (as opposed to other potential causes, such as lack of available food or habitat) was the cause of the observed impairment. It is important to independently evaluate all potential causes for the observed biological condition, as impairment could be caused by any (or all) of these potential limiting factors.

Directly sampling fish populations can yield a variety of useful information about the causes and relative degree of biological impairment. Qualitative fish sampling, through methods such as hook-and-line fishing or *spot* electrofishing, can be a cost-effective means of evaluating the presence or absence of fish in MIW. Quantitative fish sampling methods, such as multiple-pass depletion electrofishing protocols, produce fish population metrics (e.g., population size, species composition, biomass), which can be compared with reference-stream measurements (Farag et al. 2003; Reynolds 1996; Bervoets et al. 2005). Measurements taken on individual organisms can also be used as a line of evidence in documenting an impaired aquatic ecosystem. For example, simple measurements of fish lengths and weights can be used to calculate and then compare average condition factors for groups of individual fish (i.e., fish this long should weigh this much) from impacted and reference waterways (Todd et al. 2007). Further, fish captured during surveys can be employed through the use of standard fisheries techniques (e.g., tagging, radio telemetry) to answer additional important questions about the fish population (i.e., is the fish population resident year-round?) (Hoar 2005; Goldstein et al. 1999; Scullion and Edwards 1980). Finally,

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stocked fish can be utilized in the field to evaluate the site-specific suitability of water quality conditions through the use of in situ exposure experiments (e.g., Todd et al. 2007; Farag et al. 2003; Harper et al. 2008).

In the laboratory, fish can be used to develop additional information in isolating the root cause of observed impairments. For example, toxicity testing is often conducted to establish threshold levels of a given contaminant of concern (e.g., Zn) to a given organism (e.g., rainbow trout), with these thresholds then compared to field measurements of the contaminant to assess risk to the organism from that contaminant (USEPA 2002b). While this type of traditional reference toxicity testing can prove useful in many ways, because of the chemical complexity of most site waters and resultant implications for toxicity, toxicity testing using serial dilutions of site waters can also yield relevant, site-specific toxicity information (Besser and Leiby 2007; USEPA 2002b). Fish tissues have also been analyzed in the laboratory to serve as lines of evidence linking metal exposure pathways to the physiological malfunction in the organism. For example, tissue metal concentrations have been employed to serve as a link between elevated aqueous metals concentrations and observed fish impairment (Todd et al. 2007; Farag et al. 2003; Bervoets et al. 2005). This tissue-residue approach does not always yield useful information, as metals concentrations can be so acutely toxic that tissue concentrations do not have a chance to build before the organism perishes. Additional biochemical measurements can be used as measures of metal exposure, including the measurement of lipid peroxidation (e.g., Farag et al. 2003) and the metal-detoxification protein metallothionein (e.g., Hamilton et al. 1987).

Measurement of lower aquatic trophic levels (e.g., benthic macroinvertebrates, algae) can also be an important component to studies evaluating the ecological impacts of past, present, or future mining in a given area (e.g., Griffith et al. 2004). Largely because of their widespread presence in aquatic environments, their limited mobility, and their ease of collection, benthic macroinvertebrates themselves are frequently employed as direct indicators of aquatic ecosystem health as community parameters such as organism diversity and abundance respond to anthropogenic stressors such as ARD. Specifically, benthic macroinvertebrate metric categories have been defined (e.g., taxa richness, species composition, pollution tolerance, functional feeding groups) to represent assessments of community structure and function, and appropriate combinations of these metrics can yield a useful index characterizing the health of the resident aquatic community (Barbour et al. 1999). Although there are hundreds of metrics that can be used to assess community structure and/or function, a few (e.g., richness, abundance of mayflies, stoneflies, and caddisflies) have been found to predictably respond to increasing metal concentrations (Carlisle and Clements 1999; Clements et al. 2000). Benthic macroinvertebrate communities are routinely used as indicators of water quality and ecosystem health in both state and federal protocols, and many states are moving toward the adoption of benthic macroinvertebrate-based biocriteria for the assessment of attainment of the aquatic life use (e.g., CWQCC 2010). Similarly, algal community metrics (i.e., diversity, biomass) can be utilized to evaluate the ecological implications of a stressor such as ARD (Niyogi et al. 2002; Barbour et al. 1999).

A number of factors can strongly influence the character of a benthic invertebrate community observed between sample locations, even in the presence of a strong stressor (Clements and Kiffney 1995). For example, resident taxa are influenced by changes in elevation (Clements and Kiffney 1995), time of year (Clark and Clements 2006), and biogeography (Hawkins et al. 2000). As a result, it can be difficult to make accurate predictions regarding the character of a benthic macroinvertebrate community among sites sampled within a large study area (such as a state or ecoregion). Predictive models, which often account for differences in site characteristics that cannot be directly anthropogenically influenced (e.g., elevation, latitude, longitude, weather), can be

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used to assess differences in taxonomic completeness of benthic macroinvertebrate, fish, or algae communities, across these gradients (Hawkins et al. 2000, Carlisle et al. 2008). These models evaluate the relationship between the taxa that are observed (O) at a site with that which would be expected (E) to be present at that site (O/E ratios). In this manner, this approach can be used to attribute differences in O/E ratios to a change in the habitat quality of the sampling location. Increasingly, state and federal agencies are moving toward the use of such predictive models to measure biological impairment (Stribling et al. 2008; USEPA 2006c).

In the evaluation of ecological impacts from mining, lower trophic levels (i.e., algae, benthic macroinvertebrates) are useful beyond direct measurement of their community metrics. These organisms are often utilized in a similar manner as fish, including through use in lab and in situ toxicity exposures (e.g., Clark and Clements 2006) and evaluation of trace-metal bioaccumulation (e.g., Rainbow 2007; Winterbourn et al. 2000). Importantly, benthic macroinvertebrates serve as an important link in both stream and riparian food webs. As such, potential dietary toxicity pathways may be an essential component in evaluating mining- and process-related risk to higher trophic levels such as fish (e.g., Besser et al. 2001) or riparian birds (Custer et al. 2009).

Several important considerations must be recognized when considering the use of lower trophic levels such as benthic macroinvertebrates and algae. Primarily, both algal and macroinvertebrate communities have distinct seasonal cycles, and as such, care must be taken to ensure that sample collection dates are well documented to avoid inappropriate comparisons. Many sampling protocols recommend a single *index period* to avoid potential complications (e.g., baseflow for algae); however, this specificity will not always be appropriate, depending on the study design (Barbour et al. 1999). While fish populations typically demonstrate less seasonality, with exceptions including migration and/or spawning times, considerations for timing fish sampling might include safety (i.e., not during peak run-off) and targeted sampling during periods of the year demonstrating highest risk (e.g., highest aqueous metal concentrations). Additionally, in sampling aquatic biota, it is critical to recognize and plan for the diversity of aquatic habitat and resultant differences in expected aquatic assemblages. For example, the benthic macroinvertebrate community sampled from a pool is likely to be very different from that sampled from a neighboring riffle, so sampling representative habitat is important for obtaining meaningful results (Barbour et al. 1999).

Measurement of components of the aquatic biological community can be used as evidence in identifying ecological impacts from anthropogenic stressors, such as ARD. In measuring and interpreting the data from these biological communities, it is important to be mindful that these assemblages are a function of physical, chemical, and biological interactions integrated over time. As such, a given biological sample should be viewed as a snapshot of that community's condition at that given time, and multiple lines of evidence should be developed to support any conclusions regarding the causes of observed impairment.

3.7 AIR SAMPLING CONSIDERATIONS*

There are a variety of reasons to sample air quality at mine sites including monitoring for combustible gases or vapors (e.g., methane), hazardous gases, vapors, aerosols (e.g., hydrogen cyanide, mercury, sulfur dioxide, and acid from solvent extraction–electrowinning [SX-EW] plants), particles (e.g., mercury salts, silica dust, diesel, asbestos, dust), oxygen deficiency, total suspended particulate matter or particulate matter <10 μm (PM_{10}) levels in ambient air, individual exposure

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AIR EMISSIONS

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Airborne emissions occur over the entire mine life cycle and ultimately can affect air and water quality, personnel health, health of fauna and flora in the environment, and can cause increased wear to machinery (AGDR 2009). During activities such as blasting, extraction, and processing, dust and larger size particles derived from the mechanical breakdown of rocks and soils are dispersed by the wind. These particles can oxidize or alter during transport before being deposited, in many cases within local water supplies. Gas emissions from fuel combustion or mineral processing can include sulfur dioxide (SO_2) and nitrogen dioxide (NO_2) and ultimately affect water quality. Mercury can be released in some mines and processes, especially in some gold operations, including autoclaving.

Although most mining and processing sites in the United States will require extensive sampling and monitoring of air quality in order to meet national and local regulatory requirements for permit application and the National Environmental Policy Act, additional sampling and monitoring could be required in order to evaluate whether air quality could adversely affect water quality, such as contributing to acid drainage. Local meteorological stations and data will also be necessary for air resources analyses to enable computer modeling of potential air-quality impacts (see Appendix 3). The station should record wind speed, wind direction, rainfall, temperature, evaporation, barometric pressure, and humidity. During mining, beneficiation, and mineral processing of most commodities, particulate material and gases can be emitted that can affect water quality and soils downwind of the mining facilities. Particulate materials include flue dusts (from sinter, roaster, smelter, or refinery stacks) and fugitive dusts (from crushers, tailings facilities, road use, blasting). Most of these are regulated in current mining operations by the U.S. Environmental Protection Agency and state and local government agencies; however, in the past, not all of these activities were controlled and monitored. Monitoring before mining or processing can be performed to establish baseline conditions. High-volume particulate matter (i.e., dust) or deposition monitoring is generally used to determine compliance with relevant air-quality standards and to ensure that the dust removal and suppression techniques are adequately working.

Targeted emissions monitoring can be performed to address site-specific issues, such as crystalline silica concentration, radon, or radioactivity. Other equipment is used to measure regulated pollutants such as sulfur oxides, which can affect downwind environments by dry or wet acid deposition. Zinc, arsenic, mercury, and cadmium will vaporize when heated during processing and can be released into the environment as gases that later precipitate or fall as dry deposition, sometimes many kilometers from the origin. All of these can affect water quality and soils downwind of the mining facilities. Special source sampling and monitoring programs should be developed to determine if these possible contaminants are issues. Sampling and monitoring of air quality, including various sampling procedures, sample collection devices, and real-time instruments, are summarized by McDermott (2004). After the World Trade Center collapse, the U.S. Geological Survey (USGS) examined the environmental effects of that disaster and summarized their results in Clark et al. (2001). Many of the techniques used by USGS can be employed at mine sites. For instance, scanning electron microscopy and X-ray microanalysis was useful in defining signature components of dust dispersed by the collapse of the World Trade Center (Lowers et al. 2005; Meeker et al. 2005).

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level, or compliance with regulatory requirements. The constituent and type of air sampling used will depend on the objectives of the investigation. Commonly used types of air sampling include

- Personal exposure sampling,
- Area airborne sampling,
- Surface or underground sampling,
- Ambient sampling of criteria pollutants, and
- Emission sampling.

Personal exposure sampling is performed for occupational health and safety purposes. In personal exposure sampling, a sampling device is placed on an individual within his or her breathing zone. In area airborne sampling, sampling devices are employed to discern sources of contaminants or the effectiveness of cleanup procedures. Area samples should not be used as a substitute for personal exposure sampling. Surface sampling generally entails use of wipe samples to determine the presence of particulates or the effectiveness of equipment decontamination procedures. Underground sampling involves specialized equipment or monitors that can be connected to warning devices and ventilation systems for carbon monoxide and diesel emissions (Sengupta 1990). Ambient sampling involves collection of data over a wide area and includes fugitive dust (particulate matter) at property boundaries. These particles can be weighed for reporting the particulate mass and can also be submitted for elemental analysis. Emission sampling includes kiln, stack, or equipment sampling for milling and processing units that have regulated emission standards by agencies.

It is good practice to collect periodic samples of exhaust mine air. Some specific air-quality concerns exist at many mine sites (see Air Emissions sidebar). Metals present in respirable dust may pose health concerns (e.g., from blasting; see Figure 3.10). For example, respirable silica dust is typically produced when drilling, blasting, or cutting silica-containing rock. Exposure to respirable silica-bearing dust can result in silicosis, a potentially fatal lung disease (e.g., Chen et al. 2005). Potential issues for gases or vapors include radon daughter products, sulfur dioxide, mercury, hydrogen cyanide, methane, or hydrogen sulfide depending on the mine and processing type and activity. The use of diesel engines underground can produce a mixture of gases, vapors, and particulate matter including carbon monoxide, nitrogen dioxide, sulfur dioxide, volatile organic compounds, aldehydes, and polycyclic aromatic hydrocarbons. Diesel particulate matter is thought to be a probable human carcinogen (see USEPA 2003a).

3.8 QUALITY ASSURANCE/QUALITY CONTROL CONSIDERATIONS

QA/QC is a system of procedures, checks, audits, and corrective actions to ensure that samples are collected, stored, shipped, and analyzed in an acceptable manner with a stated level of confidence, and that technical aspects and reporting are adequate to ensure credible results (Taylor 1987). QA/QC is part of the overall USEPA Quality System and is generally described in the quality assurance project plan (QAPP). USEPA guidance states that the QAPP must specify the level of QA/QC activities necessary for the investigation and be composed of elements covering the entire project from planning, through implementation, to assessment (USEPA 2001b). The following four main groups of elements are included in a QAPP (USEPA 2001b):

1. **Project Management:** Project/task organization (roles and responsibilities), problem definition/background, project/task description, quality objectives and criteria, special training/certification, documents and records
2. **Data Generation and Acquisition:** Sampling design; sampling methods; sample preparation, handling, and custody; analytical methods; QC; instrument/equipment testing,

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Courtesy of Virginia T. McLemore.

FIGURE 3.10 Blast at the Chino porphyry copper mine, Santa Rita district, New Mexico

inspection, and maintenance; instrument/equipment calibration and frequency; inspection/acceptance of supplies and consumables; nondirect measurements; data management

3. **Assessment and Oversight:** Assessments and response actions; reports to management
4. **Data Validation and Usability:** Data review, verification, and validation; verification and validation methods; reconciliation with user requirements

Traditionally, data quality concerns have largely concentrated on analytical errors. It is important to keep in mind that field sampling, sample preparation, and laboratory subsampling are generally the major sources of error in environmental investigations (Gy 1998; Ramsey and Hewitt 2005). Separate QA/QC procedures need to exist for all aspects of the sampling and monitoring program, including the field sampling program (collection, preservation, filtration, shipping, chain of custody, etc.), sample preparation (replicates at each stage of comminution), and the analytical procedures (laboratory). QA/QC is extremely important because "all analytical measurements are wrong: it's just a question of how large the errors are, and whether they are acceptable" (Thompson 1989).

As previously discussed, DQOs are qualitative and quantitative statements derived from systematic planning processes that clarify study objectives, define appropriate types of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions (USEPA 2006b). Data quality indicators (DQIs) are qualitative statistics and quantitative descriptors that are used to interpret the degree of acceptability or utility of data to the user and are part of the DQOs. The principal DQIs are precision, bias, representativeness, completeness, and comparability (USEPA 2006b), which are defined as follows (USEPA 2012).

- **Precision**—The degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; determined from multiple analysis of many sample replicates or standards.
- **Bias**—The systematic or persistent distortion of a measurement process.

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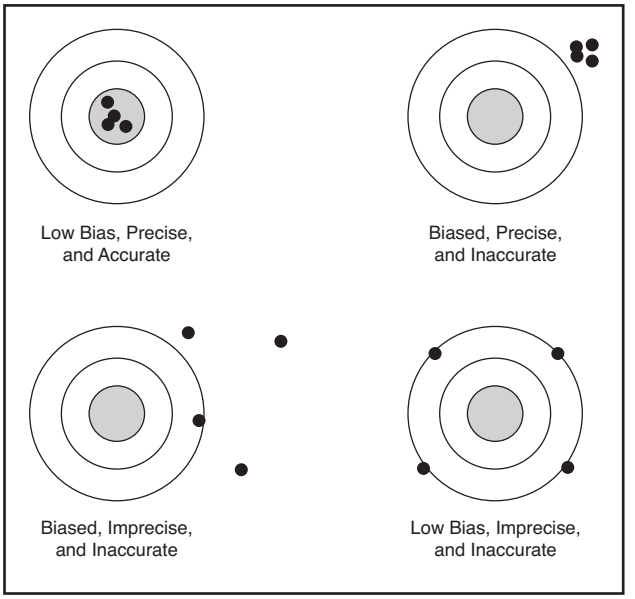


FIGURE 3.11 Diagram illustrating the difference between bias, precision, and accuracy for four measurements. The center of the target represents the true value and the dots represent individual measurements. For the bottom right target, note that individual measurements (dots) are inaccurate, but the mean of the four measurements is accurate.

- **Representativeness**—The extent to which measurements actually reflect the sampling unit from which they were taken, as well as the degree to which samples actually represent the target population at the time the samples were collected.
- **Completeness**—The amount of valid data obtained compared to the planned amount; usually expressed as a percentage.
- **Comparability**—The degree to which different methods, data sets, and/or decisions agree or can be represented as similar.

Myers (1997) defines the term *accuracy* as a summary of the closeness of measurements to the true (but possibly unknown) value. USEPA (2012) defines it as the degree of agreement between an observed value and an accepted reference value; determined by analyzing certified standards as if they were unknown samples and comparing those values with known certified values. *Precision*, *bias*, and *accuracy* should not be confused or used synonymously. Bias is usually a result of a systematic error (or errors). Figure 3.11 illustrates the classic target analogy for the distinction between bias, precision, and accuracy for repetitive analysis of one sample.

Environmental field measurements present challenges for estimating uncertainty. Qualitative and quantitative factors can contribute to field measurement uncertainty. Qualitative factors can include environmental conditions, sample handling, and instrument maintenance. Qualitative factors can be addressed through the preparation of standard operating procedures and the use of proper sampling equipment. Quantitative factors can include such things as instrument precision and accuracy, and calibration standards (USEPA 2008).

The goal of QC sampling is to identify, quantify, and document bias and variability in data that result from the collection, processing, shipping, and handling of samples (USGS, variously dated). The types of QC samples to be collected and their temporal and spatial frequency and

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distribution depend on the objectives of the investigation, data quality requirements, site conditions, and management or regulatory policy.

Accuracy can be evaluated by comparative sampling, performing analyses by alternate methods, or by analyzing certified reference materials using the same methods as are used in the investigation. Use of certified reference materials is an important part of a QA/QC program. An ARD-certified reference material can be obtained from the Canada Centre for Mineral and Energy Technology (CANMET) in Ottawa. Mine-waste reference materials also are available through the National Institute of Standards and Technology and USGS. Some Web sites for certified reference materials are listed in Appendix 1.

Generally, replicate samples or splits are used to determine precision for each stage of sample preparation in a sampling and monitoring program. Standards can also be analyzed repetitively to obtain a measure of precision. It is good practice and cost-effective to develop site-specific standard materials early in the sampling and monitoring program. Such standard materials need to be representative of the material being sampled in the sampling and monitoring plan. Multiple analyses of these standard materials, as well as analysis of sample replicates, can yield information about precision throughout the course of the investigation. Precision is usually expressed as a standard deviation, variance, or range in either absolute or relative terms. Evaluation of precision and accuracy needs to be built into every sampling and monitoring program.

Comparability can be addressed by using standardized sampling and analytical methods, and standardized units of reporting with similar sensitivity. For example, if one investigation collects a particular size fraction of waste material, the same fraction should be collected in a subsequent investigation to make the data comparable. It is always important to document any modifications of standard methods.

Blanks are used to assess possible contamination or inadvertent introduction of an analyte during the various stages of sampling and analysis. Types of blanks can include trip blanks (samples prepared prior to sampling that are not opened in the field but travel with the field samples), field blanks (samples prepared prior to sampling that are opened in the field during collection of a field sample), equipment blanks (water samples collected in the field from rinsing of sampling equipment), filter blanks (for water samples, clean water passed through filtration devices), and laboratory or method blanks (used to test for contamination from laboratory apparatus, sample preparation, and analysis).

Other types of QC samples can include matrix spikes, splits, and replicates. A matrix spike is a sample to which a known amount of the analyte is added to test for bias due to analytical interferences or matrix effects. Matrix effects refer to sample characteristics that interfere with the analytical method and can result in unreliable data. Splits are field and sample preparation samples that are split into two or more subsamples to allow for separate analysis to test laboratory performance. Laboratory splits of the prepared sample can be used when the same sample is being sent to different laboratories or to test different methods in the same laboratory. Replicates (or duplicates) are additional field samples collected at the same location under the same conditions with the same sampling protocol to test for sample variability and sampling error. The field replicate is one of the most important QC samples and should be collected every 10 to 20 samples. Replicates should be submitted *blind*; that is, with a designation that does not allow the laboratory to identify the original sample with which the replicate is associated. Table 3.2 summarizes information provided by some QC samples.

Methods of sample and data analysis need to be specified and pertinent literature referenced for standard, modified, and nonstandard methods. When using analytical techniques, each instrument needs to be calibrated with standard solutions prepared by the analyst or with QC

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TABLE 3.2 Information provided by quality control samples

	Contamination					Precision			
QC Type	Containers	Field	Equipment	Cross-Contamination	Laboratory	Sampling	Splitting	Subsampling	Analysis
Blanks									
Trip	X			X	X				
Field	X	X		X	X				
Equipment	X	X	X	X	X				
Method					X				
Replicates and Splits									
Field splits							X	X	X
Field replicates						X		X	X
Lab splits							X	X	X
Lab replicates						X		X	X
Sample prep replicates							X	X	X

Source: Adapted from Wait and Ramsey 2007.

standards. Instrument calibration is carried out to measure sensitivity, determine resolution, and check linearity. The calibration data are maintained on file so as to monitor the performance of the instrument over time.

A chain of custody may be necessary as part of a QA/QC program. A chain of custody includes a record of all personnel who have handled the sample (with date, time, and place information) and documents all samples, fractions, splits, and sample disposal. This is also termed a *cradle to grave* record for a sample. Generally, chain of custody is used for legal purposes. However, it can be useful when trying to trace the source of anomalous data.

The QA/QC process does not end once the data are reported from the laboratory. Whenever possible, analytical results and laboratory documentation need to be independently reviewed for errors or omissions. Data verification is a process in which QC parameters are evaluated against a set of predetermined criteria or guidelines (e.g., DQIs). Data verification generally includes examination of holding times, calibrations, blanks, spikes, duplicates, laboratory control samples, and blind standards. It is common to have censored data when analyzing environmental samples. For example, environmental data commonly contain values that are below the detection limit (nondetects), and these values are generally reported as less than the limit of detection. Censored data pose problems when applying statistical methods to summarize and interpret data. Helsel (2005) provides guidance on dealing with censored data.

Table 3.3 is an example of a tool that could be used for data verification. The values listed are the generally accepted range for a number of parameters in water. If data fall outside the generally accepted range, it could indicate an error and the data should be flagged. These data should then be examined on a case-by-case basis during the data validation process. Several online sources of information about the natural abundance of elements are available that could be useful during the data verification phase. For example, WebElements.com (see Section 3.13) provides elemental

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TABLE 3.3 High and low values used by the STORET database since November 1983 to screen or error-check data

Parameter	High Value	Low Value
pH (standard units)	12	0.9
Specific conductance (µmhos/cm)	60,000	1.0
Dissolved oxygen (mg/L)	30	0
Hardness, total (mg/L as $CaCO_3$)	5,000	0
Alkalinity, total (mg/L as $CaCO_3$)	1,000	0
Acidity, total (mg/L as $CaCO_3$)	1,000	0
Dissolved organic carbon (mg/L as C)	100	0
Calcium, dissolved (mg/L)	1,000	0
Magnesium, dissolved (mg/L)	1,000	0
Sodium, dissolved (mg/L)	5,000	0
Potassium, dissolved (mg/L)	175	0
Chloride, total in water (mg/L)	22,000	0
Sulfate, dissolved (mg/L)	2,500	0
Arsenic, dissolved (µg/L)	5,000	0
Barium, dissolved (µg/L)	2,000	0
Cadmium, dissolved (µg/L)	500	0
Copper, dissolved (µg/L)	2,000	0
Lead, dissolved (µg/L)	1,000	0
Manganese, dissolved (µg/L)	5,000	0
Mercury, dissolved (µg/L)	10	0
Nickel, dissolved (µg/L)	2,000	0
Zinc, dissolved (µg/L)	25,000	0

Source: Adapted from NPS 2001.

abundance information for a variety of media. Abundance information is also compiled by Smith and Huyck (1999) for different types of sampling media.

Data usability is determined during the data validation process and should be conducted by personnel familiar with the site, study objectives, and intended applications of the data. Data validation involves a detailed examination of the data using professional judgment to determine if the data are adequate to meet the needs of the investigation.

The DQO process is essential to the proper application of a QA/QC program. Francoeur (1997) discusses the consequences of having a *disconnect* between DQOs, QA/QC, and the verification of data quality. He describes *data time bombs* that result from this disconnect. In these cases, laboratory data go directly from the analytical instrument to data reports in which conclusions are drawn. He also points out that the mere performance of QC procedures does not ensure valid or usable data in the laboratory report and states that "the DQO process is the only certain means by which the data user is certain to generate data of sufficient quality to meet an intended purpose" (Francoeur 1997).

Because contaminants can occur at much higher levels at mine sites and because there can be physical hazards at mine sites, it is good practice to also develop a site health and safety plan prior to conducting field investigations. Personal protective equipment and specific training requirements for health and safety need to be considered at the outset of the investigation.

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3.9 ANALYTICAL CHEMISTRY CONSIDERATIONS

Major causes of bias in environmental sampling and analysis include the following (USEPA 2006b):

- Nonrepresentative sampling
- Instability or contamination of samples between sampling and analysis
- Interferences and matrix effects in analysis
- Inability to determine the relevant forms of the parameter being measured
- Calibration
- Failure to blank-correct

It is not always appreciated how important sampling is in the analytical process. In many cases, sampling errors are the main contributor to the total error of the process (Gy 1998; Ramsey and Hewitt 2005), especially when trace constituents are being measured (see Section 3.4.8).

Selection of analytical methods is driven by the objectives of the investigation. Once the objectives have been identified, it is necessary to identify the appropriate sampling media and analytes of interest. Many federal and state regulations have established lists of parameters that may be required, but it is critical to also consider the ultimate application of the data. For example, if the objective of the investigation is to produce a risk assessment or provide data for input to a numerical model, then it is necessary to ensure that adequate data are collected and that the analytical detection limits are low enough to meet the requirements of these applications. Therefore, after establishing the media and analytes, selection of appropriate analytical methods needs to take into account the data needs of the investigation. It is important that personnel with adequate analytical chemistry expertise be involved in this planning phase of the investigation.

Appendix 3 provides a brief discussion of several analytical methods and media types. Some of the most popular references for analytical methods include *Standard Methods for the Examination of Water and Wastewater* (Rice et al. 2012), *Methods for Chemical Analysis of Water and Wastes* (USEPA 1983), *Test Methods for Evaluating Solid Waste (SW 846)* (USEPA, variously dated), and various ASTM methods (see also Appendix 2). The National Environmental Methods Index is an online database of method summaries that provides information to compare one method with another for the purpose of method selection, and can be used to search and compare regulatory and nonregulatory analytical methods (Keith et al. 2005; see Section 3.13). Some additional sources of online information are listed in Appendix 1. Compilations of methods can also be found in Dulski (1996), Crompton (2001), Csuros and Csuros (2002), Greenwood (2006), and Wagner (1998).

Deciding on appropriate analytical methods for an investigation involves several considerations, such as (1) analytes and detection limits needed; (2) suitability of sampling media for various analytical methods; (3) accuracy and precision needed; (4) limitations and interferences of various analytical instruments; (5) total or partial digestion, or direct analysis for solid materials; (6) need for nondestructive analysis; and (7) sample size limitations. Methods need to be sufficiently sensitive to reliably measure the concentration (i.e., adequate detection limits) and be free from serious interference problems. It is essential to ensure that detection limits are well below action levels and current regulatory guidelines for the contaminants of interest in the investigation. In the case of mineralized samples, upper detection limits also need to be considered when selecting analytical methods. It is essential that a person familiar with the data needs and applications, and the strengths and limitations of various analytical techniques, be involved in the choice of analytical methods for the investigation. It is also important to ensure that the user understands any method modifications or shortcuts employed by the laboratory.

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Some other common analytical issues to consider include matrix effects and interferences from concomitant elements; volatility of constituents during sample collection, preparation, decomposition, or analysis; incomplete decomposition of sample when total analysis is desired; laboratory subsampling; and contamination problems. All analytical methods are potentially subject to interference by the physical or chemical constituents of the sample. Types of analytical interferences include spectral (for techniques such as inductively coupled plasma–atomic emission spectroscopy [ICP-AES]), physical matrix (which affects introduction of the sample into an analytical instrument), and space-charge effects (e.g., lighter elements are lost in transmission). The more complex the sample matrix, the more likely it is that interferences can cause significant analytical problems.

ICP-AES has become a commonly used analytical technique because of its multi-element capabilities (generally 30–40 elements), its large linear range (4 to 5 orders of magnitude; ppm to ppb), and its rapid throughput of samples (Crompton 2001). When lower limits of detection are needed, inductively coupled plasma–mass spectroscopy (ICP-MS) is often used. ICP-MS can determine 60+ elements and has a linear range of 6 to 7 orders of magnitude with detection limits at the ppb (parts per billion) to ppt (parts per trillion) level (see Wolf 2005 for additional information). A drawback of the ICP techniques is that they require liquid samples, so solid samples must undergo decomposition. If sample decomposition poses problems, an alternative technique is instrumental neutron activation analysis, which is a nondestructive technique that can determine 30 to 40 elements with a linear range of 5 orders of magnitude (ppm to ppb levels) (Crompton 2001). Laser ablation ICP-MS requires no digestion and has less interferences than most techniques. Its real strength is in spatial mapping of elements and their ratios in grains, inclusions, coatings, and so on (see USGS 2013a for additional information). Combustion-infrared methods are also widely used for carbon and sulfur mineral estimation methods. See Appendix 3 for additional information on these techniques.

Determining constituents in sampling media generally necessitates sample preparation (e.g., drying, disaggregation, sieving, crushing, grinding), subsampling for analysis, sample decomposition, and sample analysis. Water samples generally do not require preparation other than filtration and preservation. For solid samples, sample preparation considerations might include the need for staged comminution and subsampling to prepare a laboratory sample for assay, selection of a particular size fraction, segregation of minerals of interest, contamination from equipment, and loss of elements (e.g., loss of mercury if drying exceeds about 60°C [140°F]). Sample decomposition can be total, partial, or selective, depending on the requirements of the analytical technique and the purpose of the analysis. Sample decomposition can be problematic if an element(s) of interest resides in minerals that resist decomposition techniques. It is advisable to consider sample decomposition techniques when choosing analytical methods. Some sample preparation, decomposition, and analytical methods are discussed in Appendices 2 and 3.

The importance of sample preparation and laboratory subsampling warrants more attention than they have received. The effects of sample heterogeneity, sampling error, and sampling theory need to be considered in sample preparation and laboratory subsampling. Sample preparation subsampling errors can occur because the portion of a field sample that the laboratory analyzes from the prepared sample is typically *much* smaller than the field sample (Ramsey and Suggs 2001). When a sample preparation subsample is collected to prepare the laboratory sample, it is important that each subsample taken during sample preparation and laboratory analysis be representative of the field sample. For example, it is poor practice to scoop a few grains of a solid sample from the top of the sample container. This poor practice can result in significant errors because the concentration of the analyte at the top of the container is likely different from that at

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the bottom of the container. Gerlach et al. (2003) found that subsampling accuracy was at least 2 orders of magnitude worse than the accuracy of most analytical methods. USEPA provides guidance on laboratory subsampling (Gerlach et al. 2002; Gerlach and Nocerino 2003). For solid samples, crushing and grinding prior to subsampling can be an effective way to reduce sampling error (e.g., Walsh et al. 2002). Several devices have been designed to split samples (e.g., rotary sample dividers and riffle splitters), and the advantages and disadvantages of various designs are discussed by Schumacher et al. (1990) and Gerlach et al. (2002).

Cost-effectiveness, which differs from absolute cost, needs to be considered when choosing analytical methods. Often, a small increase in cost to optimize sampling, sample preparation, or sample analysis can greatly improve the overall effectiveness of the sampling and monitoring program. Conversely, an expensive technique needs to be evaluated as to its benefit to the program. Also, less expensive techniques can be considered for screening purposes at the beginning of the sampling program so that more samples can be analyzed (see Triad Resource Center, n.d.).

Selection of a qualified analytical laboratory is a critical step and needs to be conducted by personnel who are familiar with the investigation and the necessary analytical chemistry methods and QA/QC requirements. The laboratory should have the certifications (e.g., USEPA, State) necessary for the investigation, if permitting or compliance is involved, have a good reputation, have the analytical methods and detection limits appropriate for the investigation, be in a location where samples can be easily shipped or delivered, and be flexible enough to be able to meet any special requirements necessary for the investigation. Laboratory certification is not a substitute for data verification and data validation by personnel familiar with the site and the data applications. A laboratory should never be selected based solely on their analytical costs.

3.10 CONTAMINATION CONSIDERATIONS

During and after collection, the integrity of the sample needs to be maintained. It is nearly impossible to eliminate all forms of contamination. The most common causes of sample contamination during sample collection include poor sample-handling techniques, input from atmospheric sources, inadequately cleaned equipment, and use of equipment constructed of materials inappropriate for the analytes targeted for study (USGS, variously dated). Sample preservation and sample preparation methods and equipment can also contribute potential contaminants. In general, an awareness of possible routes of contamination, use of appropriate inert containers for the analytes of interest, and sufficient blank samples can help avoid many contamination problems. It is often necessary to use specially prepared or cleaned sampling containers. For example, DOC samples require glass containers that have been baked at high temperature to eliminate organic materials, and metals samples require inert containers that have been precleaned with acid. Numerous ASTM, federal, and state guidelines are available that specify proper containers for different kinds of media. It is important to investigate any pertinent requirements. Sample containers also need to be inert to the preservatives being used.

It is important to employ good field practices and clean sampling methods, and to routinely collect QC samples. Table 3.4 lists some common sources of contamination for water quality samples. Gasparon (1998) discusses ways to minimize contamination of water samples by trace metals. USGS (variously dated) lists the following activities that constitute good field practices for water quality sampling:

- Be aware of and record potential contamination sources at field sites.
- Wear appropriate, disposable, powderless gloves (ensure that gloved hands do not come in contact with the water sample, and change gloves before each new step of sample collection and processing).

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TABLE 3.4 Common sources of contamination of water quality samples collected in the field

Contaminant Source Type	Examples
Sampling environment	Airborne particulates; precipitation; dust, soil, solid particles; fumes from engine exhaust, chemical preservatives, upwind industrial emissions
Sample-collection equipment	Pumps, isokinetic samplers, bailers, sample tubing, semipermeable membrane devices
Sample-processing equipment	Filtration devices, churn splitter, cone splitter, bottles, water (deionized/distilled water of ASTM grade 1 or better, tap, blank)
Sample-cleaning processes	Cleaning equipment (basins, brushes); carryover from cleaning solutions or tainted water; methanol carryover; insufficient decontamination or rinsing
Transport and shipping	Field vehicles; coolers or shipping containers; improperly closed or protected sample bottles
Storage	Warehouse; refrigerator; field vehicle; office laboratory; office storage space
Personnel	Dirty hands; sweat; sunscreen; DEET (active ingredient commonly used in insect repellents); nicotine, caffeine, and alcohol (breath); dirty gloves; gloved or ungloved contact with the sample to be analyzed; shedding clothing; hair and dandruff

Source: Adapted from USGS, variously dated.

- Use equipment constructed of materials that are relatively inert with respect to the analytes of interest.
- Use only equipment that has been precleaned according to prescribed procedures.
- Field-rinse equipment as directed.
- Use correct sample-handling procedures (minimize the number of sample-handling steps).
- Collect and process samples in enclosed chambers to minimize contamination from atmospheric sources.
- Collect sufficient blanks and other QC samples.
- Follow a prescribed order for collecting samples.

Human-induced contamination can originate from several sources such as sweat (which contains calcium, magnesium, lead, potassium, ammonium [NH_4^+], sulfate [SO_4^{2-}], phosphate [PO_4^{3-}], and cadmium for those who smoke), cosmetics (which can contain aluminum, beryllium, calcium, copper, chromium, potassium, iron, manganese, titanium, and zinc), eye makeup (which can contain mercury as a preservative), and dandruff shampoo (which can contain significant selenium) (see Gaines, n.d.-b).

Use of blanks can help identify contamination issues. It is good practice to prepare blanks for all steps of field sampling, sample handling, preservation, preparation, decomposition, and analysis. Specific types of blank samples are discussed in Section 3.8. It is advisable to sequence samples for analysis so that the blanks are analyzed first, which will prevent blanks from becoming contaminated by carryover contamination (i.e., samples with high concentrations contaminating those samples that follow) during analysis. Blank samples (e.g., pure deionized water) can also be used to test for carryover contamination by sequencing them throughout a set of samples. To be able to detect sample preparation and carryover contamination problems, it is necessary to know the order that samples were prepared and analyzed. If samples are randomly sequenced prior to preparation and analysis (a good practice), it is useful to first segregate them into groups expected to contain high concentrations versus low concentrations of analytes.

Use of chemical preservatives can be an important source of sample contamination. All sample preservatives need to be pre-approved by the analytical laboratory for the analytical techniques

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that will be used. It is good practice to use preservatives from the same lot for a given set of samples and record that information in the notes.

In collecting solid materials, contamination from sieves or other sampling apparatus needs to be considered. Also, contamination from a crushing and grinding apparatus can be an issue; common contaminants from tungsten carbide equipment can include tungsten, chromium, manganese, vanadium, cobalt, iron, copper, zirconium, and zinc (see Gaines, n.d.-a).

3.11 SAMPLE PRESERVATION, HANDLING, AND STORAGE CONSIDERATIONS

Sample preservation, handling, and storage can influence the integrity of the sample. Sample preservation is a very important consideration. Sample preservation by addition of chemical preservatives or refrigeration depends on the analyte and the analytical method to be used. All sample preservatives need to be pre-approved by the analytical laboratory for the analytical method and sample matrix. Sliwka-Kaszynska et al. (2003) provide a review of preservation and storage of water samples; Namiesnik and Szefer (2010) discuss considerations for preservation, storage, and analysis of water samples; and Biziuk et al. (2010) gives guidance about water-preservation methods.

Some issues that need to be considered during sampling, handling, and storage include

- Volatilization (and subsequent loss of analyte),
- Decomposition (agents can include temperature, ultraviolet radiation, microbial activity), and
- Chemical reactions (agents can include container walls, oxygen, carbon dioxide).

Some actions that can be taken to prevent these issues include

- Use of preservatives,
- Protecting samples from external agents, and
- Refrigeration or freezing.

For solid materials, analyte losses can occur during drying, crushing, grinding, decomposition, or heating, especially for volatile analytes. It is good practice to follow accepted preservation and storage protocols for various types of media. Appendix 3 provides discussions about different types of sampling media. Table 3.5 lists USEPA-recommended holding times and preservation methods for aqueous and solid samples. Some MIW samples may require special sample preservation procedures or shorter holding times.

3.12 SUMMARY

This chapter highlights information, resources, and research studies that relate to environmental sampling at mining and processing sites. Supplemental information relevant to this chapter can be found in Appendix 1 (online resources), Appendix 2 (ASTM methods), Appendix 3 (methods summaries), Appendix 5 (case studies), and Appendix 6 (geo-environmental models). This chapter is not a prescriptive guide for sampling at mining and processing sites. Rather, it points out selected sampling issues that need to be considered in the context of the sampling objectives for a given mining or processing site investigation. One of the most important aspects of sampling and monitoring is problem formulation (i.e., articulating the reason for sampling). This is a prevalent theme throughout this handbook and is also essential to the sampling considerations in this chapter. This chapter includes specialized sections about drillhole/borehole sampling considerations, biological (aquatic) sampling issues, microbial sampling considerations, and air sampling matters.

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TABLE 3.5 USEPA-recommended sample holding times and preservation for selected inorganic analyte determinations in aqueous and solid samples

Analyte	Matrix	Fraction	Preservation	Holding Time
Metals, except Hg and Cr(VI)	Aqueous	Total	HNO_3 to pH<2	6 months
		Dissolved	Filter on site; HNO_3 to pH<2	6 months
	Solid	Suspended	Filter on site	6 months
		Total	None	6 months
Hexavalent chromium	Aqueous		≤6°C (43°F)	24 hours
	Solid		≤6°C (43°F)	30 days to extraction; 7 days from extraction to analysis
Mercury	Aqueous	Total	HNO_3 to pH<2	28 days
		Dissolved	Filter; HNO_3 to pH<2	28 days
	Solid	Total	≤6°C (43°F)	28 days
Chloride	Aqueous		≤6°C (43°F)	28 days
Cyanide	Aqueous		≤6°C (43°F); NaOH to pH>12	14 days
	Solid		≤6°C (43°F)	14 days
Fluoride	Aqueous		≤6°C (43°F)	28 days
Nitrate	Aqueous		≤6°C (43°F)	28 days
pH	Aqueous		NA	Analyze immediately
	Solid		NA	Analyze immediately
Specific conductance	Aqueous		NA	Analyze immediately
Sulfate	Aqueous		≤6°C (43°F)	28 days
Sulfide	Aqueous		4 drops 2 N zinc acetate/ 100 mL sample; NaOH to pH>9; minimize aeration; store headspace free at ≤6°C (43°F)	7 days
	Solid		Fill sample surface with 2 N zinc acetate until moistened; store headspace free at ≤6°C (43°F)	7 days
Organic carbon, total	Aqueous		≤6°C (43°F); store in dark; HCl or H_2SO_4 to pH<2	28 days
	Solid		≤6°C (43°F)	28 days

Source: Adapted from USEPA 2007e.

The sampling step for solid materials is often the main contributor to the total error in the overall process, especially when trace constituents are being measured. Many modern analytical techniques have errors <5%, whereas poor sampling or sample preparation can result in errors >100%. A sample needs to be properly collected and prepared to be representative of the target population, and the target population needs to be carefully defined based on the objectives of the investigation. Sample representativeness can be addressed by using a sampling theory approach. The major source of uncertainty in environmental data sets is due to the heterogeneity of environmental materials, and use of sampling theory can help minimize these sampling errors. Sampling theory is discussed in detail in this chapter.

Knowledge of the geology of an area is fundamental to planning for sampling programs. Frequently, one objective of a sampling program is to determine background, baseline, or premining conditions. GEMs are a geology-based classification system for identifying environmental

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challenges associated with extraction of specific types of mineral deposits and can provide an initial prediction of water quality at a given mine site based on the type of mineral deposit. GEMs are discussed in this chapter and also in Chapter 2 and Appendix 6. Mineralogical and petrographic characterization is also a source of fundamental information for solid materials and can lend important insights into potential for acid generation, neutralization, and contaminant release. Several aspects of mineralogical characterization are discussed in this chapter.

Some water-sampling issues covered in this chapter include filtration, unstable constituents, and parameters and constituents that need to be measured in the field or processing facility. Temporal variation of metal concentrations in surface water can be significant in streams draining mined and mineralized areas and in treated process water, and this needs to be considered in the context of the sampling objectives. Stream-water flow/discharge measurements can be invaluable when comparing solute concentrations between different sampling events and are necessary to calculate the load of constituents in water. Groundwater/surface-water interactions can be important and need to be considered in the overall sampling plan when pathways are being identified. For groundwater sampling, the importance of well design and purging methods are discussed. Water balances are fundamental to the conceptualization of hydrologic systems at all scales. Developing a water balance includes collection of hydrological (e.g., flow or discharge measurements, groundwater flow parameters) and climatological (e.g., precipitation, temperature, evapotranspiration) information.

QA/QC procedures need to exist for all aspects of the sampling and monitoring program, including the field sampling program (collection, preservation, filtration, shipping, chain of custody, etc.), sample preparation, and the analytical procedures (laboratory). Selection of analytical methods is driven by the objectives of the investigation. Once the objectives have been identified, it is necessary to identify the appropriate sampling media and analytes of interest. During and after collection, the integrity of the sample needs to be maintained. Sample preservation, handling, preparation, and storage can influence the integrity of the sample, and are all discussed in this chapter.

3.13 INTERNET RESOURCES

General Water Resources

National Water Quality Monitoring Council: http://acwi.gov/monitoring/
USEPA Field Sampling Procedures: www.epa.gov/region9/qa/fieldsamp.html
USEPA Water Home Page: http://water.epa.gov/
USGS Water Resources of the United States: http://water.usgs.gov/
USGS Water Quality Information Pages: http://water.usgs.gov/owq/
Water on the Web: www.waterontheweb.org/

Groundwater Resources

Global Groundwater Information System: www.igrac.net/
USEPA Ground Water and Drinking Water: http://water.epa.gov/drink/
USEPA Ground Water: http://water.epa.gov/type/groundwater/
USGS Groundwater Information Pages: http://water.usgs.gov/ogw/
USGS Water Resources Groundwater Software: http://water.usgs.gov/software/lists/groundwater/

Specialized Resources

ASTM International: www.astm.org/
Biotic Ligand Model (BLM) Research Version: www.hydroqual.com/wr_blm.html
EnviroStat's Definition of *Multi Increment*: www.envirostat.org/multiincrementdefinition.htm

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National Environmental Methods Index: www.nemi.gov

Triad Resource Center: www.triadcentral.org

USEPA Agency-Wide Quality System Guidance Documents: www.epa.gov/quality/qa_docs.html

USEPA Decision Error Feasibility Trials (DEFT) software available for download: www.epa.gov/esd/databases/datahome.htm

USEPA Environmental Sampling: www2.epa.gov/region8/environmental-sampling

USEPA Soil Screening Guidance: www.epa.gov/superfund/health/conmedia/soil/index.htm

USGS Mineral Resources On-Line Spatial Data: http://mrdata.usgs.gov/

Visual Sample Plan (VSP) software tool available for download: http://vsp.pnnl.gov/

WebElements.com, Elemental Abundance Information: www.webelements.com/

Training

Contaminated-Site Clean-up Information (CLU-IN): www.clu-in.org

- Soil Sampling and Decision Making Using Incremental Sampling Methodology (recurring training)
- Hardrock Mining Geochemistry and Hydrology, Workshop 1 (archived at www.clu-in.org/conf/tio/r10hardrock/)
- Hardrock Mining Geochemistry and Hydrology, Workshop 2 (archived at www.clu-in.org/conf/tio/r10hardrock2/)
- Hardrock Mining Geochemistry and Hydrology, Workshop 3 (archived at www.clu-in.org/conf/tio/r10hardrock3_030513/)

EnviroStat: www.envirostat.org/training.htm

Interstate Technology and Regulatory Council (ITRC): www.itrcweb.org/ism-1/

Natural Water Quality Monitoring Council: http://acwi.gov/monitoring/webinars/

3.14 REFERENCES

AGDR (Australian Government Department of Resources). 2009. Airbourne contaminants, noise and vibration: Leading Practice Sustainable Development Program for the Mining Industry Report, www.ret.gov.au/resources/Documents/LPSDP/AirborneContaminantsNoiseVibrationHandbook_web.pdf. Accessed May 2013.

Al-Abed, S.R., Hageman, P.L., Jegadeesan, G., Madhavan, N., and Allen, D. 2006. Comparative evaluation of short-term leach tests for heavy metal release from mineral processing waste. *Sci. Total Environ*. 364:14–23.

Alaska Department of Environmental Conservation, Division of Spill Prevention and Response, Contaminated Sites Program. 2009. *Draft Guidance on Multi-Increment Soil Sampling*. www.dec.alaska.gov/spar/csp/guidance/multi_increment.pdf. Accessed May 2013.

Alaska DNR (Department of Natural Resources). 2013. Pebble project. Anchorage, AK: DNR. http://dnr.alaska.gov/mlw/mining/largemine/pebble/. Accessed 10/4/13.

Alley, W.M., Healy, R.W., LaBaugh, J.W., and Reilly, T.E. 2002. Flow and storage in groundwater systems. *Science* 296:1985–1990.

Alpers C.N., and Nordstrom, D.K. 1999. Geochemical modelling of water–rock interactions in mining environments. In *The Environmental Geochemistry of Mineral Deposits—Part A: Processes, Techniques, and Health Issues*. Reviews in Economic Geology, Vol. 6A. Littleton, CO: Society of Economic Geologists. pp. 289–324.

Alpers, C.N., and Nordstrom, D.K. 2000. Estimation of pre-mining conditions for trace metal mobility in mineralized areas—An overview. In *Proceedings, 5th International Conference on Acid Rock Drainage (ICARD 2000)*. Littleton, CO: SME. pp. 463–472.

Amy, P.S., and Haldeman, D.L., eds. 1997. *The Microbiology of the Terrestrial Deep Subsurface*. New York: CRC Lewis.

Appelo, C.A.J., and Postma, D. 2005. *Geochemistry, Groundwater and Pollution*, 2nd ed. Leiden, The Netherlands: A.A. Balkema.

ASTM D 4448-01. 2007. *Standard Guide for Sampling Ground-Water Monitoring Wells*. West Conshohocken, PA: ASTM International. Available from www.astm.org.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

ASTM D 4840-99. 2010. *Standard Guide for Sampling Chain-of-Custody Procedures*. West Conshohocken, PA: ASTM International. Available from www.astm.org.

ASTM D 6452-99. 2005. *Standard Guide for Purging Methods for Wells Used for Ground Water Quality Investigations*. West Conshohocken, PA: ASTM International. Available from www.astm.org.

Backhus, D.A., Ryan, J.N., Groher, D.M., McFarlane, J.K., and Gschwend, P.M. 1993. Sampling colloids and colloid-associated contaminants in ground water. *Ground Water*. 31(3):466–479.

Baedecker, M.J., and Cozzarelli, I.M. 1992. The determination and fate of unstable constituents of contaminated groundwater. In Lesage, Suzanne, and Jackson, R.E., eds., *Groundwater contamination and analysis at hazardous waste sites*. New York: Marcel Dekker. pp. 425–461.

Balistrieri, L.S., Seal, R.R., II, Piatak, N.M., and Paul, B. 2007. Assessing the concentration, speciation, and toxicity of dissolved metals during mixing of acid-mine drainage and ambient river water downstream of the Elizabeth Copper Mine, Vermont, USA. *Appl. Geochem*. 22:930–952.

Barbour, M.T., Gerritsen, J., Snyder, B.D., and Stribling, J.B. 1999. *Rapid Bioassessment Protocols for Use in Streams and Wadeable Rivers: Periphyton, Benthic Macroinvertebrates and Fish*, 2nd ed. EPA 841-B-99-002. Washington, DC: U.S. Environmental Protection Agency, Office of Water.

Barcelona, M.J., Gibb, J.P., Helfrich, J.A., and Garske, E.E. 1985. Practical Guide for Ground-Water Sampling. Champaign, IL: Illinois State Water Survey, SWS Contract Report 374 and U.S. Environmental Protection Agency EPA/600/S2-85/104. www.epa.gov/oust/cat/pracgw.pdf. Accessed May 2013.

Barcelona, M.J., Varlijen, M.D., Puls, R.W., and Kaminski, D. 2005. Ground water purging and sampling methods: History vs. hysteria. *Ground Water Monit. Remed*. 25:52–62.

BCMOE (British Columbia Ministry of Environment). 2012. Water and Air Baseline Monitoring Guidance Document for Mine Proponents and Operators, interim version. www.env.gov.bc.ca/epd/industrial/mining/pdf/water_air_baseline_monitoring.pdf. Accessed May 2013.

Bencala, K.E. 2000. Hyporheic zone hydrological processes: *Hydrol. Process*. 14:2797–2798.

Bencala, K.E. 2005. Hyporheic exchange flows. In *Encyclopedia of Hydrological Sciences*. Edited by M.G. Anderson. 3(10):1–7. New York: Wiley. http://water.usgs.gov/nrp/proj.bib/Publications/2005/bencala.2005.pdf. Accessed May 2013.

Bencala, K.E., Gooseff, M.N., and Kimball, B.A. 2011. Rethinking hyporheic flow and transient storage to advance understanding of stream-catchment connections. *Water Resources Res*. 47:W00H03.

Bervoets, L., Knaepkens, G., Eens, M., and R. Blust. 2005. Fish community responses to metal pollution. *Environ. Pollut*. 138:338–349.

Besser, J.M., and Leiby, K.J. 2007. Toxicity of metals in water and sediment to aquatic biota. In *Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado*. Edited by S.E. Church, P. von Guerard, and S.E. Finger. Professional Paper 1651. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/pp/1651/. Accessed May 2013.

Besser, J.M., Brumbaugh, W.G., May, T.W., Church, S.E., and Kimball, B.A. 2001. Bioavailability of metals in stream food webs and hazards to brook trout (*Salvelinus fontinalis*) in the upper Animas River Watershed, Colorado. *Arch. Environ. Contam. Toxicol*. 40:48–59.

Bish, D.L., and Howard, S.A. 1988. Quantitative phase analysis using the Rietveld method. *J. Appl. Crystallogr*. 21:86–91.

Bish, D.L., and Post, J.E., eds. 1989. *Modern Powder Diffraction*. Reviews in Mineralogy, Vol. 20. Washington, DC: Mineralogical Society of America.

Bish, D.L. and Post, J.E. 1993. Quantitative mineralogical analysis using the Rietveld full-pattern fitting method: *Am. Mineral*. 78:932–940.

Biziuk, M., Beyer, A., and Zukowska, J. 2010. Preservation and storage of water samples. In *Analytical Measurements in Aquatic Environments*. Edited by J. Namiesnik and P. Szefer. Taylor and Francis Group, CRC Press. pp. 19–39.

Borrok, D.M., Wanty, R.B., Ridley, W.I., Lamothe, P.J., Kimball, B.A., Verplanck, P.L., and Runkel, R.L. 2009. Distinguishing between natural and anthropogenic metal sources in a mountain watershed using iron and zinc isotopes. *Appl. Geochem*. 24:1270–1277.

Bove, D.J., Mast, M.A., Dalton, J.B., Wright, W.G., and Yager, D.B. 2007a. Major styles of mineralization and hydrothermal alteration and related solid- and aqueous-phase geochemical signatures. In *Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado*. Edited by S.E. Church, P. von Guerard, and S.E. Finger. Professional Paper 1651. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/pp/1651/. Accessed May 2013.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Bove, D.J., Yager, D.B., Mast, M.A., and Dalton, J.B. 2007b. *Alteration Map Showing Major Faults and Veins and Associated Water-Quality Signatures of the Animas River Watershed Headwaters near Silverton, Southwest Colorado*: Scientific Investigations Map 2976. pamphlet, 1 plate, scale 1:24,000. Reston, VA: U.S. Geological Survey.

Boyer, E.W., Hornberger, G.M., Bencala, K.E., and McKnight, D.M. 1997. Response characteristics of DOC flushing in an alpine catchment. *Hydrol. Processes*. 11:1635–1647.

Brumbaugh, W.G., Petty, J.D., Huckins, J.N., and Manahan, S.E. 2002. Stabilized liquid membrane device (SLMD) for the passive, integrative sampling of labile metals in water. *Water, Air, Soil Pollut*. 133:109–119.

Cabri, L.J., and Vaughan, D.J., eds. 1998. *Modern Approaches to Ore and Environmental Mineralogy*. Short Course Series, Vol. 27. Ottawa, Ontario: Mineralogical Association of Canada.

Callender, E. 2003. Heavy metals in the environment—Historical trends. *Treatise on Geochemistry*. 9:67–105.

Carlisle, D.M., and Clements, W.H. 1999. Sensitivity and variability of metrics used in biological assessment of running waters. *Environ. Toxicol. Chem*. 18(2):285–291.

Carlisle, D.M., Hawkins, C.P., Meador, M.R., Potapova, M., and Falcone, J. 2008. Biological assessment of Appalachian streams based on predictive models for fish, macroinvertebrate, and diatom assemblages. *J. N. Am. Benthol. Soc*. 27(1):16–37.

Carter, M.R., and Gregorich, E.G., eds. 2008. *Soil Sampling and Methods of Analysis*, 2nd ed. Canadian Society of Soil Science, Taylor and Francis Group, CRC Press.

Caruso, B.S., Cox, T.J., Runkel, R.L., Velleux, M.L., Bencala, K.E., Nordstrom, D.K., Julien, P.Y., Butler, B.A., Alpers, C.N., Marion, A., and Smith, K.S. 2008. Metals fate and transport modelling in streams and watersheds: State of the science and USEPA workshop review. *Hydrol. Processes.* 22:4011–4021.

Castendyk, D.N., and Eary, L.E., eds. 2009. *Mine Pit Lakes: Characteristics, Predictive Modeling, and Sustainability*. Management Technologies for Metal Mining Influenced Water series, Vol. 3. Littleton, CO: SME.

CDPHE (Colorado Department of Public Health and Environment). 2011. *Clear Creek*: Hazardous Materials and Waste Management Division, Central City/Clear Creek. www.colorado.gov/cs/Satellite/CDPHE-HM/CBON/1251615994932. Accessed May 2013.

Chapelle, F.H. 1993. *Ground-Water Microbiology and Geochemistry*. New York: John Wiley and Sons.

Chapin, T.P., and Todd, A.S. 2012. MiniSipper: A new in situ water sampler for high-resolution, long-duration acid mine drainage monitoring. *Sci. Total Environ*. 439:343–353.

Chen, W., Henizdo, E., Chen, J.-Q., Attfield, M.D., Gao, P., Hearl, F., Lu, J., and Wallace, W.E. 2005. Risk of silicosis in cohorts of Chinese tin and tungsten miners, and pottery workers (I): An epidemiological study. *Am. J. Ind. Med*. 48:1–9.

Choate, L.M., Ranville, J.F., Blumenstein, E.P., and Smith, K.S. 2008. Simple screening tests to determine the potential metal toxicity of mining-waste leachates. In *Understanding Contaminants Associated with Mineral Deposits*. Edited by P.L. Verplanck. Circular 1328, pp. 24–27. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/circ/1328/. Accessed May 2013.

Church, P.E., and Granato, G.E. 1996. Bias in ground-water data caused by well-bore flow in long-screen wells. *Ground Water* 34:262-273.

Church, S.E., Fey, D.L., and Unruh, D.M. 2007a. Trace elements and lead isotopes in modern streambed and terrace sediment—Determination of current and premining geochemical baselines. In *Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado*. Edited by S.E. Church, P. von Guerard, and S.E. Finger. Professional Paper 1651. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/pp/1651/. Accessed May 2013.

Church, S.E., Kirschner, F.K., Choate, L.M., Lamothe, P.J., Budahn, J.R., and Brown, Z.A. 2007b. *Determination of Premining Geochemical Background, Blue Creek Drainage, Stevens County, Washington*. Scientific Investigations Report 2007-5262. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/sir/2007/5262/. Accessed May 2013.

Church, S.E., von Guerard, P., and Finger, S.E., eds. 2007c. *Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado*. Professional Paper 1651. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/pp/1651/. Accessed May 2013.

Clark, J.L., and Clements, W.H. 2006. The use of in situ and stream microcosm experiments to assess population- and community-level responses to metals. *Environ. Toxicol. Chem*. 25:2306–2312.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Clark, R.N., Green, R.O., Swayze, G.A., Meeker, G., Sutley, S., Hoefen, T.M., Livo, K.E., Plumlee, G., Pavri, B., Sarture, C., Wilson, S., Hagemen, P., Lamothe, P., Vance, J.S., Boardman, J., Brownfield, I., Gent, C., Morath, L.C., Taggart, J., Theodorakos, P.M., and Adams, M. 2001. *Environmental studies of the World Trade Center area after the September 11, 2001 attack.* U.S. Geological Survey, Open-File Report 01-0429, http://pubs.usgs.gov/of/2001/ofr-01-0429/. Accessed May 2013.

Clements, W.H., and Kiffney, P.M. 1995. The influence of elevation on benthic community responses to heavy metals in Rocky Mountain streams. *Can. J. Fish. Aq. Sci.* 542:1966–1977.

Clements, W.H., Carlisle, D.M., Lazorchak, J.M., and Johnson, P.C. 2000. Heavy metals structure benthic communities in Colorado mountain streams: *Ecol. Appl*. 10:626–638.

Cochran, W.G. 1977. *Sampling Techniques*, 3rd ed. New York: John Wiley and Sons.

Cohen, D.R., and Ward, C.R. 1991. SEDNORM—A program to calculate a normative mineralogy for sedimentary rocks based on chemical analyses. *Computers Geosci.* 17:1235–1253.

Craig, J.R., and Vaughan, D.J. 1994. *Ore Microscopy and Ore Petrography*, 2nd ed. New York: John Wiley and Sons.

Cravotta, C.A., III. 1994. Secondary iron-sulfate minerals as sources of sulfate and acidity. In *Environmental Geochemistry of Sulfide Oxidation*, ACS Symposium Series, Vol. 550. Edited by C.N. Alpers and D.W. Blowes. Washington, DC: American Chemical Society.

Crompton, T.R. 2001. *Determination of Metals and Anions in Soils, Sediments, and Sludges*. London: Taylor and Francis Group, Spon Press.

Crumbling, D.M., Lynch, K., Howe, R., Groenjes, C., Shockley, J., Keith, L., Lesnik, B., Van E, J., and McKenna, J. 2001. Managing uncertainty in environmental decisions. *Environ. Sci. Technol*. 35:404A–409A.

Csuros, M., and Csuros, C. 2002. *Environmental Sampling and Analysis for Metals*. Boca Raton, FL: CRC Press, Lewis Publishers.

Custer, C.M, Yang, C., Crock, J.G., Shearn-Bochsler, V., Smith, K.S., and Hageman, P.L. 2009. Exposure of insects and insectivorous birds to metals and other elements from abandoned mine tailings in three Summit County drainages, Colorado. *Environ. Monit. Assess*. 153:161–177.

CWQCC (Colorado Water Quality Control Commission). 2010. *Aquatic Life Use Attainment—Methodology to Determine Use Attainment for Rivers and Streams*, Policy Statement 10-1, October 2010. Colorado Department of Public Health and Environment. www.colorado.gov/cs/Satellite?blobcol=urldata&blobheadername1=Content-Disposition&blobheadername2=Content-Type&blobheadervalue1=inline%3B+filename%3D%22Policy+10-1.pdf%22&blobheadervalue2=application%2Fpdf&blobkey=id&blobtable=MungoBlobs&blobwhere=1251853603204&ssbinary=true. Accessed May 2013.

Danielsson, L.G. 1982. On the use of filters for distinguishing between dissolved and particulate fractions in natural waters. *Water Res*. 16:179–182.

Degens, E.T., Kempe, S., and Richey, J.E., eds. 1991. *Biogeochemistry of Major World Rivers*, SCOPE Vol. 42. International Council of Scientific Unions, Scientific Committee on Problems of the Environment (SCOPE). www.scopenvironment.org/downloadpubs/scope42/index.html. Accessed May 2013.

Delvigne, J.E. 1998. *Atlas of Micromorphology of Mineral Alteration and Weathering*, Special Publication No. 3. Canadian Mineralogist.

Desborough, G.A., Smith, K.S., Lowers, H.A., Swayze, G.A., Hammarstrom, J.M., Diehl, S.F., Leinz, R.W., and Driscoll, R.L. 2010. Mineralogical and chemical characteristics of some natural jarosites. *Geochim. Cosmochim. Acta* 74:1041–1056.

Diehl, S.F., Hageman, P.L., and Smith, K.S. 2006. What's weathering? Mineralogy and field leach studies in mine waste, Leadville and Montezuma mining districts, Colorado. In *Proceedings, 7th International Conference on Acid Rock Drainage (ICARD 7)*. Lexington, KY: American Society of Mining and Reclamation. pp. 507–527. www.imwa.info/docs/imwa_2006/0507-Diehl-CO.pdf. Accessed May 2013.

Diehl, S.F., Koenig, A.E., Hageman, P.L., Smith, K.S., Fey, D.L., and Lowers, H.A. 2007. From the micro to the macro scale: A textural and chemical perspective of characterizing waste-rock material. In *Proceedings, Society for Mining, Metallurgy, and Exploration (SME) Annual Meeting and Exhibit, and the 109th National Western Mining Conference*, Preprint 07-021. Littleton, CO: SME.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Diehl, S.A., Hageman, P.L., and Smith, K.S. 2008. What is weathering in mine waste? Mineralogic evidence for sources of metals in leachates. In *Understanding Contaminants Associated with Mineral Deposits*. Edited by P.L. Verplanck. Circular 1328. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/circ/1328/. Accessed May 2013.

Diehl, S.F., Hageman, P.L., Koenig, A.E., Seal, R.R., II, Smith, K.S., Benzel, W.M., and Driscoll, R.L. 2010. Techniques to understand metal leaching from ore minerals in mine-waste material. In *Proceedings, SME Annual Meeting and Exhibit*. Littleton, CO: SME.

Di Toro, D.M., Allen, H.E., Bergman, H.L, Meyer, J.S., Paquin, P.R., and Santore, R.C. 2001. A biotic ligand model of the acute toxicity of metals. I. Technical basis. *Environ. Toxicol. Chem*. 20:2383–2396.

Downing, B. 2011. ARD sampling and sample preparation: *EnviroMine*. http://technology.infomine.com/enviromine/ard/sampling/intro.html. Accessed May 2013.

Drexler, J.W., and Brattin, W.J. 2007. An in vitro procedure for estimation of lead relative bioavailability: with validation: *Human Ecol. Risk Assess.* 13:383–401.

Druschel, G.K., Baker, B.J., Gihring, T.M., and Banfield, J.F. 2004. Acid mine drainage biogeochemistry at Iron Mountain, California. *Geochem. Trans*. 5(2):13–31.

du Bray, E.A., ed. 1995. *Preliminary Compilation of Descriptive Geoenvironmental Mineral Deposit Models*. Open-File Report 95-0831. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/of/1995/ofr-95-0831/. Accessed May 2013.

Dulski, T.R. 1996. *A Manual for the Chemical Analysis of Metals*. West Conshohocken, PA: ASTM International.

Eary, L.E., Jakubowski, R., Eshleman, J., and Watson, A. 2009. Applying numerical hydrogeochemical models as decision support tools for mine closure planning. In *Proceedings, Tailings and Mine Waste '08*. London: Taylor and Francis Group, CRC Press/Balkema.

ECA (European Chemicals Agency). 2008. *Guidance on information requirements and chemical safety assessment—Appendix R.7.13-2: Environmental risk assessment for metals and metal compounds*: European Chemicals Agency, Guidance for the implementation of REACH. http://echa.europa.eu/documents/10162/13632/information_requirements_r7_13_2_en.pdf. Accessed June 2013.

Edwards, T.K., and Glysson, G.D. 1999. Field methods for measurement of fluvial sediment. *Techniques of Water-Resources Investigations*, Book 3. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/twri/twri3-c2/. Accessed May 2013.

Environment Canada. 2009. *Environmental Code of Practice for Metal Mines*. www.ec.gc.ca/lcpe-cepa/default.asp?lang=En&n=CBE3CD59-1. Accessed May 2013.

EnviroStat. n.d. Definition of *Multi Increment*. www.envirostat.org/multiincrementdefinition.htm. Accessed 12/15/13.

Farag, A.M., Skaar, D., Nimick, D.A., MacConnell, E., and C. Hogstrand, C. 2003. Characterizing aquatic health using salmonid mortality, physiology, and biomass estimates in streams with elevated concentrations of arsenic, cadmium, copper, lead and zinc in the Boulder River watershed, Montana. *Trans. Am. Fish. Soc.* 132:450–467.

Fenter, P.A., Rivers, M.L., Sturchio, N.C., and Sutton, S.R., eds. 2002. *Applications of Synchrotron Radiation in Low-Temperature Geochemistry and Environmental Science*. Reviews in Mineralogy and Geochemistry, Vol. 49. Washington, DC: Mineralogical Society of America.

Fetter, C.W. 2000. *Applied Hydrogeology*, 4th ed. Upper Saddle River, NJ: Prentice Hall.

Ficklin, W.H., and Mosier, E.L. 1999. Field methods for sampling and analysis of environmental samples for unstable and selected stable constituents. In *The Environmental Geochemistry of Mineral Deposits—Part A: Processes, Techniques, and Health Issues*. Reviews in Economic Geology, Vol. 6A. Littleton, CO: Society of Economic Geologists. pp. 249–264.

Fleet, M.E. 2005. XANES spectroscopy of sulfur in earth materials. *Can. Mineral*. 43:1811–1838.

Foster A.L., Brown, G.E., Jr., Tingle, T., and Parks, G.A. 1998. Quantitative arsenic speciation in mine tailings using X-ray absorption spectroscopy. *Am Mineral.* 83:553–568.

Francoeur, T. L. 1997. Quality control: The great myth. In *Proceedings of the Field Analytical Methods for Hazardous Wastes and Toxic Chemicals Conference*, January 29–31, 1997, Las Vegas, NV. Pittsburgh, PA: Air and Waste Management Association. pp. 651–657.

Freeze, R.A., and Cherry, J.A. 1979. *Groundwater*. Upper Saddle River, NJ: Prentice Hall.

Fuller, C.C. and Harvey, J.W. 2000. Reactive uptake of trace metals in the hyporheic zone of a mining-contaminated stream, Pinal Creek, Arizona. *Environ. Sci. Technol*. 34:1150–1155.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Gaines, P. n.d.-a. Part 3: Sampling and subsampling. In *Trace Analysis Guide*. Web series. Christiansburg, VA: Inorganic Ventures. www.inorganicventures.com/sampling-and-subsampling. Accessed 10/4/13.

Gaines, P. n.d.-b. Part 10: Contamination from the analyst and apparatus. In *Trace Analysis Guide*. Web series. Christiansburg, VA: Inorganic Ventures. www.inorganicventures.com/tech/trace-analysis/contamination-from-the-analyst-and-apparatus. Accessed 10/4/13.

Gałuszka, A. 2007. A review of geochemical background concepts and an example using data from Poland. *Environ. Geol*. 52(5):861–870.

Gammons, C.H., Nimick, D.A., Parker, S.R., Cleasby, T.E., and McCleskey, R.B. 2005. Diel behavior of iron and other trace metals in a mountain stream with acidic to neutral pH: Fisher Creek, Montana, USA. *Geochim. Cosmochim. Acta* 69:2505–2516.

Gammons, C.H., Milodragovich, L., and Belanger-Woods, J. 2007. Influence of diurnal cycles on monitoring of metal concentrations and loads in streams draining abandoned mine lands: An example from High Ore Creek, Montana: *Environ. Geol*. 53:611–622.

Gandy, C.J., Smith, J.W.N., and Jarvis, A.P. 2007. Attenuation of mining derived pollutants in the hyporheic zone: A review: *Sci. Total Environ*. 373:435–446.

Gasparon, M. 1998. Trace metals in water samples: Minimizing contamination during sampling and storage. *Environ. Geol*. 36:207–214.

Gerlach, R.W., and Nocerino, J.M. 2003. *Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples*. EPA/600/R-03/027. Washington, DC: U.S. Environmental Protection Agency. www.epa.gov/osp/hstl/tsc/Gerlach2003.pdf. Accessed May 2013.

Gerlach, R.W., Dobb, D.E., Raab, G.A., and Nocerino, J.M. 2002. Gy sampling theory in environmental studies. 1. Assessing soil splitting protocols. *J. Chemometr*. 16:321–328.

Gerlach, R.W., Nocerino, J.M., Ramsey, C.A., and Venner, B.C. 2003. Gy sampling theory in environmental studies. 2. Subsampling error estimates. *Anal. Chim. Acta* 490:159–168.

Gilbert, R.O. 1987. *Statistical Methods for Environmental Pollution Monitoring*. New York: John Wiley and Sons.

Gilbert, R.O., and Pulsipher, B.A. 2005. Role of sampling designs in obtaining representative data. *Environ. Forensics*. 6:27–33.

Glasgow, H.B., Burkholder, J.M., Reed, R.E., Lewitus, A.J., and Kleinman, J.E. 2004. Real-time remote monitoring of water quality: A review of current applications and advancements in sensor, telemetry, and computing technologies: *J. Exper. Mar. Biol. Ecol*. 300:409–448.

Goldstein, J.N., Woodward, D.F., and Farag, A.M. 1999. Movements of adult Chinook salmon during spawning migration in a metals-contaminated system, Couer d'Alene River, Idaho. *Trans. Am. Fish. Soc*. 128:121–129.

Greenwood, R. 2006. Directory of "screening tools"—A toolbox of existing and emerging methods for chemical and ecological status monitoring under the Water Framework Directive. In *Screening Methods for Water Data Information in Support of the Implementation of the Water Framework Directive*, Deliverable D5.

GRI (Global Reporting Initiative). 2010. *The GRI Mining and Metals Sector Supplement*. Global Reporting Initiative. www.icmm.com/page/36353/gri-mining-and-metals-sector-supplement. Accessed May 2013.

Griffith, M.B., Lazorchak, J.M., and Herlihy, A.T. 2004. Relationships among exceedances of metals criteria, the results of ambient bioassays, and community metrics in mining-impacted streams. *Environ. Toxicol. Chem*. 23:1786–1795.

Gschwend, P.M., and Reynolds, M.D. 1987. Monodisperse ferrous phosphate colloids in an anoxic groundwater plume. *J. Contam. Hydrol*. 1:309–327.

Gusek, J.J., and Figueroa, L.A., eds. 2009. *Mitigation of Metal Mining Influenced Water*. Management Technologies for Metal Mining Influenced Water series, Vol. 2. Littleton, CO: SME.

Gy, P. 1982. *Sampling of Particulate Materials: Theory and Practice*, 2nd ed. New York: Elsevier Science and Technology.

Gy, P. 1998. *Sampling for Analytical Purposes*. New York: John Wiley and Sons.

Hageman, P.L. 2007. *U.S. Geological Survey Field Leach Test for Assessing Water Reactivity and Leaching Potential of Mine Wastes, Soils, and Other Geologic and Environmental Materials*. Techniques and Methods, book 5. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/tm/2007/05D03/pdf/TM5-D3_508.pdf. Accessed May 2013.

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Hageman, P.L., and Briggs, P.H. 2000. A simple field leach for rapid screening and qualitative characterization of mine waste material on abandoned mine lands. In *Proceedings, 5th International Conference on Acid Rock Drainage (ICARD 2000)*. Littleton, CO: SME. pp. 1463–1475. http://crustal.usgs.gov/projects/minewaste/pdfs/hageman1.pdf. Accessed May 2013.

Hagni, A.M. 2008. Phase identification, phase quantification, and phase association determinations utilizing automated mineralogy technology. *J. Min. Met. Mat. Soc*. 60(4):33–37.

Haimes, Y.Y. 1998. *Risk Modeling, Assessment, and Management*. New York: John Wiley and Sons.

Hall, J.S. 2004. New Mexico Bureau of Mines and Mineral Resource's clay laboratory manual. Unpublished report. Socorro, NM: New Mexico Bureau of Geology and Mineral Resources.

Hamilton, S.J., Mehrle, P.M., and Jones, J.R. 1987. Evaluation of metallothionein measurement as a biological indicator of stress from cadmium in brook trout. *Trans. Am. Fish. Soc*. 116:551–560.

Hammarstrom, J.M., and Smith, K.S. 2002. Geochemical and mineralogical characterization of solids and their effects on waters in metal-mining environments. In *Progress on Geoenvironmental Models for Selected Mineral Deposit Types*. Edited by R.R. Seal II, and N.K. Foley. Open-File Report 02-0195. Reston, VA: U.S. Geological Survey. pp. 8–54. http://pubs.usgs.gov/of/2002/of02-195/. Accessed May 2013.

Harper, D.D., Farag, A.M., and Brumbaugh, W.G. 2008. Effects of acclimation on the toxicity of stream water contaminated with zinc and cadmium to juvenile cutthroat trout. *Arch. Environ. Contam. Toxicol*. 54:697–704.

Harvey, J.W., and Bencala, K.E. 1993. The effect of streambed topography on surface-subsurface water exchange in mountain catchments. *Water Resour. Res.* 29:89–98.

Harvey, J.W., and Fuller, C.C. 1998. Effect of enhanced manganese oxidation in the hyporheic zone on basin-scale geochemical mass balance. *Water Resour. Res*. 34:623–636.

Hauff, P.L., Peters, D.C., Coulter, D.W., Sares, M.A., Bird, D.A., Henderson, F.B., III, and Prosh, E.C. 2006. Hyperspectral sensing of acid mine drainage—Two Colorado case studies. In *Proceedings, 7th International Conference on Acid Rock Drainage (ICARD 7)*. Lexington, KY: American Society of Mining and Reclamation. pp. 738–763. www.imwa.info/docs/imwa_2006/0738-Hauff-CO.pdf. Accessed May 2013.

Hawaii Department of Health. 2009. Section 4.2: Multi-Increment sample collection. In Technical Guidance Manual for the Implementation of the Hawai'i State Contingency Plan. www.hawaiidoh.org/tgm.aspx?p=0402a.aspx. Accessed 10/4/13.

Hawkins, C.P., Norris, R.H., Hogue, J.N., and Feminella, J.W. 2000. Development and evaluation of predictive models for measuring the biological integrity of streams. *Ecol. Appl*. 10:1456–1477.

Healy, R.W., and Scanlon, B.R. 2010. *Estimating Groundwater Recharge*. New York: Cambridge University Press.

Healy, R.W., Winter, T.C., LaBaugh, J.W., and Franke, O.L. 2007. *Water Budgets: Foundations for Effective Water-Resources and Environmental Management*. Circular 1308. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/circ/2007/1308/. Accessed May 2013.

Helsel, D.R. 2005. *Nondetects and Data Analysis, Statistics for Censored Environmental Data*. New York: John Wiley and Sons.

Hem, J. D. 1985. *Study and Interpretation of the Chemical Characteristics of Natural Water*, 3rd ed. Water Supply Paper 2254. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/wsp/wsp2254/. Accessed May 2013.

Hoal, K.O., Smith, K.S., Russell, C.C., Walton-Day, K., and Stammer, J.G. 2009a. Application of quantitative micro-mineralogy to tailings and mining waste. In *Proceedings, 13th International Conference on Tailings and Mine Waste (Tailings & Mine Waste '09)*, Banff, Alberta, Canada, November 1-4, 2009. Edited by D. Sego, M. Alostaz, and N. Beier. Alberta, Canada: University of Alberta, Department of Civil and Environmental Engineering. pp. 703–709.

Hoal, K.O., Stammer, J.G., Appleby, S.K., Botha, J., Ross, J.K., and Botha, P.W. 2009b. Research in quantitative mineralogy: Examples from diverse applications. *Miner. Eng*. 22:402–408.

Hoar, C.C. 2005. *Fish Response to Discharge Events from a Power Plant Cooling Reservoir in a River Affected by Acid Mine Drainage and Thermal Influences*. West Virginia University M.S. thesis.

Hochella, M.F. 1995. Mineral surfaces: Their characterization and their chemical, physical and reactive nature. In *Mineral Surfaces*, Mineralogical Society, Series 5. Edited by D.J. Vaughan and R.A.D. Pattrick. London: Chapman and Hall. pp. 17–60.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Horowitz, A.J. 2008. Determining annual suspended sediment and sediment-associated trace element and nutrient fluxes. *Sci. Total Environ*. 400:315–343.

Horowitz, A.J., Elrick, K.A., and Colberg, M.R. 1992. The effect of membrane filtration artifacts on dissolved trace element concentrations. *Water Res*. 26:753–763.

Horowitz, A.J., Demas, C.R., Fitzgerald, K.K., Miller, T.L., and Rickert, D.A. 1994. *U.S. Geological Survey Protocol for the Collection and Processing of Surface-Water Samples for the Subsequent Determination of Inorganic Constituents in Filtered Water*. Open- File Report 94-539. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/of/1994/0539/report.pdf. Accessed May 2013.

Horowitz, A.J., Lum, K.R., Garbarino, J.R., Hall, G.E.M., LeMieux, C., and Demas, C.R. 1996. Problems associated with using filtration to define dissolved trace element concentrations in natural water samples. *Environ. Sci. Technol*. 30:954–963.

Horsburgh, J.S., Jones, A.S., Stevens, D.K., Tarboton, D.G., and Mesner, N.O. 2010. A sensor network for high frequency estimation of water quality constituent fluxes Using surrogates. *Environ. Model. Software* 25:1031–1044.

ICMM (International Council on Mining and Metals). 2008. *Sustainable Development Framework: A Sustained Commitment to Improved Industry Performance*. London: International Council on Mining and Metals. www.icmm.com/document/429. Accessed May 2013.

Idowu, O.A. 2007. Hydrological processes of interaction between surface water and groundwater—A review. *ASSET Series B* 6:172–190.

INAP (International Network for Acid Prevention). 2009. *Global Acid Rock Drainage Guide* (GARD Guide). www.gardguide.com/. Accessed May 2013.

ITRC (Interstate Technology & Regulatory Council). 2012. Incremental Sampling Methodology. ISM-1. Washington, DC: Interstate Technology & Regulatory Council, Incremental Sampling Methodology Team. www.itrcweb.org/team/public?teamid=11. Accessed May 2013.

Jambor, J.L. 2003. Mine-waste mineralogy and mineralogical perspectives of acid-base accounting. In *Environmental Aspects of Mine Wastes*. Edited by J.L. Jambor, D.W. Blowes, and A.I.M. Ritchie. Ottawa, ON: Mineralogical Association of Canada. pp. 117–145.

Jambor, J.L., and Blowes, D.W. 1998. Theory and applications of mineralogy in environmental studies of sulfide-bearing mine wastes. In *Modern Approaches to Ore and Environmental Mineralogy*, Short Course Handbook, Vol. 27. Ottawa, ON: Mineralogical Association of Canada. pp. 367–401.

Jambor, J.L., Nordstrom, D.K., and Alpers, C.N. 2000. Metal-sulfate salts from sulfide mineral oxidation. In *Sulfate Minerals: Crystallography, Geochemistry, and Environmental Significance*, Reviews in Mineralogy and Geochemistry, Vol. 40. Edited by C.N. Alpers, J.L. Jambor, and D.K. Nordstrom. Washington, DC: Mineralogical Society of America and Geochemistry Society. pp. 303–350.

Jambor, J.L., Dutrizac, J.E., Groat, L.A., and Raudsepp, M. 2002. Static tests of neutralization potentials of silicate and aluminosilicate minerals. *Environ. Geol*. 43:1-17.

Jambor, J.L., Dutrizac, J.E., and Raudsepp, M. 2007. Measured and computed neutralization potentials from static tests of diverse rock types. *Environ. Geol*. 52:1173–1185.

Johnson, C.A., and Thornton, I. 1987. Hydrological and chemical factors controlling the concentrations of Fe, Cu, Zn, and As in a river system contaminated by acid mine drainage. *Water Resour*. 21:359–365.

Jordan, G. 2009. Sustainable mineral resources management: from regional mineral resources exploration to spatial contamination risk assessment of mining. *Environ. Geol*. 58(1):153–169.

Kearl, P.M., Korte, N.E., and Cronk, T.A. 1992. Suggested modifications to ground water sampling procedures based on observations from the colloidal borescope. *Ground Water Monit. Rev*. 12:155–160.

Kearl, P.M., Korte, N.E., Stites, M., and Baker, J. 1994. Field comparison of micropurging vs. traditional ground water sampling. Ground Water Monit. Remed. 14:183–190.

Keith, L.H. 1991. *Environmental Sampling and Analysis: A Practical Guide*. Boca Raton, FL: CRC Press, Lewis Publishers.

Keith, L.H., Brass, H.J., Sullivan, D.J., Boiani, J.A., and Alben, K.T. 2005. An introduction to the National Environmental Methods Index. *Environ. Sci. Technol*. 39:173A–176A.

Kelly, S.D., Hesterberg, D., and Ravel, B., eds. 2008. Analysis of soils and minerals using X-ray absorption spectroscopy. In *Methods of Soil Analysis*, Part 5. Mineralogical Methods. Madison, WI: Soil Science Society of America. pp. 387–464.

Kennedy, E.J. 1984. Discharge ratings at gaging stations: U.S. Geological Survey Techniques of Water-Resources Investigations, book 3. http://pubs.usgs.gov/twri/twri3-a10/. Accessed May 2013.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Kerr, P.F. 1977. *Optical Mineralogy*, 4th ed. New York: McGraw-Hill.

Kilpatrick, F.A., and Cobb, E.D. 1985. Measurement of discharge using tracers: U.S. Geological Survey Techniques of Water-Resources Investigations, book 3. http://pubs.usgs.gov/twri/twri3-a16/. Accessed May 2013.

Kim, C.S., Rytuba, J.J., and Brown, G.E., Jr. 2004. Geological and anthropogenic factors influencing mercury speciation in mine wastes: An EXAFS spectroscopy study. *Appl. Geochem*. 19:379–393.

Kimball, B.A. 1997. *Use of Tracer Injections and Synoptic Sampling to Measure Metal Loading from Acid Mine Drainage*. Fact Sheet 245-96. Reston, VA: U.S. Geological Survey. http://ut.water.usgs.gov/publications/fs245-96/). Accessed May 2013.

Kimball B.A., Runkel R.L., Walton-Day K., and Bencala K.E. 2002. Assessment of metal loads in watersheds affected by acid mine drainage by using tracer injection and synoptic sampling: Cement Creek, Colorado, USA. *Appl. Geochem*. 17:1183–1207.

Kimball, B.A., Walton-Day, K., and Runkel, R.L. 2007. Quantification of metal loading by tracer injection and synoptic sampling, 1996–2000. In *Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado*. Edited by S.E. Church, P. von Guerard, and S.E. Finger. Professional Paper 1651. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/pp/1651/. Accessed May 2013.

Kimball, B.A., Runkel, R.L., and Walton-Day, K. 2010. An approach to quantify sources, seasonal change, and biogeochemical processes affecting metal loading in streams: Facilitating decisions for remediation of mine drainage: *Appl. Geochem*. 25:728–740.

Klein, T.L., Thamke, J.N., Harper, D.D., Farag, A.M., and Nimick, D.A. 2003. *Water-Quality, Bed-Sediment, and Biological Data, for Streams in the Upper Prickly Pear Creek Watershed, Montana, 2001*. Open-File Report 03-032. Reston, VA: U.S. Geological Survey.

Kleinmann, R.L.P., ed. 2000. *Prediction of Water Quality at Surface Coal Mines*. Acid Drainage Technology Initiative. Morgantown, WV: The National Mine Land Reclamation Center.

Koch, G.S., Jr., and Link, R.F. 2002. *Statistical Analysis of Geological Data*. New York: Dover Publications.

Kwong, Y.T.J. 1993. *Prediction and Prevention of Acid Rock Drainage from a Geological and Mineralogical Perspective*. MEND Project 1.32.1. Ottawa, ON: Environment Canada. www.mend-nedem.org/reports/files/1.32.1.pdf. Accessed May 2013.

Kwong, Y.T.J. 2003. Comprehensive environmental ore deposit models as an aid for sustainable development. *Explor. Min. Geol*. 12:31–36.

Lapakko, K., and Berndt, M. 2003. Comparison of acid production from pyrite and jarosite. In *Proceedings, Sixth International Conference on Acid Rock Drainage (ICARD)*. Victoria, Australia: Australasian Institute of Mining and Metallurgy (AusIMM).

Lee, L., and Helsel, D. 2005. Baseline models of trace elements in major aquifers of the United States. *Appl. geochem*. 20(8):1560–1570.

LeGrand, H.E., and Rosen, L. 1998. Putting hydrogeologic site studies on track (editorial). *Ground Water* 36:193.

LeGrand, H.E., and Rosen, L. 2000. Systematic makings of early stage hydrogeologic conceptual models. *Ground Water* 38:887–893.

Lowers, H.A., Meeker, G.P., and Brownfield, I.K. 2005. *Analysis of Background Residential Dust for World Trade Center Signature Components Using Electron Microscopy and X-ray Microanalysis*. U.S. Geological Survey, Open-File Report 2005-1073. http://pubs.usgs.gov/of/2005/1073/pdf/OF2005-1073_508.pdf. Accessed May 2013.

Madrid, Y., and Zayas, Z.P. 2007. Water sampling: Traditional methods and new approaches in water sampling strategy. *Trends Anal. Chem*. 26:293–299.

Maest, A.S., Nordstrom, D.K., and LoVetere, S.H. 2004. *Questa Baseline and Pre-Mining Ground-Water Quality Investigation. 4. Historical Surface-Water Quality for the Red River Valley, New Mexico, 1965 to 2001*. Scientific Investigations Report 2004-5063. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/sir/2004/5063/. Accessed May 2013.

Maney, J.P., and Wait, A.D. 2005. The role of representativeness in the project life cycle. *Environ. Forensics*. 6:17–19.

Manning, A.H., Verplanck, P.L., Mast, M.A., and Wanty, R.B. 2008. *Hydrogeochemical Investigation of the Standard Mine Vicinity, Upper Elk Creek Basin, Colorado*. Scientific Investigations Report 2007–5265. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/sir/2007/5265/. Accessed May 2013.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Mason, B.J. 1992. *Preparation of Soil Sampling Protocols: Sampling Techniques and Strategies*. EPA/600/R-92/128. Washington, DC: U.S. Environmental Protection Agency, Office of Research and Development. www.epa.gov/oust/cat/mason.pdf. Accessed May 2013.

McCarthy, J., and Shevenell, L. 1998. Obtaining representative ground-water samples in a fractured and karstic formation. *Ground Water* 36:251–260.

McCarthy, J.F. and Zachara, J.M. 1989. Subsurface transport of contaminants. *Environ. Sci. Technol*. 5(23):496–502.

McDermott, H.J. 2004. *Air Monitoring for Toxic Exposures*, 2nd ed. New York: John Wiley and Sons.

McGee, J.J., and Klaus, K. 2001. Application of electron probe microanalysis to the study of geological and planetary materials. *Micros. Microanal*. 7:200–210.

McKnight, D.M., and Bencala, K.E. 1990. Chemistry of iron, aluminum, and dissolved organic material in three acidic, metal-enriched, mountain streams, as controlled by watershed and in-stream processes. *Water Resour. Res*. 26:3087–3100.

McKnight, D.M., Kimball, B.A., and Bencala, K.E. 1988. Iron photoreduction and oxidation in an acidic mountain stream. *Science* 240:637–640.

McLemore, V.T., Smith, K.S., Russell, C.C., and the Sampling and Monitoring Committee of the Acid Drainage Technology Initiative-Metal Mining Sector (ADTI-MMS). 2007. Sampling and monitoring for closure. Reviews in Engineering Geology, Vol. 17. In *Understanding and Responding to Hazardous Substances at Mine Sites in the Western United States*. Edited by J.V. DeGraff. Boulder, CO: Geological Society of America. pp. 171–180.

McLemore, V., Heizler, L., Donahue, K., and Dunbar, N. 2009a. Characterization of weathering of mine rock piles: Example from the Questa Mine, New Mexico, USA. In *Proceedings, Securing the Future and 8th International Conference on Acid Rock Drainage (ICARD)*. Sweden: SveMin. http://geoinfo.nmt.edu/staff/mclemore/documents/Virginia_McLemore_P_T2_Characterization-of-weathering-of-mine-rock-piles-example-from-the-Qu.pdf. Accessed May 2013.

McLemore, V., Sweeney, D., Dunbar, N., Heizler, L. and Phillips, E. 2009b. Determining quantitative mineralogy using a combination of petrographic techniques, whole rock chemistry, and MODAN. Preprint 09-20. In *Proceedings of the SME Annual Meeting and Exhibit*. Littleton, CO: SME.

Meeker, G.P., Bern, A.M., Lowers, H.A., and Brownfield, I.K. 2005. *Determination of a Diagnostic Signature for World Trade Center Dust Using Scanning Electron Microscopy Point Counting Techniques*. U.S. Geological Survey, Open-File Report 2005-1031. http://pubs.usgs.gov/of/2005/1031/pdf/OF2005_1031_508.pdf. Accessed May 2013.

Milhous, R.T. 2007. Application of physical habitat simulation in the evaluation of physical habitat suitability. In *Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado*. Edited by S.E. Church, P. von Guerard, and S.E. Finger. Professional Paper 1651. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/pp/1651/. Accessed May 2013.

Miranda, M., Chambers, D., and Coumans, C. 2005. *Framework for Responsible Mining: A Guide to Evolving Standards*. Center for Science in Public Participation (CSP2). www.frameworkforresponsiblemining.org. Accessed May 2013.

Moore, O.M., and Reynolds, R.O., Jr. 1989. *X-ray Diffraction and the Identification and Analyses of Clay Minerals*. New York: Oxford University Press.

Munroe, E.A. 1999. Geology and geochemistry of waste rock piles in the Hillsboro mining district, Sierra County, New Mexico. M.S. thesis, New Mexico Institute of Mining and Technology, Socorro.

Munroe, E.A., McLemore, V.T., and Kyle, P. 1999. Waste rock pile characterization, heterogeneity, and geochemical anomalies in the Hillsboro mining district, Sierra County, New Mexico. *J. Geochem. Explor*. 67:391–405.

Munroe, E.A., McLemore, V.T., and Dunbar, N.W. 2000. Mine waste rock pile geochemistry and mineralogy in southwestern New Mexico, USA, Vol. II. In *Proceedings, 5th International Conference on Acid Rock Drainage (ICARD 2000)*. Littleton, CO: SME. pp. 1327–1336.

Muste, M., Vermeyen, T.R., Hotchkiss, R., and Oberg, K.A. 2007. Acoustic velocimetry for riverine environments. *J. Hydraul. Eng*. 133:1297–1298.

Myers, J.C. 1997. *Geostatistical Error Management: Quantifying Uncertainty for Environmental Sampling and Mapping*. New York: Van Nostrand Reinhold.

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Nagorski, S.A., Moore, J.N., McKinnon, T.E., and Smith, D.B. 2003. Scale-dependent temporal variations in stream water geochemistry. *Environ. Sci. Technol*. 37:859–864.

Namiesnik, J., and Szefer, P., eds. 2010. *Analytical Measurements in Aquatic Environments*. Boca Raton, FL: CRC Press.

Nesse, W.D. 2004. *Introduction to Optical Mineralogy*, 3rd ed. New York: Oxford University Press.

Neubert, J.T. 2000. *Naturally Degraded Surface Waters Associated with Hydrothermally Altered Terrane in Colorado*. Open-File Report 00–16, CD-ROM pub. Denver, CO: Colorado Geological Survey.

Neuendorf, K.K.E., Mehl, J.P., Jr., and Jackson, J.A. 2005. *Glossary of Geology*, 5th ed. Alexandria, VA: American Geological Institute.

Nielsen, D.M., ed. 2006. *Practical Handbook of Environmental Site Characterization and Ground-Water Monitoring*, 2nd ed. Boca Raton, FL: CRC Press.

Nielsen, D.M., and Nielsen, G.L. 2007. *The Essential Handbook of Ground-Water Sampling*. Boca Raton, FL: CRC Press.

Nimick, D.A., Gammons, C.H., Cleasby, T.E., Madison, J.P., Skaar, D., and Brick, C.M. 2003. Diel cycles in dissolved metal concentrations in streams: Occurrence and possible causes. *Water Resour. Res*. 39:citation1247.

Nimick, D.A., Cleasby, T.E., and McCleskey, R.B. 2005. Seasonality of diel cycles of dissolved trace-metal concentrations in a Rocky Mountain stream. *Environ. Geol*. 47:603–614.

Nimick, D.A., Gammons, C.H., and Parker, S.R. 2011. Diel biogeochemical processes and their effect on the aqueous chemistry of streams: A review. *Chem. Geol*. 283:3–17.

Niyogi, D.K., Lewis, W.M., and D.M. McKnight. 2002. Effects of stress from mine drainage on diversity, biomass, and function of primary producers in mountain streams. *Ecosystems* 5:554–567.

Nolan, K.M, Gray, J.R., and Glysson, G.D. 2005. *Introduction to Suspended-Sediment Sampling*. Scientific Investigations Report 2005-5077, CD-ROM pub. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/sir/2005/5077/. Accessed May 2013.

Nordstrom, D.K. 2004. Modeling low-temperature geochemical processes. In *Surface and Ground Water, Weathering, and Soils: Treatise on Geochemistry*. Edited by J.I. Drever. Amsterdam: Elsevier. pp. 37–72.

Nordstrom, D.K. 2008. *Questa Baseline and Pre-Mining Ground-Water Quality Investigation. 25. Summary of Results and Baseline and Pre-Mining Ground-Water Geochemistry, Red River Valley, Taos County, New Mexico, 2001-2005*. Professional Paper 1728. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/pp/1728/pdf/p1728_508.pdf. Accessed May 2013.

Nordstrom, D. K. 2011. Hydrogeochemical processes governing the origin, transport and fate of major and trace elements from mine wastes and mineralized rock to surface waters. *Applied Geochemistry*. 26(11):1777-1791.

Nordstrom, D.K., and Alpers, C.N. 1999. Geochemistry of acid mine waters. In *The Environmental Geochemistry of Mineral Deposits—Part A: Processes, Techniques, and Health Issues*. Reviews in Economic Geology, Vol. 6A. Littleton, CO: Society of Economic Geologists. pp. 133–160.

Nordstrom, D.K., and Munoz, J.L. 2006. *Geochemical Thermodynamics*, 2nd ed. Blackburn Press.

Nordstrom, D.K., and Wilde, F.D. 2005. Reduction-oxidation potential (electrode method). In *Field Measurements: U.S. Geological Survey Techniques of Water-Resources Investigations, book 9*. Edited by F.D. Wilde. http://water.usgs.gov/owq/FieldManual/Chapter6/6.5_v_1.2.pdf. Accessed May 2013.

Nordstrom, D.K., Nicholson, A., Verberg, R., and Mayer, U., eds. Forthcoming. *Geochemical Modeling for Mine Site Characterization and Remediation of Mine Wastes*. Management Technologies for Metal Mining Influenced Water series, Vol. 4. Englewood, CO: SME.

NPS (National Park Service). 2001. Appendix C, STORET water quality control/edit checking. In *Baseline Water Quality Data Inventory and Analysis—Rocky Mountain National Park*. Technical Report NPS/NRWRD/NRTR-2000/268. Washington, DC: National Park Service.

Nuffer, L.L., Hassig, N.L., Sego, L.H., Pulsipher, B.A., Wilson, J.E., and Matzke, B. 2009. *Validation of Statistical Sampling Algorithms in Visual Sample Plan (VSP): Summary Report*. Report PNNL-18253. Pacific Northwest National Laboratory. www.pnl.gov/main/publications/external/technical_reports/PNNL-18253.pdf. Accessed May 2013.

Nuffield, E.W. 1966. *X-ray Diffraction Methods*. New York: Wiley.

Oberg, K.A., and Mueller, D.S. 2007. Validation of streamflow measurements made with Acoustic Doppler Current Profilers. *J. Hydraul. Eng*. 133:1421–1432.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Oerter, E., Brimhall, G.H., Jr., Redmond, J., and Walker, B. 2007. A method for quantitative pyrite abundance in mine rock piles by powder X-ray diffraction and Rietveld method. *Appl. Geochem*. 22:2907–2925.

Paktunc, A.D. 1998. MODAN: An interactive computer program for estimating mineral quantities based on bulk composition. *Computers Geosci*. 24:425–431.

Paktunc, A.D. 2001. MODAN—A Computer program for estimating mineral quantities based on bulk composition: Windows version. *Computers Geosci*. 27:883–886.

Parbhakar, A., Edraki, M., Bradshaw, D., and Walters, S. 2009. Mineralogical characterization techniques for predicting acid rock drainage. In *Proceedings, Securing the Future and 8th International Conference on Acid Rock Drainage (ICARD)*. Sweden: SveMin.

Parker, L.V. 1994. The effects of ground water sampling devices on water quality: A literature review. *Ground Water Monit. Remed*. 14:130–141.

Piatak, N.M., Seal II, R.R., and Hammarstrom, J.M. 2004. Mineralogical and geochemical controls on the release of trace elements from slag produced by base-and precious-metal smelting at abandoned mine sites. *Appl. Geochem*. 19(7):1039–1064.

Pirrie, D., and Rollinson, G.K. 2011. Unlocking the applications of automated mineral analysis. *Geology Today*. 27:226-235.

Pirrie, D., Butcher, A.R., Power, M.R., Gottlieb, P., and Miller, G.L. 2004. Rapid quantitative mineral and phase analysis using automated scanning electron microscopy (QEMSCAN); Potential applications in forensic geoscience. In *Forensic Geoscience; Principles, Techniques and Applications*, Geological Society Special Publication 232. London: The Geological Society. pp. 123–136.

Pirrie, D., Power, M.R., Rollinson, G.K., Wiltshire, P.E.J., Newberry, J., Campbell, H.E. 2009. Automated SEM-EDS (QEMSCAN®) mineral analysis in forensic soil investigations: Testing instrumental reproducibility. In *Criminal and Environmental Soil Forensics*. London: Springer Science. pp. 411–430.

Pitard, F.F. 1993. *Pierre Gy's Sampling Theory and Sampling Practice: Heterogeneity, Sampling Correctness, and Statistical Process Control*, 2nd ed. Boca Raton, FL: CRC Press.

Pitard, F.F. 2010. Theoretical, practical, and economic difficulties in sampling for trace constituents. *J. S. Afr. Inst. Min. Metall*. 110:313–321.

Plumlee, G. S. 1999. The environmental geology of mineral deposits. In *The Environmental Geochemistry of Mineral Deposits—Part A: Processes, Techniques, and Health Issues*. Reviews in Economic Geology, Vol. 6A. Littleton, CO: Society of Economic Geologists. pp. 71–116.

Plumlee, G. 2009. Report from ground zero—How geoscientists aid in the aftermath of environmental disasters. *Earth Mag*. October. American Geological Institute. www.earthmagazine.org/earth/article/278-7d9-a-1. Accessed May 2013.

Plumlee, G.S. and Nash, J.T. 1995. Geoenvironmental models of mineral deposits—Fundamentals and applications. In *Preliminary Compilation of Descriptive Geoenvironmental Mineral Deposit Models*. Open-File Report 95-0831. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/of/1995/ofr-95-0831/. Accessed May 2013.

Plumlee, G.S., Smith, K.S., and Ficklin, W.H. 1994. *Geoenvironmental Models of Mineral Deposits, and Geology-Based Mineral-Environmental Assessments of Public Lands*. Open-File Report 94-203. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/of/1994/ofr-94-0203/94-203.pdf. Accessed May 2011.

Plumlee, G.S., Streufert, R.K., Smith, K.S., Smith, S.M., Wallace, A.R., Toth, M., Nash, J.T., Robinson, R.A., Ficklin, W.H., and Lee, G.K. 1995. *Map Showing Potential Metal-Mine Drainage Hazards in Colorado, Based on Mineral-Deposit Geology* (1 over-size sheet, scale 1:750,000). Open-File Report 95-26. Reston, VA: U.S. Geological Survey. Map Series 29. Denver, CO: Colorado Geological Survey. http://geosurveystore.state.co.us/p-986-potential-metal-mine-drainage-hazards-in-co-based-on-mineral-deposit-geology.aspx. Accessed May 2013.

Plumlee G.S., Smith, K.S., Montour, M.R., Ficklin, W.H., and Mosier, E.L. 1999. Geologic controls on the composition of natural waters and mine waters draining diverse mineral-deposit types. In *The Environmental Geochemistry of Mineral Deposits—Part B: Case Studies and Research Topics*. Reviews in Economic Geology, Vol. 6B. Littleton, CO: Society of Economic Geologists. pp. 373–432.

Power, M. 2002. Assessing fish population response to stress. In *Biological Indicators of Aquatic Ecosystem Stress*. Edited by S.M. Adams. Bethesda, MD: American Fisheries Society.

Price, W.A. 2009. *Prediction Manual for Drainage Chemistry from Sulphidic Geologic Materials*. MEND Report 1.20.1. Ottawa, ON: Natural Resources C www.mend-nedem.org/reports/files/1.20.1.pdf. Accessed May 2013.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Puls, R.W., and M.J. Barcelona. 1989. Filtration of ground water samples for metals analyses. *Haz. Waste Haz. Mater*. 6(4):385–393.

Puls, R.W., and Barcelona, M.J. 1996. *Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures*. Ground Water Issue. EPA/540/S-95/504. Washington, DC: U.S. Environmental Protection Agency. www.epa.gov/superfund/remedytech/tsp/download/lwflw2a.pdf. Accessed May 2013.

Puls, R.W., and Powell, R.M. 1992. Acquisition of representative ground water quality samples for metals. *Ground Water Monit. Rev*. 12:167–176.

Puls, R.W., J.H. Eychaner, and R.M. Powell. 1990. *Colloidal-Facilitated Transport of Inorganic Contaminants in Ground Water: Part I. Sampling Considerations*. EPA/600/M-90/023. NTIS PB 91-168419. Washington, DC: U.S. Environmental Protection Agency.

Puls, R.W., D.A. Clark, B. Bledsoe, R.M. Powell, and C.J. Paul. 1992. Metals in Ground Water: Sampling Artifacts and Reproducibility. *Haz. Waste Haz. Mater*. 9(2):149–162.

Rainbow, P.S. 2007. Trace metal bioaccumulation: Models, metabolic availability and toxicity. *Environ. Int*. 33:576–582.

Ramsey, C.A., and Hewitt, A.D. 2005. A methodology for assessing sample representativeness. *Environ. Forensics* 6:71–75.

Ramsey, C.A., and Suggs, J. 2001. Improving laboratory performance through scientific subsampling techniques. *Environ. Test. Anal*. 10:12–16.

Rantz, S.E., and others. 1982. *Measurement and Computation of Streamflow: Volume 1. Measurement of Stage and Discharge. Volume 2. Computation of Discharge*. Water-Supply Paper 2175. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/wsp/wsp2175/. Accessed May 2013.

Ranville, J.F. and Schmiermund, R.L. 1999. General aspects of aquatic colloids in environmental geochemistry. In *The Environmental Geochemistry of Mineral Deposits—Part A: Processes, Techniques, and Health Issues. Reviews in Economic Geology*, Vol. 6A. Littleton, CO: Society of Economic Geologists. pp. 183–199.

Ranville, J., Blumenstein, E., Adams, M., Choate, L., Smith, K., and Wildeman, T. 2006. Integrating bioavailability approaches into waste rock evaluations. In *Proceedings, 7th International Conference on Acid Rock Drainage (ICARD 7)*. Lexington, KY: American Society of Mining and Reclamation. pp. 1642–1653. www.imwa.info/docs/imwa_2006/1642-Ranville-CO.pdf. Accessed May 2013.

Reed, S.J.B. 1996. *Electron Microprobe Analysis and Scanning Electron Microscopy in Geology*. New York: Cambridge University Press.

Reimann, C., and Garrett, R.G. 2005. Geochemical background—concept and reality. *Scie. Total Environ*. 350(1):12–27.

Reynolds, J.B. 1996. Electrofishing. In *Fisheries Techniques*, 2nd ed. Edited by B.R. Murphy and D.W. Willis. Bethesda, MD: American Fisheries Society.

Rice, E.W., Baird, R.B., Eaton, A.D., and Clesceri, L.S., eds. 2012. *Standard Methods for the Examination of Water and Wastewater*, 22nd edition. Washington, D.C.: American Public Health Association, American Water Works Association, and Water Environment Federation.

Ritchey, J. 2002. Low-flow purging and sampling ground water, evolution of technology and standards. *ASTM Stand. News*, April 2002, pp. 18–23.

Riviere, J.C., and Myhra, S., eds. 2009. *Handbook of Surface and Interface Analysis—Methods for Problem-Solving*, 2nd ed. Boca Raton, FL: CRC Press.

Rockwell, B.W. 2011. *Description and Validation of an Automated Methodology for Mapping Mineralogy, Vegetation, and Hydrothermal Alteration Type from ASTER Satellite Imagery with Examples from the San Juan Mountains, Colorado*. Scientific Investigations Map 3190. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/sim/3190/. Accessed May 2013.

Rockwell, B.W. 2013. Comparative mineral mapping in the Colorado Mineral Belt using AVIRIS and ASTER remote sensing data: U.S. Geological Survey Scientific Investigations Map 3256. pamphlet, 1 map sheet, scale 1:150,000, http://pubs.usgs.gov/sim/3256/. Accessed July 2013.

Roig, B. 2007. Report on laboratory and field validations of screening tools based on performance criteria evaluation. In *Screening Methods for Water Data Information in Support of the Implementation of the Water Framework Directive*, Deliverable D44.

Rollinson, H.R. 1993. *Using Geochemical Data: Evaluation, Presentation, Interpretation*. Harlow, England: Pearson Education Ltd., Prentice Hall, Longman Group.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Ruby, M.V., Davis, A., Link, T.E., Schoof, R., Chaney, R.L., Freman, G.B., and Bergstrom, P. 1993. Development of an in vitro screening test to evaluate the in vivo bioaccessibility of ingested mine-waste lead. *Environ. Sci. Technol*. 27(13):2870–2877.

Runkel, R.L., Kimball, B.A., Walton-Day, K., and Verplanck, P.L. 2007. A simulation-based approach for estimating premining water quality: Red Mountain Creek, Colorado. *Appl. Geochem*. 22(9):1899–1918.

Runnells, D.D., Shepard, T.A., Angino, E.E. 1992. Metals in water: determining natural background concentrations in mineralized areas. *Environ. Sci. Technol*. 26:2316–2322.

Runnells, D.D., Shields, M.J., and Jones, R.L. 1997. Methodology for adequacy of sampling of mill tailings and mine waste rock. In *Proceedings, Tailings and Mine Waste '97*. Rotterdam: Balkema. pp. 561–563.

Russell, C. 2006. Mine drainage challenges from A to Z in the United States. In *Proceedings, 7th International Conference on Acid Rock Drainage (ICARD 7)*. Lexington, KY: American Society of Mining and Reclamation. pp. 1785–1802. www.asmr.us/Publications/Conference%20Proceedings/2006/1785-Russell-CO.pdf. Accessed May 2013.

Santore, R.C., Di Toro, D.M., Paquin, P.R., Allen, H.E., and Meyer, J.S. 2001. Biotic ligand model of the acute toxicity of metals. 2. Application to acute copper toxicity in freshwater fish and Daphnia. *Environ. Toxicol. Chem*. 20:2397–2402.

Schmidt, T.S., Clements, W.H., Wanty, R.B., Verplanck, P.L., Church, S.E., San Juan, C.A., and Klein, T.L. 2012. Geologic processes influence the effects of mining on aquatic ecosystems. *Ecolog. Appl*. 22(3):870–879.

Schmiermund, R.L., and Drozd, M.A. 1997. Acid mine drainage and other mining influenced waters (MIW). In *Mining Environmental Handbook*. Edited by J.J. Marcus. London: Imperial College Press. pp. 599–617.

Schmiermund, R.L., and Ranville, J. 2006. Application of geo-environmental and biotic ligand models in the life cycle of mine development—A flowchart for integration. In *Proceedings, 7th International Conference on Acid Rock Drainage (ICARD 7)*. Lexington, KY: American Society of Mining and Reclamation. pp. 1920–1925. www.imwa.info/docs/imwa_2006/1920-Schmiermund-CO-3.pdf. Accessed May 2013.

Schmiermund, R.L., Lazo, M.C., and Parnow, C.C. 2006. Application of geo-environmental models to accelerated EIA and permitting processes for an Andean porphyry Cu-Au deposit. In *Proceedings, 7th International Conference on Acid Rock Drainage (ICARD 7)*. Lexington, KY: American Society of Mining and Reclamation. pp. 1903–1911. www.imwa.info/docs/imwa_2006/1903-Schmiermund-CO-1.pdf. Accessed May 2013.

Schreuder, H.T., Ernst, R., and Ramirez-Maldonado, H. 2004. *Statistical Techniques for Sampling and Monitoring Natural Resources*. General Technical Report-RMRS-GTR-126. Fort Collins, CO: U.S. Department of Agriculture, Rocky Mountain Research Station. www.fs.fed.us/rm/pubs/rmrs_gtr126.pdf. Accessed May 2013.

Schumacher, B.A., Shines, K.C., Burton, J.V., and Papp, M.L. 1990. *A Comparison of Soil Sample Homogenization Techniques*. EPA 600//X-90/043. Washington, DC: U.S. Environmental Protection Agency, Office of Research and Development. www.epa.gov/esd/cmb/research/papers/bs120.pdf. Accessed May 2013.

Scullion, J., and Edwards, R.W. 1980. The effect of pollutants from the coal industry on the fish fauna of a small river in the South Wales coalfield. *Environ. Pollut. (Series A)* 21:141–153.

Seal, R.R. II, and Foley, N.K., eds. 2002. *Progress on Geoenvironmental Models for Selected Mineral Deposit Types*. Open-File Report 02-195. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/of/2002/of02-195/. Accessed May 2013.

Seal, R.R. II, and Hammarstrom, J.M. 2003. Geoenvironmental models of mineral deposits: Examples from massive sulfide and gold deposits. In *Environmental Aspects of Mine Wastes*. Edited by J.L. Jambor, D.W. Blowes, and A.I.M. Ritchie. Short Course Series, Vol. 31. Ottawa, ON: Mineralogical Association of Canada.

Seal, R.R., II, Piatak, N.M., Levitan, D.M., Hageman, P.L., and Hammarstrom, J.M. 2009. Comparison of geochemical characteristics of modern-style mine wastes from a variety of mineral deposit types for insights into environmental challenges associated with future mining. In *Proceedings, Securing the Future and 8th International Conference on Acid Rock Drainage (ICARD)*. Sweden: SveMin.

Sengupta, M. 1990. *Mine Environmental Engineering, Vol. I*. Boca Raton, FL: CRC Press.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Sevee, J.E., White, C.A., and Maher, D.J. 2000. An analysis of low-flow ground water sampling methodology. *Ground Water Monit. Remed*. 20(2):87–93.

Shaw, S., and Mills, C. 2012. Petrology and mineralogy in ARD prediction. Infomine. http://technology.infomine.com/enviromine/ard/mineralogy/petrology%20and%20mineralogy.htm. Accessed May 2013.

Skousen J.G., J.C. Sencindiver, and R.M. Smith. 1987. *A Review of Procedures for Surface Mining and Reclamation in Areas with Acid-Producing Materials, in cooperation with the West Virginia Surface Mine Drainage Task Force, the West Virginia University Energy and Water Research Center, and the West Virginia Mining and Reclamation Association*. Morgantown, WV: The Center.

Sliwka-Kaszyńska, M., Kot-Wasik, A., and Namieśnik, J. 2003. Preservation and storage of water samples. *Crit. Rev. Environ. Sci. Technol*. 33:31–44.

Smith, K.S. 1999. Metal sorption on mineral surfaces: An overview with examples relating to mineral deposits. In *The Environmental Geochemistry of Mineral Deposits—Part A: Processes, Techniques, and Health Issues*. Reviews in Economic Geology, Vol. 6A. Littleton, CO: Society of Economic Geologists. pp. 161–182.

Smith, K.S. 2007. Strategies to predict metal mobility in surficial mining environments. Reviews in Engineering Geology, Vol. 17. In *Understanding and Responding to Hazardous Substances at Mine Sites in the Western United States*. Edited by J.V. DeGraff. Boulder, CO: Geological Society of America. pp. 25–45.

Smith, K.S. 2011. Approach for environmental baseline water sampling. In *Proceedings of the 2011 SME Annual Meeting and Exhibit*. Preprint 11-157. Littleton, CO: SME.

Smith, K.S., and Huyck, H.L.O. 1999. An overview of the abundance, relative mobility, bioavailability, and human toxicity of metals. In *The Environmental Geochemistry of Mineral Deposits—Part A: Processes, Techniques, and Health Issues*. Reviews in Economic Geology, Vol. 6A. Littleton, CO: Society of Economic Geologists. pp. 29–70.

Smith, K.S., Ramsey, C.A., and Hageman, P.L. 2000. Sampling strategy for the rapid screening of mine-waste dumps on abandoned mine lands. In *Proceedings, 5th International Conference on Acid Rock Drainage (ICARD 2000)*. Littleton, CO: SME. pp. 1453–1461. http://crustal.usgs.gov/projects/minewaste/pdfs/kathy.pdf. Accessed May 2013.

Smith, K.S., Campbell, D.L., Desborough, G.A., Hageman, P.L., Leinz, R.W., Stanton, M.R., Sutley, S.J., Swayze, G.A., and Yager, D.B. 2002. Toolkit for the rapid screening and characterization of waste piles on abandoned mine lands. In *Progress on Geoenvironmental Models for Selected Mineral Deposit Types*. Edited by R.R. Seal II, and N.K. Foley. Open-File Report 02-0195. Reston, VA: U.S. Geological Survey. pp. 55–64. http://pubs.usgs.gov/of/2002/of02-195/. Accessed May 2013.

Smith, K.S., Ranville, J.F., Adams, M.K., Choate, L.M., Church, S.E., Fey, D.L., Wanty, R.B., and Crock, J.G. 2006. Predicting toxic effects of copper on aquatic biota in mineralized areas by using the biotic ligand model. In *Proceedings, 7th International Conference on Acid Rock Drainage (ICARD 7)*. Lexington, KY: American Society of Mining and Reclamation. pp. 2055–2077. www.imwa.info/docs/imwa_2006/2055-Smith-CO.pdf. Accessed May 2013.

Smith, K.S., Hageman, P.L., Briggs, P.H., Sutley, S.J., McCleskey, R.B., Livo, K.E., Verplanck, P.L., Adams, M.G., and Gemery-Hill, P.A. 2007. *Questa Baseline and Pre-Mining Ground-Water Quality Investigation: 19. Leaching Characteristics of Composited Materials from Mine Waste-Rock Piles and Naturally Altered Areas near Questa, New Mexico*. Scientific Investigations Report 2006-5165. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/sir/2006/5165/. Accessed May 2013.

Smith, K.S., Ranville, J.F., Diedrich, D.J., McKnight, D.M., and Sofield, R.M. 2009. Consideration of iron-organic matter interactions when predicting aquatic toxicity of copper in mineralized areas. In *Proceedings, Securing the Future and 8th International Conference on Acid Rock Drainage (ICARD)*. Sweden: SveMin. www.proceedings-stfandicard-2009.com/pdfer/Kathleen_Smith_B5_T6_Consideration-of-Iron-Organic-Matter-Interactions-when-Predicting-Aquatic-Toxicity-of-Copper-in-Mineralized-Areas.pdf. Accessed May 2013.

Smith, K.S., Hoal, K.O., Walton-Day, K., Stammer, J.G., and Pietersen, K. 2013. Automated quantitative micro-mineralogical characterization for environmental applications. In *Proceedings, 2013 SME Annual Meeting and Exhibit*. Paper 13-140. Englewood, CO: SME.

Sobek, A.A., Schuller, W.A., Freeman, J.R., and Smith, R.M. 1978. *Field and Laboratory Methods Applicable to Overburden and Minesoils*. U.S. Environmental Protection Agency, Office of Research and Development. EPA-600/2-78-054.

Sparks, D.L. 2003. *Environmental Soil Chemistry*, 2nd ed. Boston: Academic Press.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Sparks, D.L. 2005. Toxic metals in the environment: The role of surfaces. *Elements* 1:193–197.

Stoddard, J.L., Larsen, D.P., Hawkins, C.P., Johnson, R.K., and Norris, R.H. 2006. Setting expectations for the ecological condition of streams: The concept of reference condition. *Ecol. Appl*. 16:1267–1276.

Stribling, J.B., Jessup, B.K., and Feldman, D.L. 2008. Precision of benthic macroinvertebrate indicators of stream condition in Montana. *J. N. Am. Benthol. Soc*. 27(1):58–67.

Sullivan, A.B., and Drever, J.I. 2001. Spatiotemporal variability in stream chemistry in a high-elevation catchment affected by mine drainage. *J. Hydrol*. 252:240–253.

Swayze, G.A., Smith, K.S., Clark, R.N., Sutley, S.J., Pearson, R.M., Vance, J.S., Hageman, P.L., Briggs, P.H., Meier, A.L., Singleton, M.J., and Roth, S. 2000. Using imaging spectroscopy to map acidic mine waste. *Environ. Sci. Technol*. 34:47–54.

Taylor, J.K. 1987. *Quality Assurance of Chemical Measurements*. Chelsea, MI: Lewis Publishers, CRC Press.

Tessier, A., Campbell, P.G.C., and Bisson, M. 1979. Sequential extraction procedure for the speciation of particulate trace metals. *Anal. Chem*. 51:844–851.

Thompson, M. 1989. Analytical quality control in theory and practice. In *Proceedings, 3rd International Symposium on the Harmonization of Quality Assurance Systems in Analytical Chemistry*. Washington, DC: ISO/REMCO 184. pp. 183–189.

Todd, A.S., McKnight, D.M., Jaros, C.L., and Marchitto, T.M. 2007. Effects of acid rock drainage on stocked rainbow trout (O. mykiss): An in-situ, caged fish experiment. *Environ. Monit. Assess*. 130:111–127.

Toth, J. 1963. A theoretical analysis of groundwater flow in small drainage basins. *J.Geophys. Res*. 68(16):4795–4812.

Tremblay, G.A., and Hogan, C.M., eds. 2001a. *MEND Manual, Vol. 2—Sampling and Analysis*. MEND document 5.4.2b. Ottawa, ON: Natural Resources Canada, Mine Environment Neutral Drainage Program.

Tremblay, G.A., and Hogan, C.M., eds. 2001b. *MEND Manual, Vol. 6—Monitoring*. MEND document 5.4.2f. Ottawa, ON: Natural Resources Canada, Mine Environment Neutral Drainage Program.

Triad Resource Center. n.d. Categorizing analytical technologies. www.triadcentral.org/mgmt/meas/key/tech/index.cfm. Accessed 10/4/13.

Truckenbrodt, D., and Einax, J. 1995. Sampling representativity and homogeneity of river sediments. *Fresenius J. Anal. Chem*. 352:437–443.

Turnipseed, D.P., and Sauer, V.B. 2010. *Discharge Measurements at Gaging Stations*. Techniques and Methods, book 3. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/tm/tm3-a8/. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 1983. *Methods for Chemical Analysis of Water and Wastes*: U.S. Environmental Protection Agency, EPA 600479020. http://yosemite.epa.gov/water/owrccatalog.nsf/1ffc8769fdecb48085256ad3006f39fa/f995d73aff86600085256b0600724015!OpenDocument. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 1984. *Characterization of Hazardous Waste Sites–A Methods Manual: Vol. II, Available Sampling Methods*, 2nd ed.: U.S. Environmental Protection Agency, EPA-600/4-84-076.

USEPA (U.S. Environmental Protection Agency). 1991. *Handbook: Ground Water Volume II: Methodology*: U.S. Environmental Protection Agency, EPA/625/6-90/016b.

USEPA (U.S. Environmental Protection Agency). 1995. *Superfund Program Representative Sampling Guidance–Volume 1: Soil*: U.S. Environmental Protection Agency, EPA 540/R-95/141 PB96-963207. www.clu-in.org/download/char/SF_Rep_Samp_Guid_soil.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 1997. Memorandum (November 5, 1997): *Establishing Site Specific Aquatic Life Criteria to Natural Background*. U.S. Environmental Protection Agency, Tudor T. Davies, Director, Office of Science and Technology. http://water.epa.gov/scitech/swguidance/standards/upload/2009_01_29_criteria_naturalback.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 1999. *Correct Sampling Using the Theories of Pierre Gy*. Technology Support Center Fact Sheet 197CMB98.FS-14 (Rev. 4/26/99): U.S. Environmental Protection Agency, Environmental Sciences Division. www.epa.gov/esd/factsheets/csutpg.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2000. *Abandoned Mine Site Characterization and Cleanup Handbook*: U.S. Environmental Protection Agency, EPA 910-B-00-001. www.epa.gov/superfund/policy/remedy/pdfs/amscch. Accessed December 2013.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

USEPA (U.S. Environmental Protection Agency). 2001a. *Data Quality Objectives Decision Error Feasibility Trials Software (DEFT)–User's Guide* (EPA QA/G-4D): U.S. Environmental Protection Agency, EPA/240/B-01/007. www.epa.gov/quality/qs-docs/g4d-final.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2001b. *EPA Requirements for Quality Assurance Project Plans* (EPA QA/R-5): U.S. Environmental Protection Agency, EPA/240/B-01/003. www.epa.gov/quality/qs-docs/r5-final.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2001c. *Performing Quality Flow Measurements at Mine Sites*: U.S. Environmental Protection Agency, Office of Research and Development, EPA/600/R-01/043. http://nepis.epa.gov/Adobe/PDF/30002H0Y.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2001d. *Abandoned Mine Site Characterization and Cleanup Handbook*: U.S. Environmental Protection Agency, EPA 530-C-01-001.

USEPA (U.S. Environmental Protection Agency). 2002a. *Guidance on Choosing a Sampling Design for Environmental Data Collection for Use in Developing a Quality Assurance Project Plan* (EPA QA/G-5S): U.S. Environmental Protection Agency, EPA/240/R-02/005. www.epa.gov/quality/qs-docs/g5s-final.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2002b. *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms*, Fifth Edition: U.S. Environmental Protection Agency, Office of Water, EPA-821-R-02-012. http://water.epa.gov/scitech/methods/cwa/wet/upload/2007_07_10_methods_wet_disk2_atx.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2003a. *Diesel Exhaust in the United States*: U.S. Environmental Protection Agency, EPA420-F-03-022. www.epa.gov/cleandiesel/documents/420f03022.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2003b. *EPA and Hardrock Mining: A Source Book for Industry in the Northwest and Alaska*: U.S. Environmental Protection Agency, Region 10. http://yosemite.epa.gov/R10/WATER.NSF/840a5de5d0a8d1418825650f00715a27/e4ba15715e97ef2188256d2c00783a8e/$FILE/Maintext.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2005. *Procedures for the Derivation of Equilibrium Partitioning Sediment Benchmarks (ESBs) for the Protection of Benthic Organisms: Metal Mixtures (Cadmium, Copper, Lead, Nickel, Silver and Zinc)*: U.S. Environmental Protection Agency, EPA-600-R-02-011. www.epa.gov/nheerl/download_files/publications/metalsESB_022405.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2006a. *Data Quality Assessment: Statistical Methods for Practitioners* (EPA QA/G-9S): U.S. Environmental Protection Agency, EPA/240/B-06/003. www.epa.gov/quality/qs-docs/g9s-final.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2006b. *Guidance on Systematic Planning Using the Data Quality Objectives Process* (EPA QA/G-4): U.S. Environmental Protection Agency, EPA/240/B-06/001. www.epa.gov/quality/qs-docs/g4-final.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2006c. *Wadeable Streams Assessment: A Collaborative Survey of the Nation's Streams*: U.S. Environmental Protection Agency, EPA 841-B-06-002. http://water.epa.gov/type/rsl/monitoring/streamsurvey/upload/2007_5_16_streamsurvey_WSA_Assessment_May2007-2.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2007a. *Aquatic Life Ambient Freshwater Quality Criteria–Copper (2007 Revision)*: U.S. Environmental Protection Agency, EPA-822-R-07-001. http://water.epa.gov/scitech/swguidance/standards/criteria/aqlife/pollutants/copper/upload/2009_04_27_criteria_copper_2007_criteria-full.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2007b. *Framework for Metals Risk Assessment*: U.S. Environmental Protection Agency, EPA 120/R-07/001. www.epa.gov/raf/metalsframework/pdfs/metals-risk-assessment-final.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2007c. *Guidance for Preparing Standard Operating Procedures (SOPs)* (EPA QA/G-6): U.S. Environmental Protection Agency, EPA/600/B-07/001. www.epa.gov/quality/qs-docs/g6-final.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2007d. *Remediation System Evaluation (RSE)–Central City/Clear Creek Superfund Site, Argo Tunnel Water Treatment Plant, Idaho Springs, Colorado*: U.S. Environmental Protection Agency, EPA 542-R-07-019. www.epa.gov/tio/download/remed/rse/final_argo_wtp_rse.pdf. Accessed May 2013.

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USEPA (U.S. Environmental Protection Agency). 2007e. Table 3-2, revision 4. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846)*: U.S. Environmental Protection Agency. www.epa.gov/wastes/hazard/testmethods/sw846/. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2008. *Final Remedial Investigation Report for the Gilt Edge Superfund Site, Lawrence County, South Dakota*: U.S. Environmental Protection Agency, U.S. EPA Contract No. 68-W-05-049. www.epa.gov/region8/superfund/sd/giltedge/RI_ReportGiltEdgeVolumeI_Text.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2012. Quality assurance glossary. www.epa.gov/nrmrl/qa/glossary.html. Accessed 10/4/13.

USEPA (U.S. Environmental Protection Agency). Variously dated. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846)*: U.S. Environmental Protection Agency. www.epa.gov/wastes/hazard/testmethods/sw846/. Accessed May 2013.

USGS (U.S. Geological Survey). 2011. Chemical modeling of acid waters: Questa baseline and pre-mining ground-water quality investigation, Red River Valley Basin, New Mexico. Home page. wwwbrr.cr.usgs.gov/projects/GWC_chemtherm/questa.htm. Accessed 9/30/13.

USGS (U.S. Geological Survey). 2013a. Laser ablation ICP-MS laboratory. http://crustal.usgs.gov/laboratories/icpms/laser_ablation.html. Accessed 12/6/13..

USGS (U.S. Geological Survey). 2013b. Upper Animas River study area reports, abstracts, and papers. http://amli.usgs.gov/reports/animas_rpts.html, Accessed 9/30/13..

USGS (U.S. Geological Survey). Variously dated. *National Field Manual for the Collection of Water-Quality Data*. Techniques of Water-Resources Investigations, book 9, chaps. A1-A9. Reston, VA: U.S. Geological Survey. http://water.usgs.gov/owq/FieldManual/. Accessed May 2013.

van der Plas, L., and Tobi, A.C. 1965. A chart for judging the reliability of point counting results. *Am. J. Sci*. 263:87–90.

Vaughan, D.J., and Wogelius, R.A., eds. 2000. *Environmental Mineralogy*. Notes in Mineralogy, Vol. 2. European Mineralogical Union. Budapest: Eötvös University Press.

Verplanck, P.L., Nordstrom, D.K., Bove, D.J., Plumlee, G.S., and Runkel, R.L. 2009. Naturally acidic surface and ground waters draining porphyry-related mineralized areas of the Southern Rocky Mountains, Colorado and New Mexico. *Appl. Geochem*. 24(2):255–267.

Voss, C., and Letient, H. 2006. Peruvian mine operation using dynamic system modeling. *Southwest Hydrol*. July/August 22–23, pp. 31.

Vrana, B., Mills, G.A., Allan, I.J., Dominiak, E., Svensson, K., Knutsson, J., Morrison, G., and Greenwood, R. 2005. Passive sampling techniques for monitoring pollutants in water. *Trends Anal. Chem*. 24:845–868.

Wagner, R.E. 1998. *Guide to Environmental Analytical Methods*. New York: Genium Publishing.

Wait, D., and Ramsey, C. 2007. The measurement process. In *Introduction to Environmental Forensics*, 2nd ed. Edited by B.L. Murphy and R.D. Morrison. Amsterdam; Boston: Elsevier Academic Press. pp. 83–128.

Walker, S.W., Jamieson, H.E., Lanzirotti, A., and Andrade, C.F. 2005. Determining arsenic speciation in iron oxides derived from a gold-roasting operation: Application of synchrotron micro-XRD and micro-XANES at the grain scale. *Can. Mineral*. 43:1205–1224.

Walsh, M.E., Ramsey, C.A., and Jenkins, T.F. 2002. The effect of particle size reduction by grinding on subsampling variance for explosives residues in soil. *Chemosphere* 49:1267–1273.

Walsh, M.R. 2009. *User's Manual for the CRREL Multi-Increment Sampling Tool. ERDC/CRRELL SR-09-1*. Hanover, NH: U.S. Army Engineer Research and Development Center (ERDC), Cold Regional Research and Engineering Laboratory (CRREL). www.itrcweb.org/ism-1/references/umcrrel.pdf. Accessed May 2013.

Walton-Day, K., and Poeter, E. 2009. Investigating hydraulic connections and the origin of water in a mine tunnel using stable isotopes and hydrographs. *Appl. Geochem*. 24(12):2266–2282.

Walton-Day, K., Runkel, R.L., and Kimball, B.A. 2012. Using spatially detailed water-quality data and solute-transport modeling to support total maximum daily load development. *J Am Water Resourc. Assn*. 48(5):949–969.

Wanekaya, A.K., Chen, W., and Mulchandani, A. 2008. Recent biosensing developments in environmental security. *J. Environ. Monit*. 10:703–712.

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Wanty, R.B., Berger, B.R., and Plumlee, G.S. 1999. Environmental models of mineral deposits—A state of the art. *Geologica Hungarica Series Geologica*. 24:97–106. http://crustal.usgs.gov/projects/gem/pdfs/wanty2.pdf. Accessed May 2013.

Wanty, R.B., Goldhaber, M.B., Morrison, J.M., and Lee, L. 2009a. Regional variations in water quality and relationships to soil and bedrock weathering in the southern Sacramento Valley, California, USA. *Appl. Geochem*. 24:1512–1523.

Wanty, R.B., Verplanck, P.L., San Juan, C.A., Church, S.E., Schmidt, T.S., Fey, D.L., DeWitt, E.H., and Klein, T.L. 2009b. Geochemistry of surface water in alpine catchments in central Colorado, USA: Resolving host-rock effects at different spatial scales. *Appl. Geochem*. 24:600–610.

Ward, C.R., Taylor, J.C., and Cohen, D.R. 1999. Quantitative mineralogy of sandstones by X-ray diffractometry and normative analysis. *J. Sed. Res*. 69(5):1050–1062.

White, J.C., ed. 1985. *Applications of Electron Microscopy in the Earth Sciences*. Short Course Vol. 11. Ottawa, ON: Mineralogical Association of Canada.

Wildeman, T.R., Smith, K.S., and Ranville, J.F. 2007. A simple scheme to determine potential aquatic metal toxicity from mining wastes. *Environ. Forensics J*. 8:119–128.

Wilkin, R.T., McNeil, M.S., Adair, C.J., and Wilson, J.T. 2001. Field measurement of dissolved oxygen: A comparison of methods. *Ground Water Monit. Remed*. 21(4):124–132.

Williams, R.D., and Diehl, S.F., eds. 2014. *Techniques for Predicting Metal Mining Influenced Water*. Management Technologies for Metal Mining Influenced Water series, Vol. 5. Englewood, CO: SME.

Winter, T.C. 1999. Relation of streams, lakes, and wetlands to groundwater flow systems. *Hydrogeol. J*. 7:28–45.

Winter, T.C., Harvey, J.W., Franke, O.L., and Alley, W.M. 1998. *Ground Water and Surface Water—A Single Resource*. Circular 1139. Reston, VA: U.S. Geological Survey. http://water.usgs.gov/pubs/circ/circ1139. Accessed May 2013.

Winterbourn, M.J., McDiffett, W.R, and Eppley, S.J. 2000. Aluminium and iron burdens of aquatic biota in New Zealand streams contaminated by acid mine drainage: Effects of trophic level. *Sci. Total Environ*. 254:45–54.

Wolf, R.E. 2005. What is ICP-MS? ...and more importantly, what can it do? U.S. Geological Survey. http://crustal.usgs.gov/laboratories/icpms/intro.html. Accessed 12/6/13.

Wolkersdorfer, C. 2002. Mine water tracing. In *Mine Water Hydrogeology and Geochemistry*. Edited by P.L. Younger and N.S. Robins. Special Pub. 198. London: The Geological Society. pp. 47–60.

Yager, D.B., Johnson, R.H., Rockwell, B.W., Caine, J.S., and Smith, K.S. 2013. A GIS and statistical approach to identify variables that control water quality in hydrothermally altered and mineralized watersheds, Silverton, Colorado, USA. *Environ. Earth Sci*. 70(3):1057–1082. http://link.springer.com/article/10.1007/s12665-013-2229-y. Accessed May 2013. DOI:10.1007/s12665-013-2229-y

Yeskis, D., and Zavala, B. 2002. *Ground-Water Sampling Guidelines for Superfund and RCRA Project Managers*. EPA/542/S-02/001. Ground Water Forum Issue Paper. Washington, DC: U.S. Environmental Protection Agency. www.epa.gov/superfund/remedytech/tsp/download/gw_sampling_guide.pdf. Accessed May 2013.

Young, R.A., ed. 1993. *The Rietveld Method*. International Union of Crystallography Monographs on Crystallography, Vol. 5. Oxford: Oxford University Press.

Younger, P.L. 2007. *Groundwater in the Environment: An Introduction*. Oxford: Blackwell Publishing.

Zhang, C. 2007. *Fundamentals of Environmental Sampling and Analysis*. New York: John Wiley and Sons.

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CHAPTER 4

Decision Making, Risk, and Uncertainty

Carol C. Russell, Kathleen S. Smith, and Virginia T. McLemore

4.1 INTRODUCTION

This handbook places special emphasis on answering of questions by gathering reliable scientific data and analyzing the data to provide information upon which to base decisions. Risks to human health and the environment are the underpinning for each of the identified international guidance documents for sampling and monitoring. Identifying risks to valuable resources and reducing uncertainty and doubt about alternatives allow a decision maker reasonable choices among alternatives.

This chapter introduces the principles of risk assessment and risk analysis, and their usefulness to the management of risk. Different types of uncertainty are described, including their importance to decisions that might be influenced by, or concerns with, management of the environment.

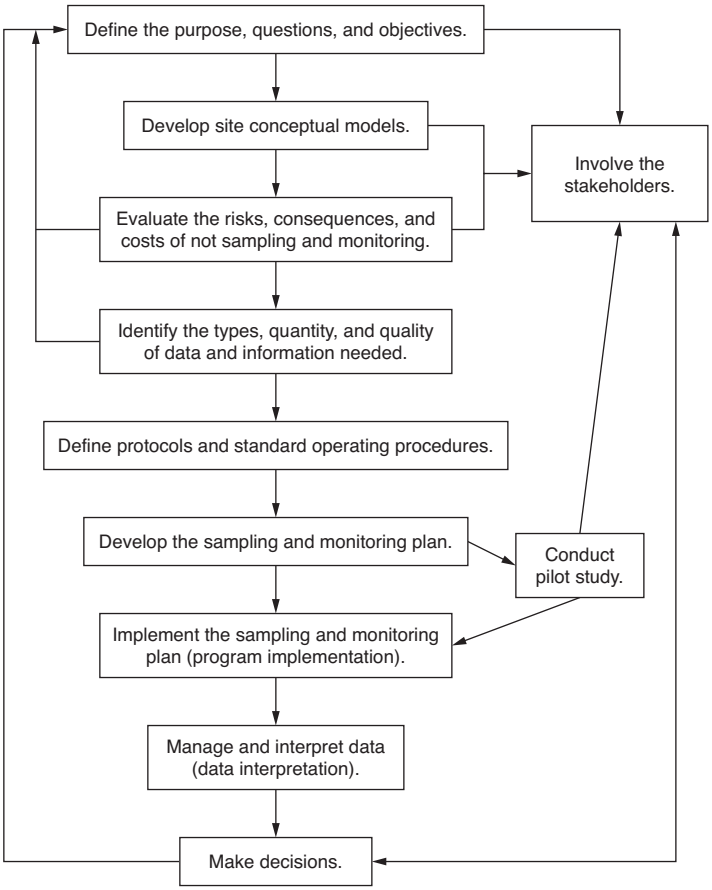
The Acid Drainage Technology Initiative–Metal Mining Sector (ADTI-MMS) recommends the step-by-step framework shown in Figure 4.1 for the development and implementation of a sampling and monitoring program.

A review of international guidance documents in English on sampling and monitoring programs for metals at mining and processing sites reveals the importance of identifying risks and their relevance for decision making. These guidance documents also provide recommended processes for quality assurance and quality control (QA/QC) to reduce uncertainty. Systematic steps in guidance documents are similar across jurisdictions and scopes of influence.

Recent emphasis on sustainability influences how the mining industry approaches environmental assessment, planning, and reporting (Fairbrother et al. 2007). For example, the International Council on Mining and Metals (ICMM) sustainable development framework (ICMM 2013) outlines approaches for biodiversity risk assessment and mitigation during the various phases of mining, and provides guidance for best management practices. Another example is the Global Reporting Initiative (GRI) reporting framework, which is intended to provide a generally accepted framework for reporting on an organization's economic, environmental, and social performance. The G3 is the third generation of the GRI reporting guidelines, and the GRI has a G3 *Mining and Metals Sector Supplement* (GRI 2010) that is designed for the mining and metals sector. This kind of sustainability reporting is a form of value reporting whereby an organization communicates their environmental, social, and economic performance, and can influence the need for the collection of specific types of data that have not traditionally been collected (ICMM 2008).

Generally, mining and processing operations throughout the world are expected to have individual and specific sampling and monitoring programs designed to address a variety of questions and issues arising during the mine life cycle. For example, monitoring of baseline water quality

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Source: Adapted from McLemore et al. 2004.

FIGURE 4.1 Steps for implementing a sampling and monitoring program

prior to initiating extraction may form the basis of future reclamation expectations and potential allocation of liabilities. Modeling of groundwater flow based on hydrogeological monitoring during the development phase could be used to predict impacts to groundwater resources at closure. Similarly, chemical analyses of exploration core and cuttings could be utilized to decide if special handling of waste rock will be necessary to ensure adequate reclamation.

4.2 RISK

The objective of sampling and monitoring is to provide adequate data to test or quantify a hypothesis to address potential environmental risks in a cost-effective manner and to minimize potential liabilities. "Mining is a high risk venture in view of the multitude of unpredictable factors in the finding, acquisition, extraction, treatment and sale of the product. Risks may be geological, technological, economic, political and/or social" (Lacy 2010). Throughout the mine life cycle, from the decision to invest in a new mine, to the safety and health issues of working or living near a mine, to the environmental issues during production and ultimate closure and the potential future land uses, there are risks. In this chapter, an attempt is made to bridge the gap between different concepts of risk as used by professionals operating in the mining, environmental, and economic realms.

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Severity of Effect	Probability of Effect: A Almost Certain	B Likely	C Possible	D Unlikely	E Rare
1 Catastrophic	Extreme	Extreme	Extreme	Extreme	High
2 Major	Extreme	Extreme	Extreme	High	High
3 Moderate	Extreme	High	High	Moderate	Moderate
4 Minor	High	High	Moderate	Low	Low
5 Insignificant	High	Moderate	Low	Low	Low

Source: Adapted from Australian Government 2008.

FIGURE 4.2 Classification of risks associated with mining operations in South Australia (the darker the shading, the higher the risk)

GEORGIUS AGRICOLA

Georgius Agricola says this about the risk of mining:

> *The critics say further that mining is a perilous occupation to pursue because the miners are sometimes killed by the pestilential air which they breathe; sometimes their lungs rot away; sometimes the men perish by being crushed in masses of rock; sometimes falling from ladders into the shafts, they break their arms, legs, or necks; and it is added, there is no compensation which should be thought great enough to equalize the extreme dangers to safety and life.*
>
> —*De Re Metallica*

The term *risk* means different things to different people. Risk is usually seen as an uncertain event that could adversely affect the achievement of objectives. Risk is a function of the consequences of an event, such as loss of life or economic damages, combined with the likelihood of its occurrence (Figure 4.2). To take Agricola's example (see sidebar) of falling off ladders, an objective risk assessment uses numbers, after the fact, to calculate the risk. In contrast, by proactively estimating potential risks in the future, alternatives can be evaluated based on projected environmental impacts and mitigation costs to design a mine plan and to set an adequate environmental bond.

Metals and metal compounds have unique characteristics that should be considered when assessing their risks. For example, metals are neither created nor destroyed by biological or chemical processes, but they can be transformed from one chemical form to another. Metals occur naturally, and some metals are essential for life in low doses but are toxic at high levels. The *Framework for Metals Risk Assessment* (USEPA 2007, 2011) is an excellent place to start to assess metals in the environment. In environmental science, the study of adverse impacts originating from a source is commonly considered to be the study of toxicology or the science of poisons. These adverse effects may occur in many forms, ranging from immediate death (acute toxicity) to subtle changes not realized until months or years later (chronic toxicity).

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4.2.1 Risk Assessment

Risk assessment is typically defined as a measure of the probability of an event happening and severity of adverse effects. Risk assessment answers the following questions (Kaplan and Garrick 1981; Suter 1993):

- What can go wrong?
- What is the likelihood that it would go wrong?
- What are the consequences?

Answers to these questions help risk analysts to identify, measure, quantify, and evaluate risks and their consequences and impacts. "A successful risk assessment leading to a reduction or elimination of risk is only as good as the knowledge, skill, and effort of those undertaking the assessment" (Caldwell 2010). One also must take into account public perceptions of risk and attempt to manage potential public misunderstandings. Risks considered most serious by the general public, in many cases, are different from those considered most serious by the technical professionals charged with reducing environmental risk. Consideration of public perceptions about selected chemicals associated with the mining and processing industry, such as cyanide leaching during processing, should be considered as a monitoring program is designed.

A human health risk assessment (HRA) consists of the evaluation of scientific information on the hazardous properties of environmental agents (hazard characterization), the dose-response relationship (dose-response assessment), and the extent of human exposure to those agents (exposure assessment). In final form, a risk assessment is a statement regarding the probability that populations or individuals so exposed will be harmed and to what degree (risk characterization). Especially vulnerable populations, such as children, are often taken into account. The two major databases used for comparison are the Integrated Risk Information System (IRIS) in the United States and Registration, Evaluation, and Authorization of Chemicals (REACH) by the European Chemicals Agency. The IRIS database contains information for more than 540 chemical substances on human health effects that may result from exposure to various substances in the environment, whereas the REACH system is more of a voluntary registration of 8,469 unique, manufactured substances. Both rely on the concept of a dose-response curve with a *safe region* of no effects transitioning to an increasing *not safe* zone. Noncancer risks are assumed to have a threshold, but cancer risks have no threshold.

Metal-specific tools such as the *Framework for Metals Risk Assessment* (USEPA 2007, 2011) in the United States and the *Health Risk Assessment Guidance for Metals* (HERAG; ICMM 2006) and *Metals Environmental Risk Assessment Guidance* (MERAG; ICMM 2007) each provide internationally relevant scientific references for the application of risk assessment to metals with regard to human health and the environment. Risk assessments conducted on specific metals in the European Union and North America and these national guidance documents acknowledge that metals have specific properties that require consideration in risk assessment. The main HERAG and MERAG documents provide an overview of the particular issues associated with the evaluation of metals. A series of associated fact sheets address selected issues in more detail (ECHA 2008). ICMM has provided a comparison of the generic risk management process and the generic environmental assessment framework (ICMM 2007). Table 4.1 provides a comparison of the risk assessment process from the Management Advisory Board Australia and the generic environmental assessment framework for U.S. Environmental Protection Agency's (USEPA's) ecological risk assessments.

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TABLE 4.1 Comparison of the generic risk assessment process

Step	Management Advisory Board Australia Process	Generic Framework	USEPA Framework for Ecological Risk Assessment
1	Establish the context	Define policy, purpose, objectives, success criteria	Problem formulation
2	Identify the risks	Concerns, consequences	Problem formulation
3	Analyze the risks	Calculations (identify concerns and possible outcomes); certainty and uncertainty	Analysis phase
4	Assess and prioritize risks	Compare with criteria; prioritize	Risk characterization
5	Treat the risks	Control, communication; develop and implement contingency/ management plans	Communication
6	Review and monitor	Continue reviewing and monitoring; assess effectiveness of treatment	Review and monitor; assess effectiveness of treatment

Source: Compiled by ICMM (2008), as adapted from Beer and Ziolkowski (1995).

TABLE 4.2 Comparison of terminology used in sampling and monitoring with that used in risk assessment and risk management

Sampling and Monitoring	Risk Assessment	Risk Management
Source—Geoavailability	Hazard identification	Alternative development (options for releases)
	Exposure assessment	Evaluation of alternatives
Pathway	Dose-response	Mitigation
		Recommendations
Receptor	Toxicity assessment	Action (implementation)

4.2.2 Risk Management

Risk management is commonly distinguished from risk assessment in that it focuses on what can be done regarding what has or what may happen (see Table 4.2 and Chapter 5). Risk management builds on the risk assessment evaluating the following:

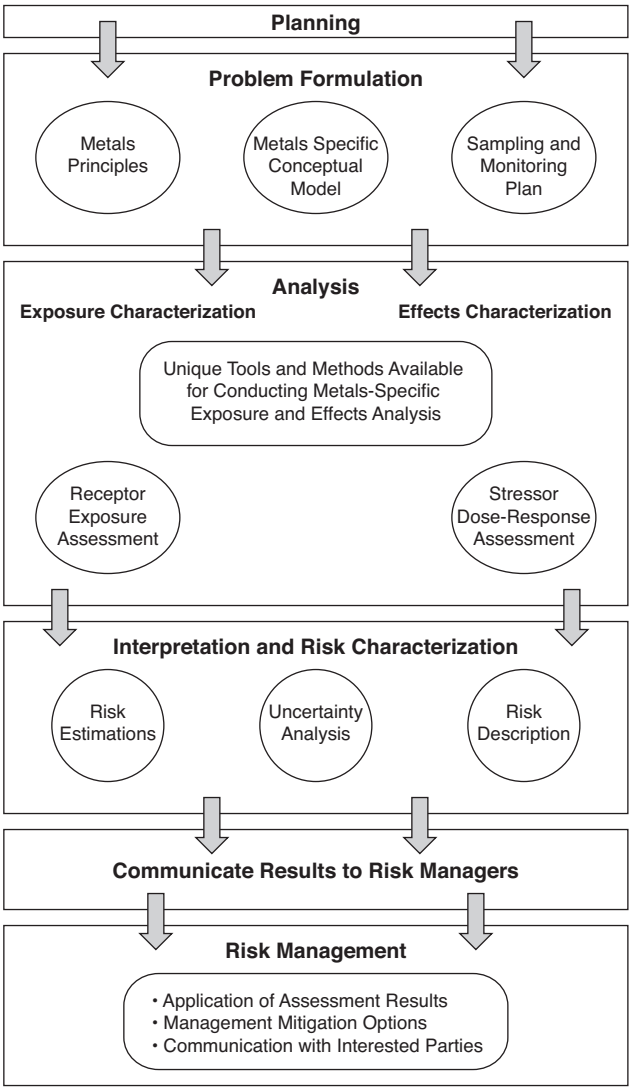
- What can be done and what options are available?
- What are the associated trade-offs in terms of all costs, benefits, and risks?
- What are the impacts of current management decisions on future options?

Risk management is the assessment and prioritization of risks (defined in ISO 31000 as the effect of uncertainty on objectives, whether positive or negative), followed by coordinated and economical application of resources to minimize, monitor, and control the probability and/or impact of unfortunate events or to maximize the realization of opportunities. Strategies to manage risk include transferring the risk to another party, avoiding the risk, reducing the negative effect of the risk, and accepting some or all of the consequences of a particular risk (Failing et al. 2007). Figure 4.3 illustrates the process for risk assessment and management for metals (which is discussed in more detail in USEPA 2007, 2011).

4.2.3 Environmental Monitoring—What Is at Risk?

At risk are human health and the environment as reflected by the quality of air and water, ecosystem health, and other ecosystem services and values such as endangered species, and visual, material, and historical assets. Risk assessment and risk management should be tied to the mine life cycle and take a comprehensive, integrated look at mining and mineral processing procedures,

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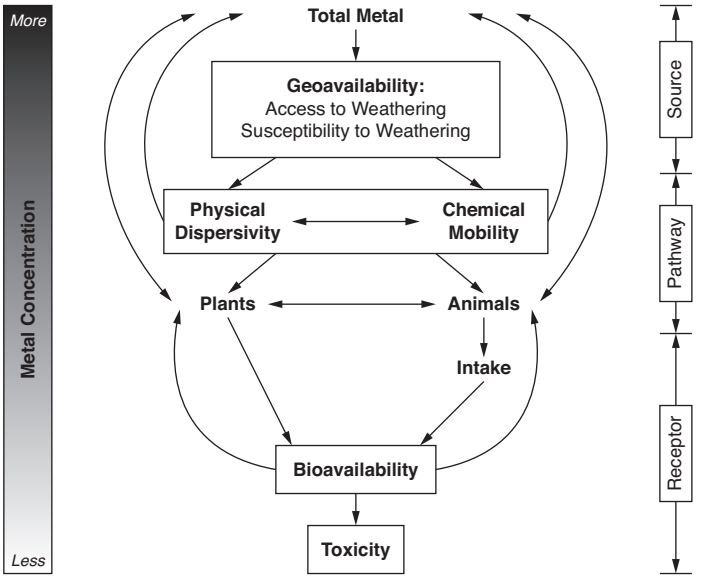


Source: USEPA 2011.

FIGURE 4.3 Risk assessment/risk management process for metals

materials produced, by-products, and final products. By integrating mining and mineral processing with an environmental risk assessment, proactive risk management decisions can be made to reduce environmental liabilities and to protect the environment. Some mine sites are large complexes that can have multiple sources of potential concern besides the mining and processed materials, and can include chemicals used in mining and processing, septic wastes, explosives, electrical transformers and power systems, fuel, lubricants, cleaners, and laboratory wastes. The HRA process includes estimating the quantity of contaminants from a site reaching human populations and determining how toxic these contaminants are to humans. In a similar fashion, the environmental risk assessment proceeds from the source via a pathway and results in a toxicity to the receptors. Risk management is the targeting of protective efforts or mitigation to the problems

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Source: Adapted from Smith and Huyck 1999; Smith 2007.

FIGURE 4.4 Source-pathway-receptor risk diagram

that seem to be the most serious or that appear to provide the most protection or mitigation for the effort expended.

A metal is only toxic if it is in a form and concentration that is bioavailable at the time of exposure (Figure 4.4; Smith and Huyck 1999; Smith 2007). Risk assessors use a variety of methods to determine metal bioavailability in aquatic systems. These methods include adjusting for water hardness, water-effect ratio (see Appendix 3), and biotic ligand models (BLMs; USEPA 2007, 2011; see Appendix 3). BLMs combine the influences of metal speciation and cationic competition on aquatic metal toxicity. Bioconcentration factors (BCFs) and bioaccumulation factors (BAFs) are commonly used to quantify metal accumulation in tissue relative to the water concentration. In the case of metals at mining and processing sites, care must be used in applying BCFs and BAFs due to the complexities involved in determining relationships between metal speciation and bioavailability, and in direct versus indirect metal toxicity (Chapman et al. 1996).

4.2.4 Risks and Consequences

Consequences of poor site characterization can be serious. In particular, water availability and water quality issues have the potential to significantly impact a company's financial performance. In general, water-related risks can be broadly classified into the categories shown in Table 4.3.

Mining companies have long been conscious of water-related risks, as evidenced by their ongoing efforts to address them and related corporate reporting (Barton 2010). Indeed, recent analysis has shown that the mining sector is a leader in terms of water quality reporting for the GRI (2010). However, corporate disclosure often does not provide a comprehensive picture of water-related risk. Current reporting frameworks do not guide companies to disclose the full scope of potential water-related risks. *Mine the Gap: Connecting Water Risks and Disclosure in the Mining Sector* by Miranda and Sauer (2010) of the World Resources Institute outlines potential water-related risks facing the mining industry and highlights important gaps in water-related disclosure. The purpose is to provide information, questions, and tools to help the financial

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TABLE 4.3 Water availability and quality risks for mining

Risks	Company's Financial Perspective
Physical	
Losses due to production	Lost revenues
Losses due to lower mineral recovery	Lower margins, lost revenues
Losses due to degraded mineral quality	Lower margins, lost revenues
Losses due to conflicts with communities	Disruption, lost revenues
Regulatory and Legal	
Losses due to operating costs	Lower margins, lost revenues
Capital expenditures	Reduced cash flow, lost revenues
Losses due to production	Lost revenues
Mine shutdown	Lost revenues, value destruction
Closure liabilities	Valuation
Reputational	
Operation disruptions	Lost revenues
Lost access to future reserves	Value destruction, lost revenues
Access to capital	Cost of capital

Source: Adapted from Miranda and Sauer 2010.

community better evaluate water-related risks facing mining companies (Miranda and Sauer 2010), specifically:

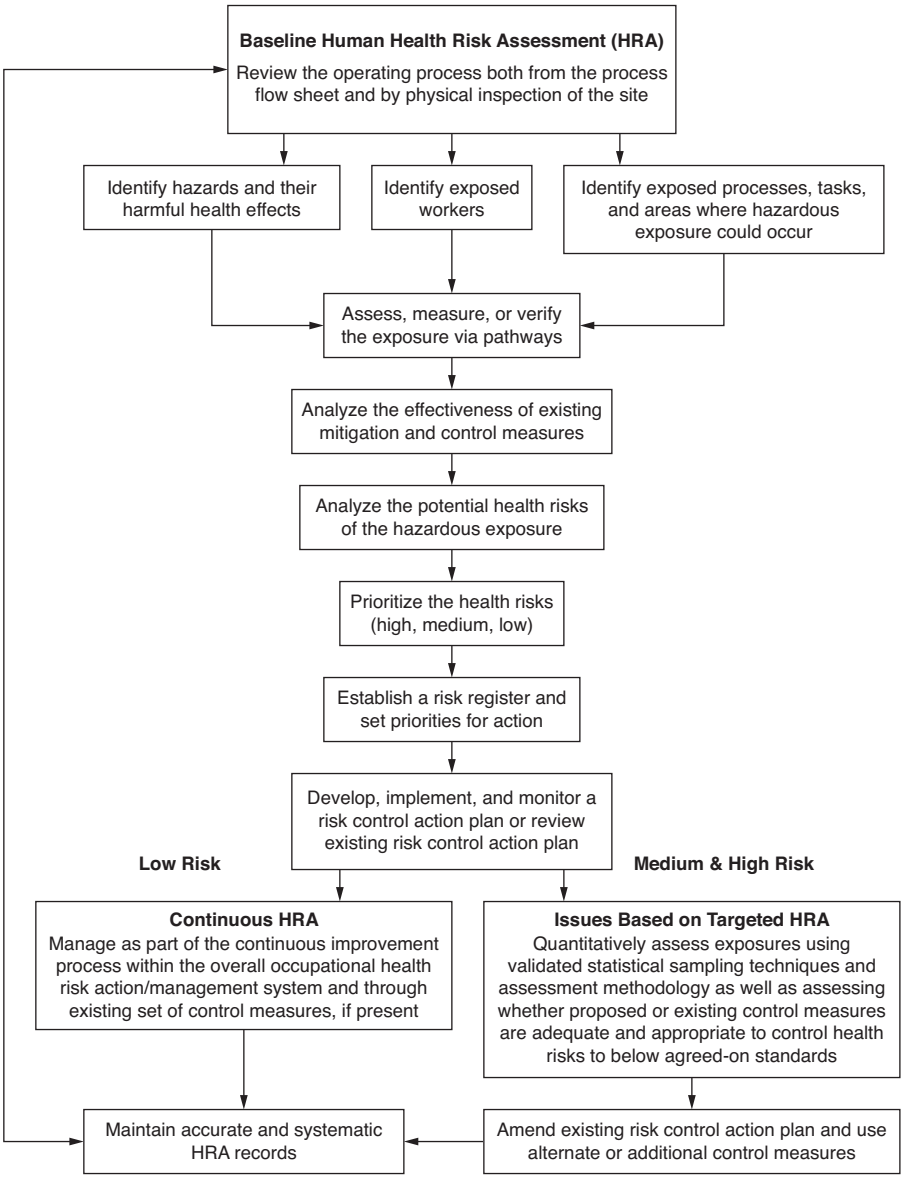
- Water-related risks span the minerals production cycle and occur in diverse operating environments.
- Water quality data are not sufficiently reported. Water quality problems are among the most serious environmental impacts associated with mining. Data on water effluents and waste management practices are either not reported or not detailed enough to understand risk. Toxic waste and mine effluents can be mobilized by water, resulting in regulatory, legal, and reputational risks for companies (see Kuipers et al. 2006 for examples).
- Work stoppages at mine or processing facilities can occur if water resources become unavailable. Mining and processing—particularly for precious metals, diamonds, copper, and nickel—requires significant volumes of water (Miranda and Sauer 2010).

4.2.5 Costs Within a Mining Risk Analysis Framework

Evaluation of the costs and potential consequences of not sampling and monitoring can be considered a type of cost/benefit evaluation or mining operation risk analysis (Figure 4.5). Throughout the mine life cycle, balancing the costs and benefits of monitoring is advised. For example, monitoring of background and baseline conditions during the exploration phase can provide the first important assessment of whether the mine site can be remediated during operation and after closure. Monitoring during design can provide better operating procedures, which can minimize environmental and bonding costs. Monitoring during mining and processing operations can be used in process control to identify problems before they become major issues and while there is still a positive cash-flow to mitigate such issues. Finally, postclosure monitoring can minimize long-term costs and expedite bond release.

Most companies strive, for obvious economic reasons, to obtain the maximum benefit at minimum cost, meaning that the number of drillholes must be just sufficient to ensure definition

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Source: Adapted from ICMM 2008.

FIGURE 4.5 Health risk assessment cycle for mining operations

of the ore zone without costing more than necessary. The inevitable question that arises is *What is the optimum drillhole spacing?* The answer is *it depends!* The level of risk that management is willing to accept determines how rigorous data collection will be at the start of a project as well as during subsequent mine development. The cost of gathering information has to be weighed against the planning and operational risk to a mining company based on the reliability of the data compared to the importance or value of the ecological, regulatory, or economic component being studied. The *Handbook of Systems Engineering and Management* (Rouse 2009) describes the issue very well. (See also Engineering Systems and Risk sidebar.)

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ENGINEERING SYSTEMS AND RISK

"Engineering systems are almost always designed, constructed, and operated under unavoidable conditions of risk and uncertainty and are often expected to achieve multiple and conflicting objectives. The identification, quantification, evaluation, and trading off of risks, benefits and costs should constitute an integral and explicit component of the overall managerial decision-making process and should not be a separate, cosmetic afterthought." (Rouse 2009).

In mineral evaluation, risks to stakeholders, specifically stockholders, is a topic of great interest and concern. The mining and mineral processing community is very familiar with the concepts of data reliability. The level of confidence in key information is the distinction between proven and probable reserves. The figures are always estimates rather than precise calculations, with margins of error that should be recognized in the way the figures are expressed. In 2002, the CRIRSCO (Combined Reserves International Reporting Standards Committee, now known as the Committee for Mineral Reserves International Reporting Standards) was formed with the mission to continue coordination between member countries regarding international resources and reserves definitions. Considerable progress has been made toward widespread adoption of standards throughout the world. However, mining companies subject to U.S. Securities and Exchange Commission (SEC) regulations must comply with Industry Guide 7 when reporting ore reserves (SEC 2013). In 2012, the Society for Mining, Metallurgy and Exploration (SME) petitioned the SEC to update its Industry Guide 7 using as a model the 2007 SME Guide based on the CRIRSCO recommendations (SME 2007). As shown in Figure 4.6, the choice of the appropriate category of mineral resource depends on the quantity, distribution and quality of data available, the level of confidence that attaches those data, and the appropriateness of the estimation methodology applied. Those data include mining, metallurgical, economic, marketing, legal, environmental, social, and governmental factors, many of them with the potential to block a new mine or make serious problems for an established one.

As noted at the bottom of Figure 4.7, environmental factors are also taken into consideration. Specifically, the Canadian requirements address QA/QC and include the following environmental requirements (CIM 2010): baseline studies, tailings management, waste rock management, acid rock drainage (ARD) issues, closure and reclamation plans, and permitting schedule.

The 2007 SME Guide with international resources and reserves definitions or their precursors (i.e., Goudarzi 1984) were accepted as part of national reporting codes and guidelines by the regulatory agencies of Australia (1989), South Africa (2000), Canada (2001), and the United Kingdom (2001). The United Nations formally adopted these definitions in 1999. In 2007, the government of Chile approved a reporting code that includes these definitions. In 2008, the Pan-European Code was issued by the Pan-European Reserves and Resources Reporting Committee, and in 2011, the Russian Code was issued by the National Association for Subsoil Examination.

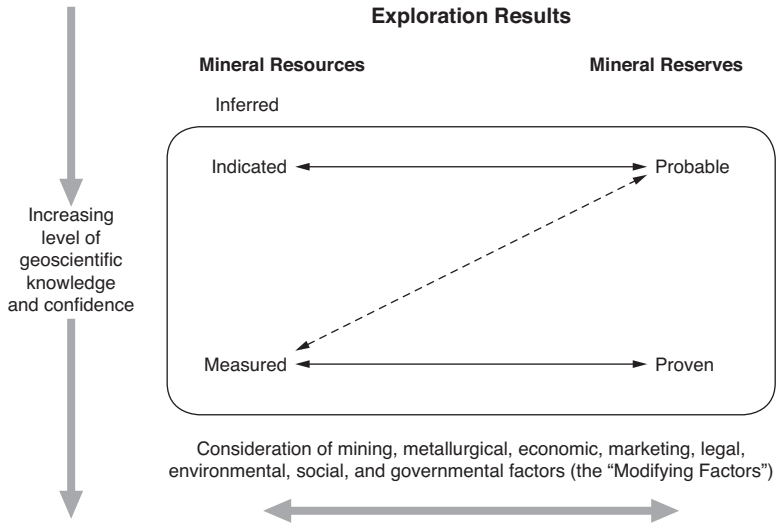
In a similar manner, planning and operational risks can be placed in perspective based on the value placed on the potentially impacted part of the environment and the reliability of the data. Environmental risk analyses for closure based on unreliable data can be underprotective or overprotective of ecosystems, with the result that there are added cost penalties on society and on industry to achieve these levels of protection. Some risks are potentially so serious, and the time for recovery so long, that risk reduction actions should be viewed as a kind of insurance premium

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ENDANGERED SPECIES AT HENDERSON, COLORADO

Carol C. Russell, U.S. Environmental Protection Agency

An example of the complications encountered when an endangered species appears to prefer an acid rock drainage environment, such as a mine tailings pond, is illustrated by the Henderson mine in central Colorado. The boreal toad (*Bufo boreas boreas*), listed as a State Endangered Species and a candidate for federal protection under the Endangered Species Act, only breeds in still or slow-moving acidic water such as the water found in a tailings impoundment. The mine took on the toad as their mascot and actively participated in the efforts to establish new (nonmine) habitat (Russell 2006).



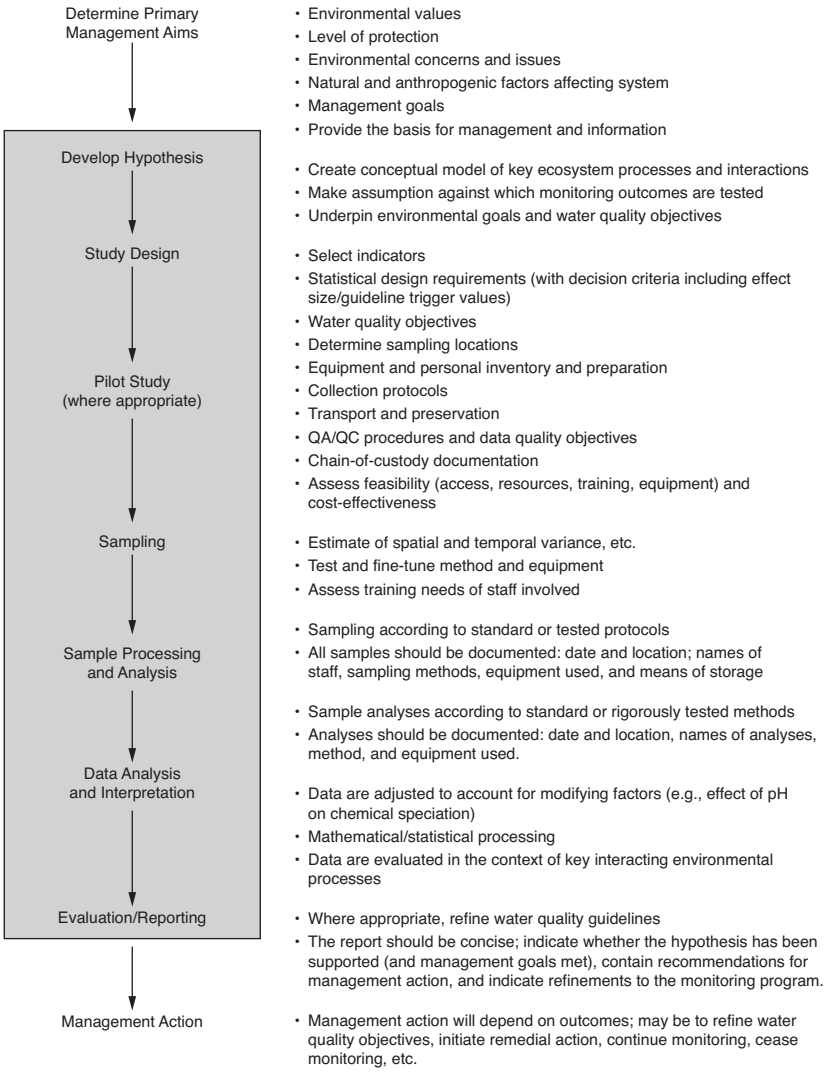
Source: SME 2007.

FIGURE 4.6 General relationships between exploration results, mineral resources, and mineral reserves

and initiated in the face of incomplete and uncertain data. The risks entailed in postponing action can be greater than the risks entailed in taking inefficient or unnecessary action. In short, the only way to decrease one's exposure to risk is to increase the reliability of the data or to decrease the risk using risk management practices. Other complications can enter into the decision of if, when, and how to remediate an area. For example, Leadville, Colorado, depends on the tourist industry for much of its economy, and the complete removal of waste rock piles would detract from the visual effect of the mining town. Therefore, special coverings of the waste rock piles were designed to protect the visual effect as well as reduce the ARD. In some areas, endangered and other species depend on mining influenced water and must be protected, such as at the Henderson mine in Colorado (see Endangered Species sidebar).

Several strategies or processes are involved in risk analysis, as shown previously in Figure 4.3. The HRA cycle for mining operations is shown in Figure 4.5 (ICMM 2008), although a simpler approach combining risk assessment with risk management is provided by Dowd (2002):

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Source: Adapted from the Monitoring and Reporting Guidelines and the framework for designing a wetland monitoring program adopted by the Ramsar Wetland Convention (Ramsar Convention 1996; Finlayson 1996; ANZECC and ARMCANZ 2000).

FIGURE 4.7 Procedural framework for the monitoring and assessment of water quality

1. Identify the risk and the sources.
2. Assess and quantify the risk, including assessment of the exposure, pathways, and transport modes.
3. Determine the minimum acceptable level of risk and the consequences of the risk.
4. Manage the residual risk by developing proper sampling and monitoring plans.
5. Reduce the risk to a minimum for adequate quality assurance and quality control.

4.3 INTERNATIONAL GUIDANCE DOCUMENT COMMONALITIES

Many countries and organizations have endorsed a data quality objectives structure for planning a sampling and monitoring program as described in Table 4.4. The following sections provide an

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overview of frameworks in various jurisdictions and by different organizations. The basic components of the international guidance documents are very similar based on the information readily available in electronic form. Much more information is available from listed references.

4.3.1 Australian and New Zealand Sampling and Monitoring Programs

The Australian and New Zealand monitoring framework focuses on risk management by protecting water quality, rather than focusing on development of sampling and monitoring guidelines. The Australian guidelines also provide risk-based decision frameworks to help the user refine guideline trigger values for application at local and/or regional scales. The guidelines initially used a very deterministic view of ecosystems, assuming that factors controlling ecosystem function could be identified and managed to prevent problems. However, now they are more inclusive of the complexity of mining and processing activities as well as potential impacts to receptors. Monitoring includes both observation (e.g., recording information about the environment) and investigation (e.g., manipulative studies such as toxicity tests where environmental conditions are controlled). Although there is great reliance on the use of biological indicators, all monitoring relies on sound practice in environmental science, as shown in Figure 4.7, including

- Explicit written definition of the sampling site, project objectives, a hypothesis, and the sampling protocol that will support the work;
- The definition of sampling sites, sampling frequency, and spatial and temporal variability that will permit appropriate statistical methods to be used;
- Rigorous attention to field and laboratory quality control and assurance;
- Incorporation of a pilot study to test the sampling protocol and determine spatial and temporal variability; and
- Mitigation measures.

Specifically, the Australian and New Zealand guidelines reference procedures for determining an unacceptable level of change (ANZECC and ARMCANZ 2000).

Environment Australia also developed an internal QA/QC document for the Australian River Assessment System in New South Wales. This was undertaken to ensure that only QA data were used in all aspects of model development, model testing, and river site assessment addressing issues of lineage, positional accuracy, attribute accuracy, logical consistency, and completeness. Past data collection and assessment demonstrated that a screening process on data was needed to ensure that it was adequate to make decisions on river conditions (Waddell 2000).

4.3.2 European Union's Sampling and Monitoring Program

The European Union water framework directive guidance provides exceptional insight and diagrams of basic questions that need exploration before a monitoring program is implemented (European Communities 2003). The directives for water and waste are media-focused based on changes to the quality of the water, soil, or air (Figure 4.8). For water, the directive provided guidance not only for surface water but also for groundwater and wetlands. However, specific monitoring guidance for mining-related sampling of water was limited.

The waste framework directive, in contrast, has provided a great deal of research on this subject of monitoring mining; specifically, several guidance documents regarding characterization of waste from extractive industries, including

- Static testing for determination of acid potential and neutralization potential of sulfidic waste,

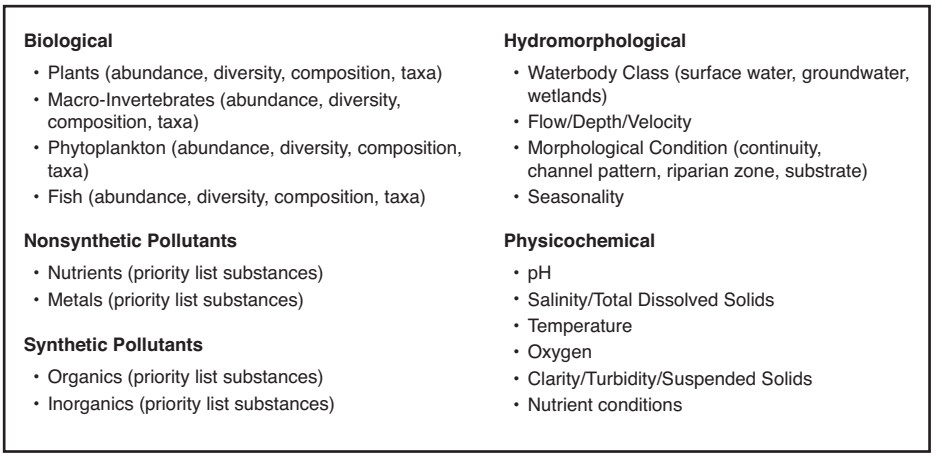
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TABLE 4.4 International sampling and monitoring elements guidance document comparison

	ADTI-MMS*	Australia and New Zealand†	European Union‡	Canada§	USEPA**	GARD Guide††
1.	Define the questions and objectives	Defines values, goals, water quality objectives, and level of protection	Issues for consideration: objectives, variability	Define the project background and goals	State the problem; identify the goals	Objectives of monitoring
2.	Develop site conceptual models	Determine appropriate guidelines—local environmental conditions	Development of a conceptual understanding	Develop a conceptual site model	Define the boundaries of the study	Conceptual and dynamic system model development
3.	Evaluate the risks, consequences, and costs	Define specific water quality to be achieved	Acceptable level of risk, precision, and confidence	Establish the investigation objectives		Risk-based approach to monitoring
4.	Identify the types, quantity and quality of data, and information needed				Identify the information inputs and analytic approach; specify performance criteria	
5.	Define protocols	Establish monitoring and assessment program after defining acceptable performance or decision criteria	Quality assurance/ quality control, standard methods			
6.	Develop the sampling and monitoring plan	Monitoring plans are left to the local agencies		Prepare a sampling and analysis plan	Develop the plan for collecting the data	Monitoring program development
7.	Conduct the sampling and monitoring plan	Monitoring is undertaken on a case-by-case basis	Implementation of quality assurance programs	Conduct the field investigation program		
8.	Analyze and manage data	Initiate management response; "Trigger values"	Expression of results	Validate and interpret the data		Data management and interpretation
9.	Decision making	Stakeholder process	Transparency of process			Auditing

* ADTI-MMS (Acid Drainage Technology Initiative–Metal Mining Sector; McLemore et al. 2004, 2007, 2009).
† Australian and New Zealand Environment and Conservation Council (ANZECC and ARMCANZ 2000).
‡ European Communities (2003); European Commission (2012).
§ Environment Canada (2004, 2005, 2012, 2013); MEND (Mine Environment Neutral Drainage; www.mend-nedem.org).
** USEPA (2006, forthcoming).
†† *Global Acid Rock Drainage Guide* (INAP 2009).

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Source: Adapted from European Communities 2003.

FIGURE 4.8 European Commission water framework—selection of quality elements for transitional waters

- Kinetic testing for assessing acid generation potential of sulfidic waste from extractive industries,
- An overall guidance document for characterization of waste from extractive industries, and
- Sampling and analysis of weak acid dissociable cyanide discharged into tailings ponds.

4.3.3 Canadian Sampling and Monitoring Program

The MEND (Mine Environment Neutral Drainage) program in Canada is known for its cooperative research program between Canadian mining companies and provincial/territorial and federal departments. Since 1989, MEND has worked to develop technologies to prevent and control acidic drainage from mining operations. The program is directed by a multistakeholder committee, with members from the mining industry, federal and provincial governments, and nongovernment organizations. Specific publications on sampling and monitoring include the following categories: (1) prediction, (2) prevention and control, (3) treatment, (4) monitoring, and (5) technology transfer. Publications of particular note are

- *Field Sampling Manual for Reactive Sulphide Tailings* (CANECT Environmental Control 1989),
- *Review of Waste Rock Sampling Techniques* (SENES Consultants Limited et al. 1994),
- *Handbook for Waste Rock Sampling Techniques* (SENES Consultants Limited 1994),
- Appendix A: Technical Summary Note in *Guideline Document for Monitoring Acid Mine Drainage* (Terrestrial and Aquatic Environmental Managers and SENES Consultants Limited 1997), and
- *Application of Remote Sensing and Geophysics to the Detection and Monitoring of Acid Mine Drainage* (Paterson, Grant and Watson Limited and Geomatics International, Inc., 1994).

In addition, in 2012, Environment Canada published *Metal Mining Technical Guidance for Environmental Effects Monitoring*. Chapter 2, titled "Study Design, Site Characterization and General Quality Assurance/Quality Control," mainly focuses on aquatic effects to fish of mining

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in Canada, but it does provide for data quality measures derived from intended data uses (e.g., hypotheses to be tested, summary statistics involved, and total uncertainty that can be tolerated; Environment Canada 2012). Section 2.2.1, Site Characterization, in that document focuses on specific media (soil, water, air) and specific contaminants within that media. The Canadian section on conceptual models is particularly useful in that details of different sites are provided as examples. Also, the document provides detailed checklists for site assessment leading to a risk assessment. The Canadian Soil Quality Guidelines for the Protection of Environment and Human Health summary tables (CCME 2007) are being revised.

British Columbia has also been very active on guidance for the environmental effects of mining with the recent *Water and Air Baseline Monitoring Guidance Document for Mine Proponents and Operators* (Ministry of Environment 2012). This guidance document outlines and defines the baseline study requirements and information considerations necessary to propose a mineral development project in the province of British Columbia. By providing these requirements to the proponent early in the project evaluation stage (exploration), the Ministry of Environment aims to promote effective study design and duration and to ensure that information collection and data usage/interpretation will assist in the initial project evaluation and be useful throughout the development, operation, and closure of a mine.

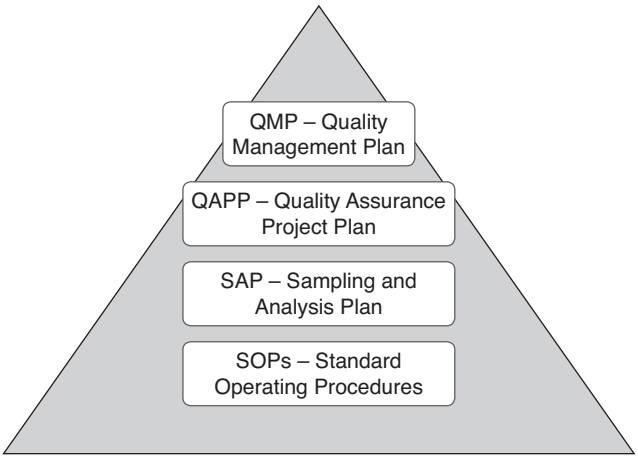
4.3.4 USEPA Sampling and Monitoring Program

The USEPA sampling and monitoring program has served as the framework for numerous other international environmental QA/QC guidance documents. The USEPA Quality System (the sampling and monitoring program) is based on developing performance and acceptance criteria, or data quality objectives (DQOs), and qualitative and quantitative statements that define the purpose for the data collection effort, clarify the kind of data needed, and specify the limits on decision errors needed for the study.

A sampling and monitoring program can be composed of several steps to document the process of QA/QC (see Section 3.13). A goal of USEPA's Quality System is to ensure that environmental programs and decisions are supported by data of the type and quality needed and expected for their intended use. The DQO process is a series of steps that serve as a guide for the resource-effective acquisition of environmental data and apply to both decision making and estimation. It is used to develop performance and acceptance criteria that clarify the objectives of the investigation, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions (USEPA 2006). The Quality System (see USEPA 2006) includes a series of guidance documents that relate to one another, as shown in Figure 4.9.

The quality assurance project plan (QAPP) describes the policy, organizational, and functional activities necessary to collect data that will stand up to legal and scientific scrutiny (USEPA 2002a). The quality management plan describes processes and procedures at the organizational level, management and staff functional responsibilities, and line of authority in the USEPA guidance QA/R-2 (USEPA 2001c). The QAPP (USEPA 2002a) is the document that details the who, what, when, where, why, and how of the project objectives (DQOs) found in QA/R-5 (USEPA 2001b) and QA/G-5 (USEPA 2002b). The field sampling plan provides the field sampling details for the sampling event. The sampling and analyses plan provides the field and analytical details for the sampling event(s) and specifies the process for obtaining environmental data of sufficient quality to satisfy the project objectives. Sampling and monitoring plans are discussed further in Chapter 6, and examples are provided in Appendix 4. Standard operating procedures describe

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Courtesy of Mary P. Goldade.

FIGURE 4.9 USEPA's Quality System

specific sampling techniques, field analytical methodologies, or analytical methods, many of which are described in Appendix 3.

Three guidance documents from the United States focus on metals and mining sampling and monitoring:

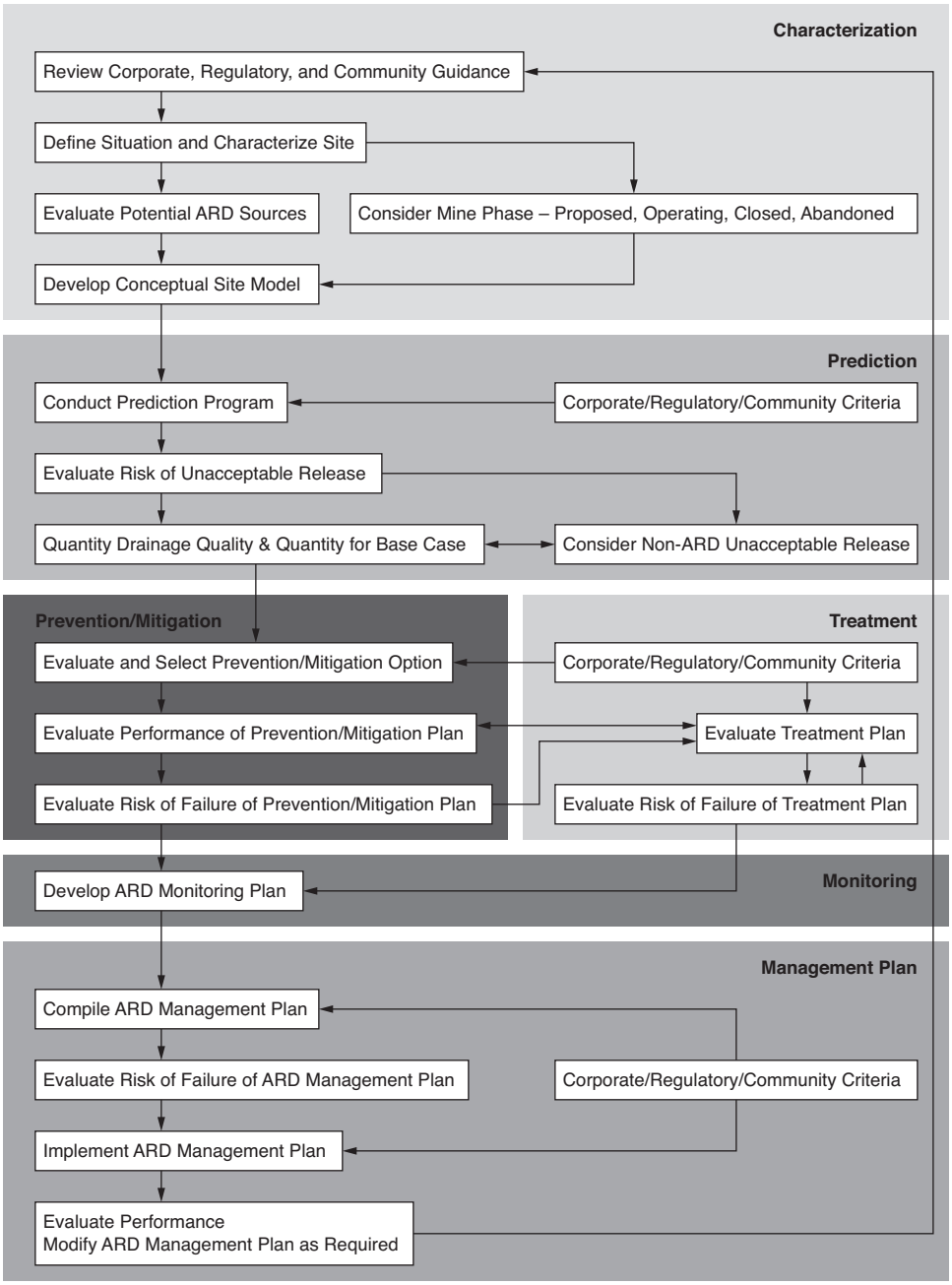
1. *Framework for Metals Risk Assessment* (USEPA 2007, 2011)
2. *EPA and Hardrock Mining: A Source Book for Industry in the Northwest and Alaska* (USEPA 2003)
3. *Abandoned Mine Site Characterization and Cleanup Handbook* (USEPA 2001a)

In addition, Karl Ford, formerly with the U.S. Department of the Interior Bureau of Land Management, wrote the book *Risk Management Criteria for Metals at BLM Mining Sites* (Ford 2004). Furthermore, the U.S. Geological Survey has numerous publications on the subject both through the U.S. government press and in the peer-reviewed literature (see http://crustal.usgs.gov/projects/minewaste/).

4.3.5 *Global Acid Rock Drainage Guide*

The *Global Acid Rock Drainage Guide (*GARD Guide) is a Web-based, actively updated resource intended as a state-of-practice summary of the best practices and technology dealing with the prediction, prevention, and management of drainage produced from sulfide mineral oxidation, often termed *acid rock drainage*, *saline drainage*, *acid mine drainage* or *acid and metalliferous drainage*, *mining influenced water*, and *neutral mine drainage*. It was developed on behalf of the International Network for Acid Prevention, an international consortium of mining companies, by consultants, academicians, mine operators, and regulators to address issues related to sulfide mineral oxidation. In Chapter 8 of the GARD Guide, *monitoring* is defined as the process of routinely, systematically, and purposefully gathering information for use in management decision making. Mine site monitoring characterizes environmental changes from mining activities to assess conditions on the site and possible impacts to receptors. Monitoring includes both observation (e.g., recording information about the environment) and investigation (e.g., manipulative studies such as toxicity tests where environmental conditions are controlled). An example of the use of monitoring in

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Source: INAP 2009.

FIGURE 4.10 GARD Guide overall management plan

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management decision making was provided to assess the effectiveness of mitigation measures to minimize the effects of environmentally detrimental processes such as mining influenced waters and implementation of adjustments to mitigation measures. Figure 4.10 is a flow diagram of the incorporation of monitoring into a mining company's decision-making process.

4.4 SUMMARY

In summary, this chapter is not proscriptive but is designed as a thought-provoking method to guide users in articulation, investigation, analysis, and documentation to provide sufficient, quality information to decision makers. In designing a monitoring program, the objectives of the sampling effort must be determined first. What questions need to be answered in order to make decisions? How detailed must the data be to answer the questions? The ability to assess environmental risks, compare them, and select strategies to reduce them all depend on the availability and sophistication of the relevant data and analytical tools. Based on the assessment of risks, the sampling program can be designed considering the necessary spatial and temporal resolution, and the number of samples and replicates needed to satisfactorily address the study objectives.

Documentation of uncertainties or limitations of the data or assumptions made are as important as the range of possible risks. The quality of the data necessary to answer the questions is based on the importance or value of the ecological, regulatory, or economic issues at risk. In fact, some long-term and widespread environmental problems should be considered relatively high risk even if the data on which the risk assessment is based are somewhat incomplete and uncertain. The risks entailed in postponing action can be greater than the risks entailed in taking inefficient or unnecessary action.

International guidance documents follow the same general format for implementation of a decision-based sampling and monitoring program. ADTI-MMS tailored these guidance documents for mining and mineral processing in an effort to standardize expectations in the same manner as the international minerals evaluation processes are coming together.

4.5 REFERENCES

ANZECC and ARMCANZ (Australian and New Zealand Environment and Conservation Council and Agriculture and Resource Management Council of Australia and New Zealand). 2000. *Australian and New Zealand Guidelines for Fresh and Marine Water Quality*. National Water Quality Management Strategy No. 7, Paper No. 4. www.environment.gov.au/water/publications/quality/pubs/nwqms-guidelines-4-vol1.pdf. Accessed 9/11/13.

Australian Government. 2008. *Risk Assessment and Management*. Commonwealth of Australia www.ret.gov.au/resources/Documents/LPSDP/LPSDP-RiskHandbook.pdf. Accessed 9/26/13.

Barton, B. 2010. *Murky Waters? Corporate Reporting on Water Risk*. Boston: Ceres. www.ceres.org/resources/reports/corporate-reporting-on-water-risk-2010. Accessed 9/16/13.

Beer, T., and Ziolkowski, F. 1995. *Environmental Risk Assessment: An Australian Perspective*. Supervising Scientist Report 102. www.environment.gov.au/ssd/publications/ssr/pubs/ssr102-printquality.pdf. Accessed 9/11/13.

Caldwell, J. 2010. Learning to live with risk. Mining.com. www.mining.com/2010/07/01/learning-to-live-with-risk/. Accessed April 2011.

CANECT Environmental Control. 1989. *Field Sampling Manual for Reactive Sulphide Tailings*. MEND Project 4.1.1. www.mend-nedem.org/reports/files/4.1.1.pdf. Accessed 9/11/13.

CCME (Canadian Council of Ministers of the Environment). 2007. Canadian soil quality guidelines for the protection of environmental and human health: Summary tables. Updated September 2007. In Canadian Environmental Quality Guidelines (1999). Winnipeg: CCME. www.popstoolkit.com/Tools/SitePrioritization/Files/Guidelines/SoilQ%20eco%20and%20human%20h.pdf. Accessed 12/20/13.

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Chapman, P.M., Allen, H.E., Godtfredsen, K., and Z'Graggen, M.N. 1996. Policy analysis, peer reviewed: Evaluation of bioaccumulation factors in regulating metals. *Environ. Sci. Technol*. 30(10):448A–452A.

CIM. 2010. CIM definitions standards for mineral resources and mineral reserves: Standing Committee on Reserve Definitions Adopted by CIM Council on November 27, 2010. http://web.cim.org/UserFiles/File/CIM_DEFINITON_STANDARDS_Nov_2010.pdf. Accessed 9/11/13.

Dowd, P.A. 2002. The assessment and analysis of financial, technical, and environmental risk in mineral resource evaluation. In *Deposit and Geoenvironmental Models for Resource Exploitation and Environmental Security*. Edited by A.G. Fabbri, G. Gaal, and R.B. McCammon. Netherlands: Kluwer. pp. 187–221.

ECHA (European Chemicals Agency). 2008. Appendix R.7.13-2: Environmental risk assessment for metals and metal compounds. In *Guidance on Information Requirements and Chemical Safety Assessment*. Guidance for the Implementation of REACH report. http://echa.europa.eu/documents/10162/13632/information_requirements_r7_13_2_en.pdf. Accessed 9/11/13.

Environment Canada. 2004. *National Air Pollution Surveillance Network Quality Assurance and Quality Control Guidelines*. AAQD 2004-1. Ottawa, ON: Environment Technology Centre.

Environment Canada. 2005. *Guidance for Fish Tissue Analysis for Mercury Using Non-Lethal Methods for the Metal Mining Environmental Effects Monitoring Program*. Report. www.ec.gc.ca/eem/pdf_publications/English/mm_fish_tissue.pdf. Accessed 9/11/13.

Environment Canada. 2012. *Metal Mining Technical Guidance for Environmental Effects Monitoring*. Report. www.ec.gc.ca/Publications/D175537B-24E3-46E8-9BB4-C3B0D0DA806D%5CCOM-1434---Tec-Guide-for-Metal-Mining-Env-Effects-Monitoring_En_02.pdf. Accessed on 9/11/13.

European Commission. 2012, Summary of EU legislation on mining waste. Directive 2006/21/EC of the European Parliament and of the Council on the Management of Waste from the extractive Industries. http://ec.europa.eu/environment/waste/mining/legis.htm and http://europa.eu/legislation_summaries/environment/waste_management/l28134_en.htm. Accessed 9/11/13.

European Communities. 2003 *Common Implementation Strategy for the Water Framework Directive (2000/60/EC)*. Guidance Document No 7: Monitoring Under the Water Framework. www.eurogeologists.de/images/content/panels_of_experts/hydrogeology/9E9DFd01.pdf. Accessed 9/11/13.

Failing, L., Gregory, R., and Harstone, R.M. 2007. Integrating science and local knowledge in environmental risk management: A decision-focused approach. *Ecolog. Econ*. 64:47–60.

Fairbrother, A., Wenstel, R., Sappington, K., and Wood, W. 2007. Framework for metals risk assessment. *Ecotoxicol. Environ. Safety* 68:145–227.

Finlayson C.M. 1996. Framework for designing a monitoring programme. In *Monitoring Mediterranean Wetlands: A Methodological Guide*. Edited by P. Tomas Vives. Slimbridge, United Kingdom; and ICN, Lisbon: Wetlands International. pp. 25–34.

Ford, K.L. 2004. *Risk Management Criteria for Metals at BLM Mining Sites*. Technical Note 390 (revised) BLM/RS/ST-97/001+1703. Denver, CO: U.S. Bureau of Land Management, National Science and Technology Center. www.blm.gov/nstc/library/pdf/TN390v04.pdf. Accessed 9/11/13.

Goudarzi, G.H. 1984. *Guide to Preparation of Mineral Survey Reports on Public Lands*. U.S. Geological Survey, Open-File Report 84-787. http://pubs.usgs.gov/of/1984/0787/report.pdf. Accessed 9/11/13.

GRI (Global Reporting Initiative). 2010. *Sustainability Reporting Guidelines and Mining and Metals Sector Supplement*. Global Reporting Report. https://www.globalreporting.org/reporting/sector-guidance/mining-andmetals/Pages/default.aspx. Accessed 9/11/13.

ICMM (International Council on Mining and Metals). 2006. *HERAG: Health Risk Assessment Guidance for Metals*. ICMM report. www.icmm.com/page/1213/health-risk-assessment-guidance-for-metals-herag. Accessed 9/11/13.

ICMM (International Council on Mining and Metals). 2007. *MERAG: Metals Environmental Risk Assessment Guidance*. www.icmm.com/page/1185/metals-environmental-risk-assessment-guidance-merag. Accessed 9/11/13.

ICMM (International Council on Mining and Metals). 2008. *A Sustained Commitment to Improved Industry Performance*. London: ICMM. www.corporate-citizenship.com/wp-content/uploads/Mining.pdf. Accessed 9/11/13.

ICMM (International Council on Mining and Metals). 2013. Sustainable development framework: 10 principles. London: ICMM. www.icmm.com/our-work/sustainable-development-framework/10-principles. Accessed 9/26/13.

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INAP (International Network for Acid Prevention). 2009. *Global Acid Rock Drainage Guide* (GARD Guide). www.gardguide.com. Accessed 9/11/13.

ISO (International Organization for Standardization) 31000:2009. *Risk Management—Principles and Guidelines*. www.iso.org/iso/home/standards/iso31000.htm. Accessed 12/16/13.

Kaplan, S., and Garrick, B.J. 1981. On the quantitative definition of risk. *Risk Anal.* 1:11–27. http://josiah.berkeley.edu/2007Fall/NE275/CourseReader/3.pdf. Accessed 9/11/13.

Kuipers, J.R., Maest, A.S., MacHardy, K.A., and Lawson, G. 2006. Comparison of predicted and actual water quality at hardrock mines—The reliability of predictions in Environmental Impact Statements. Butte, MT: Kuipers and Associates; Boulder, CO: Buka Environmental. http://pebblescience.org/pdfs/ComparisonsReportFinal.pdf. Accessed 9/11/13.

Lacy, W. 2010. *An Introduction to Geology and Hard Rock Mining*. Westminster, CO: Rocky Mountain Mineral Law Foundation. www.rmmlf.org/scitech/lacy/lacy.htm. Accessed 9/11/13.

McLemore, V.T., Russell, C.C., Smith, K.S., and the Sampling and Monitoring Committee of the Acid Drainage Technology Initiative–Metal Mining Sector. 2004. Sampling and monitoring for closure. SME Preprint No. 04-62. Littleton, CO: SME. www.onemine.org/view/?d=BB60F953960F2274EBE26EC2304AA37CA5BD2E24D37573C249E8B807896ED8E038402. Accessed 9/11/13.

McLemore, V.T., Smith, K.S., Russell, C.C., and the Sampling and Monitoring Committee of the Acid Drainage Technology Initiative–Metal Mining Sector. 2007. Sampling and monitoring for closure. In *Understanding and Responding to Hazardous Substances at Mine Sites in the Western United States*. Edited by J.V. DeGraff. Reviews in Engineering Geology, Vol. 17. Boulder, CO: Geological Society of America. pp. 171–180.

McLemore, V.T., Russell, C.C., and Smith, K.S. 2009. ADTI-MMS sampling and monitoring for the mine-life cycle: Securing the Future and 8th ICARD, June 23–26, Skelleftea, Sweden, conference proceedings. www.proceedings-stfandicard-2009.com/pdfer/Virginia_McLemore_B4_T3_ADTI-MMS-Sampling-and-Monitoring-for-the-Mine-life-Cycle.pdf. Accessed 2/8/10.

Ministry of Environment. 2012. *Water and Air Baseline Monitoring Guidance Document for Mine Proponents and Operator*: British Columbia report. www.env.gov.bc.ca/epd/industrial/mining/pdf/water_air_baseline_monitoring.pdf. Accessed 9/11/13.

Miranda, M., and Sauer, A. 2010. Mine the gap: Connecting water risks and disclosure in the mining sector. WRI Working Paper. Washington, DC: World Resources Institute. http://pdf.wri.org/working_papers/mine_the_gap.pdf. Accessed 9/11/13.

Paterson, Grant and Watson Limited and Geomatics International, Inc. 1994. *Application of Remote Sensing and Geophysics to the Detection and Monitoring of Acid Mine Drainage*: MEND Project 4.6.3. www.mend-nedem.org/reports/files/4.6.3.pdf. Accessed 9/11/13.

Ramsar Convention. 1996. Resolution VI.1. In *Proceedings of the Sixth Meeting of the Conference of the Contracting Parties of the Convention on Wetlands, Resolutions and Recommendations*, Ramsar Convention Bureau, Gland, Switzerland.

Rouse, W.B. 2009. *Handbook of Systems Engineering and Management*. New York: John Wiley and Sons.

Russell, C. 2006. Mine drainage challenges from A to Z in the United States. In *Proceedings, 7th International Conference on Acid Rock Drainage (ICARD 7)*. Lexington, KY: American Society of Mining and Reclamation. pp. 1785–1802, www.imwa.info/docs/imwa_2006/1785-Russell-CO.pdf. Accessed 9/11/13.

SEC (U.S. Securities and Exchange Commission). 2013. Industry Guide 7: Description of Property by Issues Engaged or To Be Engaged in Significant Mining Operations. Washington, DC: SEC. http://web.cim.org/standards/documents/Block474_Doc32.pdf. Accessed 9/11/13.

SENES Consultants Limited. 1994. *Handbook for Waste Rock Sampling Techniques*. MEND Project 4.5.1-2. www.mend-nedem.org/reports/files/4.5.1-2.pdf. Accessed 9/11/13.

SENES Consultants Limited, Golder Associé Ltée, and Laval University. 1994. *Review of Waste Rock Sampling Techniques*. MEND Project 4.5.1-1. www.mend-nedem.org/reports/files/4.5.1-1.pdf. Accessed 9/11/13.

SME (Society for Mining, Metallurgy and Exploration). 2007. The SME Guide for Reporting Exploration Results, Mineral Resources, and Mineral Reserves (The 2007 SME Guide). Littleton, CO: SME. www.crirsco.com/usa_sme_guide_2007.pdf. Accessed 9/11/13.

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Smith, K.S. 2007. Strategies to predict metal mobility in surficial mining environments, Ch. 3. In *Understanding and Responding to Hazardous Substances at Mine Sites in the Western United States*. Edited by J.V. DeGraff. Reviews in Engineering Geology, Vol. 17. Boulder, CO: Geological Society of America. pp. 25–45.

Smith, K.S., and Huyck, H.L.O. 1999. An overview of the abundance, relative mobility, bioavailability, and human toxicity of metals. In *The Environmental Geochemistry of Mineral Deposits—Part A: Processes, Techniques, and Health Issues*. Reviews in Economic Geology, Vol. 6A. Littleton, CO: Society of Economic Geologists. pp. 29–70.

Suter II., G.W. 1993. *Ecological Risk Assessment*. Boca Raton, FL: Lewis Publishers.

Terrestrial and Aquatic Environmental Managers and SENES Consultants Limited. 1997. Appendix A: Technical summary note. In *Guideline Document for Monitoring Acid Mine Drainage*. MEND Project 4.5.4. www.mend-nedem.org/reports/files/4.5.4%20Appendix.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2001a. *Abandoned Mine Site Characterization and Cleanup Handbook*. EPA910-B-00-001. www.epa.gov/superfund/policy/remedy/pdfs/amscch.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2001b. *EPA Requirements for Quality Assurance Project Plans* (EPA QA/R-5): U.S. Environmental Protection Agency, EPA/240/B-01/003. www.epa.gov/quality1/qs-docs/r5-final.pdf. Accessed 12/20/13.

USEPA (U.S. Environmental Protection Agency). 2001c. *EPA Requirements for Quality Management Plans* (EPA QA/R-2): U.S. Environmental Protection Agency, www.epa.gov/quality/qs-docs/r2-final.pdf. Accessed 12/20/13.

USEPA (U.S. Environmental Protection Agency). 2002a. *Guidance on Choosing a Sampling Design for Environmental Data Collection for Use in Developing a Quality Assurance Project Plan* (EPA QA/G-5S): U.S. Environmental Protection Agency, www.epa.gov/QUALITY/qs-docs/g5s-final.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2002b. *EPA Guidance for Quality Assurance Project Plans* (EPA QA/G-5): U.S. Environmental Protection Agency, www.epa.gov/quality/qs-docs/g5-final.pdf. Accessed 12/20/13.

USEPA (U.S. Environmental Protection Agency). 2003. *EPA and Hardrock Mining: A Source Book for Industry in the Northwest and Alaska*. EPS report. http://yosemite.epa.gov/R10/WATER.NSF/Sole+Source+Aquifers/hardrockmining. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2006. *Guidance on Systematic Planning Using the Data Quality Objectives Process*. EPA QA/G-4. Office of Environmental Information EPA/240/B -06/001. www.epa.gov/QUALITY/qs-docs/g4-final.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2007. *Framework for Metals Risk Assessment*. EPA 120/R-07/001. www.epa.gov/raf/metalsframework/pdfs/metals-risk-assessment-final.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2011. Framework for Metals Risk Assessment Web page. Office of the Science Advisor. www.epa.gov/raf/metalsframework/index.htm. Accessed 12/20/13.

USEPA (U.S. Environmental Protection Agency). (forthcoming). Guidance Manual for Environmental Site Characterization in support of Environmental and Human Health Risk Assessment.

Waddell, N. 2000. *Internal Quality Assurance and Quality Control for AusRivAS in NSW*. Monitoring River Health Initiative Technical Report No. 2b, Commonwealth of Australia and NSW Environment Protection Authority, Canberra and Sydney. www.environment.gov.au/water/publications/environmental/rivers/nrhp/pubs/nsw-quality.pdf. Accessed 9/11/13.

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CHAPTER 5

The Planning Process

Carol C. Russell, Kathleen S. Smith, and Virginia T. McLemore

5.1 INTRODUCTION

The planning process is a structured, systematic approach for obtaining data and providing information to make decisions. The basis of the planning process is the definition of data quality objectives (DQOs) that result in the sampling and monitoring plan, described in Chapter 6. The DQO process is used to develop performance and acceptance criteria that clarify study objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions (USEPA 2006). By selecting a representative part of a population (sampling) and observing or checking it over a period of time (monitoring), these data can be analyzed to provide information to guide decision making. Sampling strategies should be site specific and consider characteristics such as climate, geology, proximity of viable resources, and unique characteristics of the site. The primary purpose for using a quality planning process is to ensure that data is collected that is

- Needed to make decisions,
- Can be used to make decisions, and
- Will be used to make decisions.

In designing a monitoring system, the first four steps shown in Figure 4.1 in Chapter 4 constitute the planning process portion of the sampling and monitoring program. They include

- Define the questions and objectives (who is asking the question?);
- Develop the site conceptual model;
- Evaluate the risks, consequences, and costs of not sampling and monitoring; and
- Decide what is at risk if the information is incorrect or not available (how sufficient and complete the data should be to answer the questions).

Examples of sampling and monitoring programs are provided in the Types of Monitoring Programs sidebar and in Appendix 4.

5.2 DEFINE QUESTIONS AND OBJECTIVES

Defining the objectives may seem simple, but actually development of the objectives is the most important and sometimes the most difficult component of the sampling and monitoring program. What questions are being asked by decision makers and what are their objectives? Who are these decision makers, what decisions are they making, and what information do they need to make better decisions? High-quality, credible scientific information is undeniably the best foundation for environmental decision making. Quite often, the questions are associated with environmental

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TYPES OF MONITORING PROGRAMS

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Different types of monitoring programs need to be considered: compliance monitoring, trend monitoring, impact monitoring, and survey monitoring. *Compliance monitoring* is used to determine if permit objectives are being met. The objectives are site specific, but the data obtained can be used for other purposes such as modeling or prediction. *Trend monitoring* is used to detect changes over time, typically 10 years or more, to ensure that the trends are detected, and the program should be consistent throughout the entire sampling period (i.e., frequency, location, time of day, sampling and analytical techniques employed). An example of trend monitoring would be measurements of water quality in monitoring wells surrounding a tailings facility. *Impact assessment monitoring* is used to identify, characterize, and measure the effects of a mine facility or event on the environment, such as water quality. It is best that both test and control sites are included. A baseline assessment to determine premining conditions would be an example of impact assessment monitoring. *Survey or inventory monitoring* is used to characterize the site conditions at a specific location and time. A watershed approach is generally the most effective.

compliance. Consider the questions more broadly to incorporate other sustainability concerns including the economic and social aspects of the mining and processing operation in addition to environmental concerns. Potential questions include the following:

- What is the current status of the environment?
- Are there any aspects of the environment of particular value or concern?
- What are the problems?
- What is the nature and extent of the problem?
- How effective is the solution?
- How much will the mitigation cost, and do the stakeholders agree?

Mining and processing operations are as unique as the challenges associated with them. However, over time a few key lessons have been learned about answering questions regarding potential impacts at mining sites. First, knowing the environmental goals throughout the mine life cycle is essential. Second, the use of a conceptual model helps to evaluate risks by looking at the sources of potential contamination, the entity to be protected (receptor), such as human health or endangered species, and the pathway between the two. Third, defining risks at the mine site helps to define questions: from the risk of a landslide to the costs of Superfund listing. Fourth, the types of information needed to answer questions often revolve around water where using a conceptual model for the preparation of a water balance calculation throughout the mine life cycle is of paramount importance. The water balance will help to answer the inevitable question of *Will there be a discharge of mine dewatering or process wastewater during operations and/or closure, and will the discharge meet water quality standards?* Fifth, the question of mitigation measures revolves around identifying the measures to be taken at what times, in what manner, and by whom.

A corporate strategy to address environmental risks is similar to that needed for mine planning for maintaining ore grade, and they should be performed concurrently. Decision making based on scientific/engineering information assists in the evaluation of the operation's viability and results in time and cost savings. The need for additional data collection or the costly mistake of reanalysis at lower detection levels can easily be avoided with proper planning. Regulatory

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agencies would also be glad to have sufficient data to evaluate the potential environmental impacts in an efficient and effective manner.

5.2.1 Determine Data Quality Objectives

For decision problems, the DQOs are typically expressed as tolerable limits on the probability or chance (risk) of the collected data leading one to making an erroneous decision. The concept of intended use of the data is used to set the context for planning activities and focus the attention of the planning team. For example, one company's objective was to never exceed the water quality standard for copper in their water discharge. The intended use of the data was to assure the president of the company that they were in legal compliance. In order to ensure that 100% of the time that they would not discharge in violation, the average discharge concentrations needed to be reduced by more than half and a new treatment technology would need to be developed. From a cost-effective perspective, the company may have been better off to have the objective be in compliance for 98% of the time with a ±2% uncertainty. For estimation problems, the DQOs are typically expressed in terms of acceptable uncertainty (e.g., width of an uncertainty band or interval) associated with a point estimate at a desired level of statistical confidence.

5.2.2 Define the Confidence Level

A data collection design specifies the type, number, location, and physical quantity of samples and data, as well as the quality assurance/quality control (QA/QC) activities that will ensure that sampling design and measurement errors are managed sufficiently to meet the performance or acceptance criteria specified in the DQOs. As discussed previously, prior to beginning a sampling and monitoring program, it is necessary to define the reasons for sampling, the questions to be answered, and the desired degree of confidence in the answer(s). One might ask

- How much data are needed?
- What quality is sufficient?

The desired degree of confidence should be defined, as much as possible, at the inception of an investigation. A low level of certainty in the data can lead to erroneous interpretations and flawed decisions, but a higher level of certainty generally requires more sampling and higher costs. The degree of QA/QC should be in proportion to the degree of confidence that is required for the decisions to be made. Many situations exist where high-confidence data are necessary. For example, if expensive decisions are going to be made or if lives are at stake based on the outcome of the investigation, then high-confidence data are warranted. Other situations in which high-confidence data are likely desirable are for risk assessments or when data will be used in litigation. In one instance, a sampling plan was developed for the sole purpose of getting two people into the field together to allow them to talk. The quality of the data was immaterial. Table 5.1 provides an overview of the sources of uncertainty and variability.

It is necessary to select both the type of confidence and the level of confidence desired in the investigation. Types of confidence include statistical confidence and professional confidence. Confidence level is a statistical concept, which is based on probability, discussed further in Chapter 7.

Environmental samples are generally heterogeneous and subject to variability. This variability produces a pattern, which is called a *distribution*. The distribution can be described mathematically and graphically. The best-known theoretical distribution is the bell-shaped normal distribution. Most environmental and chemical data more closely follow a log-normal distribution, where the logarithms of data approximate a bell-shaped curve. A number of statistical tests and descriptions can be derived for normal (or log-normal) distributions. For example, the confidence interval is

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TABLE 5.1 Variability versus uncertainty in risk assessment

For uncertainty analysis, it is helpful to verify whether the parameter under consideration is characterized by variability, uncertainty, or both. This verification is based on the definition of *variability* and *uncertainty* and the scope of the assessment. An overview of sources of uncertainty and variability in risk assessments of metals is given in the table below.

	Variability	Uncertainty
	• Not reducible through further research • Probability distribution represents real variations actually occurring	• Reducible through further research • True value is somewhere in the probability distribution
Exposure	• Temporal variability of release • Spatial variability of soil/sediment/aquatic characteristics and background concentrations (regional assessment)	• Uncertainty of models • Uncertainty of physiochemical properties, partition coefficients, removal rates • Spatial variability of soil/sediment/aquatic characteristics and background concentrations (local assessment)
Effects	• Intra- and inter-species sensitivity • Inter- and intra-laboratory variability • Endpoint differences • Spatial (and temporal) variability of physiochemical characteristics determining bioavailability	• Probability distribution uncertainty (e.g., threshold versus nonthreshold distribution) • Diversity representativeness • Sampling uncertainty • Lab to field extrapolation

Source: Adapted from MERAG 2007.

a range of values that has a specified probability of containing the population parameter being estimated. In the case of the mean, it is the range of values that trap the population mean a certain percentage of the time (Figure 5.1).

Confidence limits are the lower and upper boundaries (or values) of a confidence interval. The confidence level is the probability value associated with a confidence interval (i.e., the probability that the population value is contained within the interval) and is often expressed as a percentage (Figure 5.1). The confidence level is an important detail to consider when designing a sampling plan (USEPA 2006).

5.2.3 Risks If the Data Are Unavailable or Incorrect

There is an entire field of science dedicated to a methodology of inquiry that is appropriate for cases where "facts are uncertain; values are in dispute; stakes are high and decisions urgent" (Funtowicz and Ravetz 1991). It is primarily applied in the context of long-term issues where there is less available information than is desired by decision makers and stakeholders. Practically speaking, the most significant risk to a mining company is time, and therefore money lost, when data are unavailable or incorrect going into the environmental permitting process. The Tale of Two Mines sidebar demonstrates the importance of defining objectives and making sure one has the information needed. In this sidebar, characteristics on the mines have been obscured, but the issues and relative span of times between the two approvals are real.

5.3 DEVELOP SITE CONCEPTUAL MODELS

The most important and probably the simplest tool for evaluating the environmental impacts of a project is the development of a conceptual model. The creation of a conceptual model for premining (exploration), during the operations, closure, and postclosure is an important step in prediction of potential environmental impacts at mining projects. The conceptual model should

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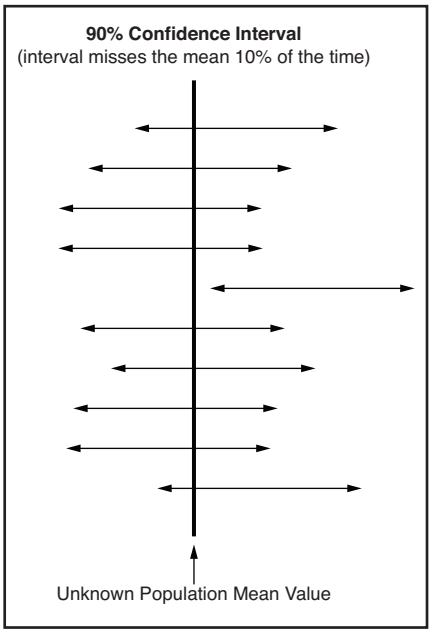


FIGURE 5.1 Concept of a 90% confidence interval

TALE OF TWO MINES

Carol C. Russell, U.S. Environmental Protection Agency

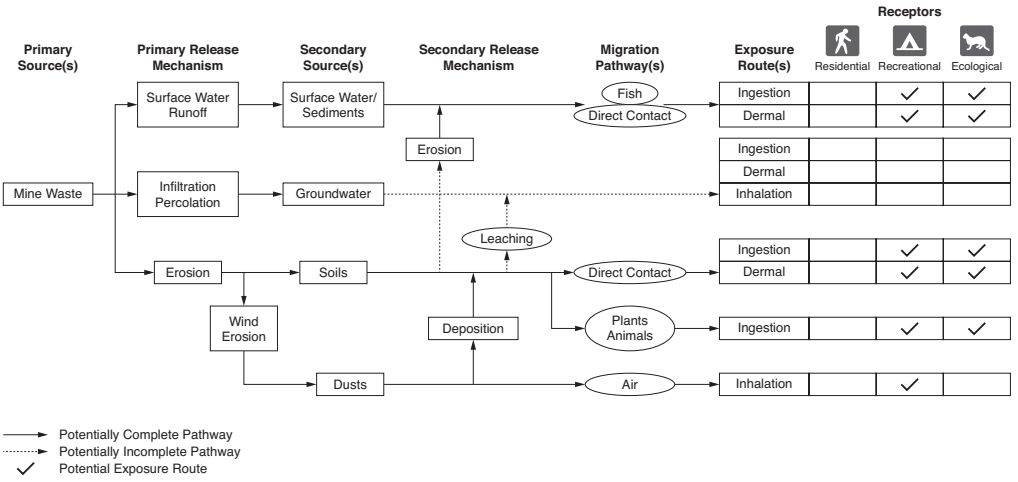
Two mining environmental impact statements (EISs) were simultaneously undergoing review by the same personnel within the same regulatory agencies. Mine One had little appropriate data to answer questions the regulatory agencies were asking: Would discharges from the mine meet water quality standards? Would wetlands and endangered species be impacted? Would the proposed water management system be adequate? Data were ±20 years out of date, with detection levels above standards, utilizing outmoded analytical methods, including sampling locations that were not applicable to the new mine design.

In comparison, Mine Two was proactive. They identified constituents of concern, implemented a sampling and monitoring program in cooperation with a local stakeholders group, and designed and implemented all necessary mitigation measures.

Mine Two got their permit in approximately a year. Mine One, after two EISs with adverse ratings and several lawsuits, has yet to receive their permit after more than 20 years.

be used to enable experts from all disciplines to communicate effectively with one another, resolve issues concerning the site, and facilitate the decision-making process. The conceptual model is used to integrate all site information and to determine whether information, including data, is missing (data gaps) and whether additional data need to be collected at the site. A conceptual model is considered to be a dynamic tool that will allow for hypothesis testing of the concepts. Furthermore, the model is used to facilitate the selection of mitigation alternatives and to evaluate the effectiveness of remedial actions in reducing the exposure of environmental receptors to contaminants.

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Source: Ford 2004.

FIGURE 5.2 Mine water conceptual model for human and ecological risk

5.3.1 Review Existing Data

The basis of a conceptual model is currently available data. Because new data collection can be expensive and time-consuming, it is important to conduct a literature and company review of all existing data and information concerning the site. This is a step that is often overlooked or cut short. These data are not merely limited to the mining company and consultants but can include data from local, state, and federal agencies; environmental groups; universities; and other entities. It is helpful to consider how future data collection can make use of or complement existing data, and how existing data can be used to help determine baseline conditions. Local land-use plans need to be investigated and nearby sensitive land-use areas identified (e.g., conservation areas, critical habitats, wetlands, national parks, cultural sites, recreational areas). It is also important to collect information that provides the *social* setting of the area (e.g., demographics; land ownership; proximity to structures, roads, and waterways; cultural and community features). This existing information can be used to build the initial conceptual model.

5.3.2 Purpose and Use of the Conceptual Model

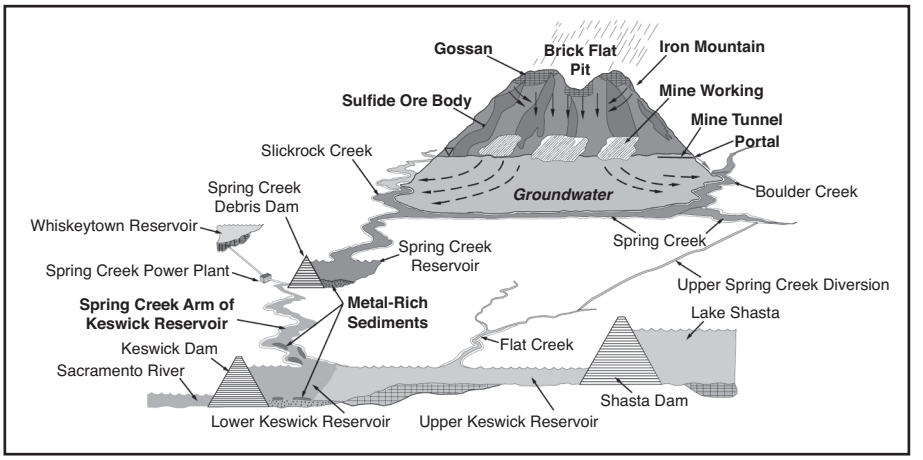
By definition, the conceptual model will be an abstraction that aims to identify those key processes that affect contaminant transport, behavior, and risk. The purpose of the conceptual model is to illustrate and describe a basic understanding of potential sources, media pathways, and possible receptors, as shown in Figure 5.2.

The ASTM standard for development of a conceptual model (E 1689-95) advises the development of (1) a flow diagram that illustrates potential sources, transport mechanisms, exposure pathways and receptors; (2) a schematic block diagram that depicts these site model components; and (3) a map of surface units and other relevant features (ASTM 2008).

5.3.3 Elements of Conceptual Models

Source. At a mine site, the source may be mine and processing waste, such as a tailings impoundment, an area of contaminated soil or sediment from which metals may be released, or the actual mine pit or underground workings.

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Source: INAP 2009.

FIGURE 5.3 Conceptual model showing metal and acid source regions at Iron Mountain and downstream transport pathways to the Sacramento River, California

Exposure pathways. The classic pathways for exposure at a mine site are transport via air (e.g., fugitive dust), groundwater (e.g., contaminated plumes), or surface water (Figure 5.2; e.g., runoff). There are situations at mine sites wherein the pathway is a flow of solid (e.g., waste-rock pile slump) or semisolid (e.g., tailings released from an impoundment) waste materials released from a waste storage facility. In addition to these types of exposure pathways that take the hazard from the source to the receptor, the receptor may actually come to the source for exposure (direct contact). Examples of this include migratory wildfowl landing on contaminated ponds or children playing in contaminated soils; both situations have been observed at historical mine sites.

Receptors. Historically, the primary receptors of concern at mine sites have been humans. This includes people living or working at the mine site, visitors to the site, and people living nearby the site. Additional receptors or endpoints may drive the project manager's response, including aquatic species (e.g., fish and invertebrates), terrestrial wildlife (e.g., invertebrates, birds, and mammals), and floral populations. Receptors may not be present at the time the sampling takes place.

Figure 5.3, a graphic of the conceptual flow pathways at the Iron Mountain mine site, depicts site model components and a map of surface units and other relevant features within the assessment boundary. The most important sources are mine waste, ore, process waste, mine workings, and ore extraction and processing. Water is a primary environmental pathway for constituents released from these sources. Transport occurs by groundwater, surface water, or infiltration through the vadose zone. Because water is a primary pathway, aquatic resources are generally the receptor of most interest.

Uncertainties associated with the conceptual site model need to be identified clearly so that efforts can be taken to reduce these uncertainties to acceptable levels. Early versions of the model, which are usually based on limited or incomplete information, will identify and emphasize the uncertainties that should be addressed.

5.3.4 Developing a Water Balance Conceptual Model

Water balance models are mathematical computation procedures to describe and quantify the spatial and temporal distribution of the water in the forms of precipitation, evapotranspiration,

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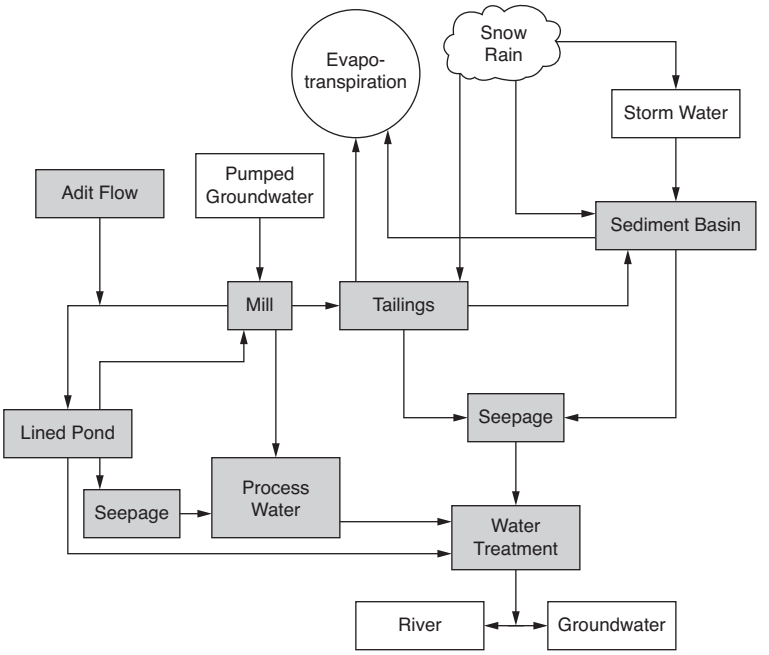


FIGURE 5.4 Flowchart describing water balance as a conceptual model

infiltration, water storage, treatment, seepage, runoff, and discharge (Figure 5.4). The water balance should include surface water and groundwater. It should be robust and provide information on the probable scenarios that might be encountered at the mine site over the full mine cycle.

Development of a site water balance can be facilitated by drafting a conceptual model. The hydrological parameters (e.g., flow or discharge measurements, groundwater flow parameters) and climatological data (e.g., precipitation, temperature, evapotranspiration; see Figure 5.5) information are the key components of a water balance. Mass balance of water sources (inputs) and losses (outputs) need to be identified in the sampling and monitoring plan to quantify each flow pathway (Figures 5.3 and 5.4). Tracers are a useful technique to determine gain or loss of water quantity in streams (see Appendix 3). Seasonal effects are often an important aspect of conceptual models of water balance to examine and quantify. The U.S. Environmental Protection Agency found that a common thread among active Superfund mine sites is that they did not have an accurate water balance (Russell 2006). The Methods for Analyzing Cyanide Needed sidebar provides an example of the importance of water balance.

5.4 CONCEPTUAL MODELS FOR NUMERICAL MODELING

Data availability is one source of uncertainty in the development of models for decision support. To minimize uncertainty, development of a reasonable conceptual model helps to identify data gaps that need to be to populated before using numerical models. Although there are other sources of uncertainty, models should be selected (simple vs. complex) in part based on the data available to support their use. For example, a simple analysis using models like those described as screening procedures could be run quickly at low cost to begin to understand the issues (Mills et al. 1985). This understanding might suggest (perhaps through sensitivity analysis) that data should be collected on current land use, or that a limited monitoring program is warranted. Following acquisition of that data, a revised (perhaps more detailed) model could be developed. This

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Courtesy of Virginia T. McLemore.

FIGURE 5.5 Measuring water infiltration using an infiltrometer, Questa rock piles, Taos County, New Mexico

METHODS FOR ANALYZING CYANIDE NEEDED—BEAL MOUNTAIN MINE

Carol C. Russell, U.S. Environmental Protection Agency

In 1999, the Montana Department of Environmental Quality and the U.S. Forest Service took over the management of water treatment for the bankrupt Beal Mountain mine near Butte, Montana. A trustee was appointed to oversee disbursement of funds, including the bond money for mine water process water treatment. Approximately 1 billion liters of heap-leach pad process solutions have been reduced to 50 million liters with the use of a biological treatment plant and a land application system (W. McCollough, personal communication, June 2003). Although the solutions met the state water-quality standards after treatment for pH and for weak-acid dissociable (WAD) cyanide, all plants within the land application area died within a short time. The operator determined, through greenhouse tests of the water, that the phytotoxicity was due to thiocyanates (B. Parker, personal communication, December 2002). During treatment for metals and pH, many of the breakdown compounds of cyanide, while generally less toxic than the original cyanide, are known to be toxic to aquatic organisms and can persist in the environment for significant periods of time. Some of these potentially toxic breakdown forms include cyanates, ammonia, nitrite, nitrate, and thiocyanates. These compounds, however, were not identified for analysis; only total cyanide or WAD analysis were requested, and thiocyanate is a known interference for the cyanide distillation methods used in the United States. For example, water samples from mining and processing sites where cyanide is used as a process chemical can have WAD and/or total cyanide concentrations that are quite low or undetected, yet when the same samples are analyzed specifically for cyanates and thiocyanates, they show tens of milligrams per liter or more of these compounds (Russell 2006). The lesson learned in this situation is that when treating for acid rock drainage, new compounds can be formed that need to be evaluated for toxicity.

strategy for data-poor situations makes efficient use of resources and targets the effort toward information and models that will reduce the uncertainty as the analysis proceeds. On the other end of the spectrum is the PHREEQC aqueous geochemistry model (Parkhurst and Appelo 2013), a very complex and useful model when sufficient data are available. Frequently, physical parameters are unavailable, making most geochemical models useless. The use of complex mechanistic models is warranted if it helps promote understanding of complex systems and as long as

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WATER QUALITY DATA NEEDS FOR TMDLs

Flow data. Critical to the process of calibrating and verifying models are flow data from sources and various locations in the receiving water. Flow data are generally high in quality if gathered as part of unidirectional stream surveys. The U.S. Geological Survey (USGS) is generally considered to be the reliable source for long-term, high-quality data sets.

Ambient water quality data. A number of federal agencies, state agencies, regional organizations, and research groups collect surface water quality data. Many of these data are retrievable over the Internet, particularly data from USGS and the U.S. Environmental Protection Agency (USEPA). Although there is no universal repository for all surface water quality data, the STORET database is the most comprehensive. Because methods of collection and analysis may vary, there is a need for quality assurance and quality control of these data.

Land use data. All states should have access to a series of land use records and projections. For ease of use, the land use data sets should be made available as geographic information system coverages. USEPA has provided default coverages as a component of its BASINS model. For total maximum daily load (TMDL) purposes, land use data are required for the time period over which water quality data are available in order to calibrate and validate models. Projected land use data are needed for predicting future scenarios. The overall quality of these land use data will vary, often as a function of the level of ground-truthing that was done or the accuracy of the predictions for future land use changes.

Point source data. Model inputs may include measured values of pollutant loading from point sources (e.g., based on information reported on National Pollutant Discharge Elimination System discharge monitoring reports submitted by permitted facilities). Other possible data sources include results from periodic compliance inspection and wasteload allocation studies or data collected as part of field surveys done in support of the TMDL. Such data are generally available and more reliable than data from point sources. This is partly because during high-flow, high-rainfall events, monitoring is only infrequently conducted. Mining processing and storm-water discharges are usually considered to be point sources.

Atmospheric deposition. Data on pollutant loadings from atmospheric deposition have been compiled by the National Atmospheric Deposition Program. Atmospheric nitrate and nitrate from explosives such as ammonium nitrate may be comingled.

Source: Adapted from NRC 2001.

uncertainties in the results are reported and incorporated into decision making. However, there may be a tendency to use complex mechanistic models to conduct water quality assessments in situations with little useful water quality data and/or involving major remediation expenditures or legal actions. In these situations, there is usually a common belief that the realism in the model can compensate for a lack of data, and the complexity of the model gives the impression of credibility. However, given that uncertainty in models is likely to be exacerbated by a lack of data, the recommended strategy is to begin with a simple modeling study and iteratively expand the analysis as needs and new information dictate.

5.4.1 Total Maximum Daily Load Models

Water quality models are used for watershed water quality modeling of contaminant loads and to evaluate the potential for violation of water quality standards or criteria. The data required for total maximum daily load (TMDL) model development is a function of the water criterion or standard and its location and the analytical procedures used to relate the stressors to the

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criterion similar to the needs of a risk-based conceptual model. Data needs can include hydrology (streamflow, precipitation), ambient water quality measures, and land use and elevation. The U.S. Geological Survey has provided excellent methods specifically for mining TMDLs as expanded further in the references (Walton-Day et al. 2002, 2012). TMDL development will also likely require data on point/nonpoint sources and pollutant loads, atmospheric deposition, the effectiveness of current best management practices, and legacy/upstream pollutant sources. Because the amount of available data varies with each site, there is no absolute minimum data requirement that can be universally set for TMDL development.

The data and information required for TMDL modeling must reflect the parameters that affect attainability of water quality standards. Many of the models used today have extremely large data requirements that must be addressed prior to project development. To ensure one has the proper data inputs, the modeler should be involved in the planning process for data collection. See the Water Quality Data Needs for TMDLs sidebar for a discussion of such data inputs.

5.5 SUMMARY

A well-designed and implemented program has the potential to deliver real value to mining companies and the public, both in terms of better environmental and financial decision making, and in the effectiveness and public credibility of the project for stockholders and stakeholders.

5.6 REFERENCES

ASTM E 1689-95 2008. *Standard Guide for Developing Conceptual Site Models for Contaminated Sites*. West Conshohocken, PA: ASTM International. www.astm.org/Standards/E1689.htm. Accessed 9/11/13.

Ford, K.L. 2004. *Risk Management Criteria for Metals at BLM Mining Sites*. Technical Note 390 (revised) BLM/RS/ST-97/001+1703. Denver, CO: U.S. Bureau of Land Management, National Science and Technology Center. www.blm.gov/nstc/library/pdf/TN390v04.pdf. Accessed 9/11/13.

Funtowicz, S., and Ravetz, J.R. 1991. A new scientific methodology for global environmental issues. In *Ecological Economics: The Science and Management of Sustainability*. Edited by R. Costanza. New York: Columbia University Press. pp. 137–152.

INAP (International Network for Acid Prevention). 2009. *Global Acid Rock Drainage Guide* (GARD Guide) www.gardguide.com. Accessed 9/11/13.

MERAG (Metals Environmental Risk Assessment Guidance). 2007. *Uncertainty Analysis*. MERAG Fact Sheet 07. www.icmm.com/document/259. Accessed 9/11/13.

Mills, W.B., Porcella, D.B., Ungs, M.J., Gherini, S.A., Summers, K.V., Mok, L., Rupp, G.L., Bowie, G.L., and Haith, D.A. 1985. *Water Quality Assessment: A Screening Procedure for Toxic and Conventional Pollutants in Surface and Groundwater* (Revised 1985). Part 1 EPA/600/6-85/002a. U.S. Environmental Protection Agency, Environmental Research Laboratory. http://yosemite.epa.gov/water/owrccatalog.nsf/e673c95b11602f2385256ae1007279fe/1d843503b32cc14485256b060072591c!OpenDocument. Accessed 9/11/13.

NRC (National Research Council). 2001. *Assessing the TMDL Approach to Water Quality Management*. Washington, DC: National Academies Press.

Parkhurst, D.L., and Appelo, C.A.J. 2013. *Description of Input and Examples for PHREEQC Version 3—A Computer Program for Speciation, Batch-Reaction, One-Dimensional Transport, and Inverse Geochemical Calculations*. U.S. Geological Survey, Techniques and Methods, Book 6. http://pubs.usgs.gov/tm/06/a43/pdf/tm6-A43.pdf. Accessed 9/11/13.

Russell, C. 2006. Mine drainage challenges from A to Z in the United States. In *Proceedings, 7th International Conference on Acid Rock Drainage (ICARD 7)*. Lexington, KY: American Society of Mining and Reclamation. pp. 1785–1802, www.imwa.info/docs/imwa_2006/1785-Russell-CO.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2006. *Guidance on Systematic Planning Using the Data Quality Objectives Process EPA QA/G-4*. EPA/240/B -06/001. Washington, DC: Office of Environmental Information. www.epa.gov/QUALITY/qs-docs/g4-final.pdf. Accessed 9/11/13.

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Walton-Day, K., Kimball, B.A., and Runkel, R.I. 2002. The use of mass-loading studies and solute transport modeling to assist in the development of TMDLs for streams affected by mine drainage. In Workshop Notebook, Colorado Department of Health and Environment 10th National Nonpoint Source Monitoring Workshop. Colorado Department of Public Health and Environment.

Walton-Day, K., Runkel, R.L., and Kimball, B.A. 2012. Using spatially detailed water-quality data and solute-transport modeling to support total maximum daily load development. *J. Am. Water Resour. Assn*. 48(5):949–969.

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CHAPTER 6

Sampling and Monitoring Program Implementation

Carol C. Russell, Kathleen S. Smith, and Virginia T. McLemore

6.1 INTRODUCTION

Implementation of a sampling and monitoring program begins with a good SMP (sampling and monitoring plan). SMPs are written documents that outline procedures for obtaining data of adequate quality to ensure that the sample, analytical data, and scientific interpretations are sufficient to meet the goals and objectives of the program. The SMP (e.g., Table 6.1) should include the project management information/objectives, data acquisition (sampling methods, measurement and analysis procedures, data collection, or generation methods), data handling/analysis and data validation/usability. Several examples of sampling plans and a U.S. Environmental Protection Agency (USEPA) checklist on quality assurance (QA) document review can be found in Appendix 4. The field and analytical methods are often referred to as standard operating procedures (SOPs), many of which are briefly described in Appendix 3 (USEPA 2007). Another document used in sampling is the quality assurance project plan (QAPP), which describes the policy, organizational, and functional activities necessary to collect data that will stand up to legal and scientific

TABLE 6.1 Sampling and monitoring plan outline

Project Management	Project Organization/Responsibilities
	Problem Identification
	Data Quality Objectives
	Training Requirements
	Documentation
Data Acquisition and Management	Sampling Design and Methods of Collection
	Sample Handling and Custody
	Analytical Methods
	Quality Control Requirements
	Instrument/Equipment Testing, Inspection, and Maintenance
	Data Management
Data Validation	Validation and Verification Methods
	Data Review, Validation, and Verification
	Correspondence with Data Quality Objectives

Source: Adapted from USEPA 2013.

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scrutiny (USEPA 2001). For our purposes, the distinctions between these documents will not be emphasized, but the need for documentation of the concepts described is the overriding issue. An example checklist is provided in Appendix 4.

6.2 SAMPLING AND MONITORING PLAN

The SMP is formal documentation of what will be measured, at what times, for what media, in what manner, and by whom based on the planning done as described in Chapter 5. At this stage, the questions and objectives have been defined, the conceptual model has been developed, the types of media and protocols have been chosen, the confidence levels with appropriate quality assurance and quality control (QA/QC; USEPA 2002a, 2002b) recognized, and the budget established. The following sections are suggested topics to consider in addition to the SMP outline. They will explore major concepts or headings with potential action items to fulfill each need.

6.2.1 Management, Leadership, and Organization

The project manager's responsibility is to the team and the program. He or she functions as the ultimate field manager to get useful data as planned in the first phases of the program. The manager is the one who ensures that the details have been addressed. For example, one key detail is that the monitoring locations are accurately identified, are safe for access, and that right of access has been secured. Permission for access to obtain samples is often facilitated by the offer to share results with the owner of the property being sampled. Other management tasks are QA/QC including chain-of-custody documentation, identification of project schedule, resources, milestones, and identification of any applicable safety requirements such as steel-toed boots or Mine Safety and Health Administration certification.

Leadership is also critical to provide team cohesiveness to meet the monitoring goals and to ensure that the sampling plan is implemented properly. The leader or project manager should be the one to officially sign off and date the SMP. Not only does the leader need to know the plan intimately but also must convey the purpose and all information to the team. One way to document the roles and responsibilities of the team is an organization chart with assigned duties. The sampling crew may be working under long and physically trying conditions. The leader is the one to observe the individuals and to call the end of the day when appropriate. The leader is also the one to facilitate positive working relationships among the team members. Coordination, communication pathways, and a process for resolving disputes in the SMP makes for a better team at the end of a trying day. The SMP should allow for unexpected delays, such as training of personnel, changes in personnel, ordering and shipping of materials, equipment malfunctions, and adverse weather conditions. In a similar fashion, the responsibility of each team member is to speak up if something is not going right.

Ultimately, the leader must have identified who the decision maker will be during each step of the SMP. For example, there is a decision maker who will be the user of the data and information developed as part of this program. Is that person the agency manager who determines if a permit is issued? Is it the new mine manager who will need to design and implement mitigation measures? Or is it the monitoring program manager who will provide information to the end user of the data? Development of the process for reporting results to the end users or decision makers, including the timing, format, and other expectations, should be anticipated before going in the field.

The leader should also have a communication strategy for proving information to stakeholders. Having a resident come up to the team and ask what you are doing requires forethought from the team member as to an appropriate response. Stakeholders are parties who may be affected by

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the results of the study and/or persons who may later use the data resulting from the data quality objective process (USEPA 2006). It is suggested that the manager develop a table listing the impacted organizations/stakeholders and the individuals who are representing those organizations. The table should also include how to contact them in order for the sampling team to know how to respond or to whom questions should be directed.

6.2.2 Team Qualifications and Training

The role of the team and the team members is to collect the samples needed from the locations identified in the proscribed manner and to document that they collected them in the way required. In order for any sampling and monitoring program to be implemented, a qualified team must be assembled and trained. The team should be aware of their surroundings and any observation that might affect the implementation of the SMP. Every trip to the field should include a review of expectations, logistics, and standard (and not so standard) procedures to be used for this sampling effort. Personnel conducting field sampling and monitoring need to have the appropriate qualifications for the field work as listed in the sampling plan, including physical capability, training, formal education, experience, and a satisfactory knowledge of the requirements of the activities to be carried out. Of special importance is health and safety training and that communication and documentation systems are operable, robust, and reliable.

Training should be sufficient to ensure that the staff can perform their duties competently and safely. The most important elements of training are about including the team members (those who are collecting the data in the field or in the laboratory) in the monitoring effort in a way that shares the understanding of the objectives, promotes personal responsibility for the reliability and usefulness of the data they generate, and creates a sense of ownership and participation of/in an important process. The whole team should review the SMP (each member should have his or her own copy), protocols, a health and safety plan (specifics should be reviewed the morning of sampling), and the roles and responsibilities of each team member.

Systematic training and SOPs improve the reliability and comparability of the data collected. For example, a technical protocol can be written for instructing the user how to perform a specific analytical method to be followed in the laboratory or field (such as field testing using a pH meter), how to collect a sample in order to preserve the sample integrity and representativeness (such as collection of samples for future analysis of volatile organic compounds or trace metals), or how to conduct a bioassessment of a freshwater site.

Another part of the team is the laboratory. It is good practice to maintain effective communication with the laboratory and analysts. Additional information about samples that might be helpful to the laboratory staff includes sample type, sample description, preservation method(s), a complete list of analytes and requested analytical methods, a list of possible interferences, the date delivered to the laboratory, and contact information for a knowledgeable person who can provide additional information about the samples and sample-collection methods. Selected sample collection methods are briefly described in Appendix 3.

6.2.3 Define Field Operating Procedures—Protocols/Standard Operating Procedures

Protocols or SOPS are written procedures or instructions of practices and methods used in sampling, monitoring, analytical techniques, and data management (USEPA 2007). Standard protocols and SOPs are important because they provide scientists, managers, stakeholders, and other parties involved with the project, including newcomers, with technical information on how particular jobs are performed. The SOPs can be written by a variety of entities, but ultimately they

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must be incorporated into the sampling plan and approved by those signing off on the plan. This step is very important because standard protocols may not be appropriate or adequate in all situations. Therefore, protocols should be available to the team prior to sampling and also available in the field for consultation if questions arise. Protocols include methods of sampling, sample collection, sample preservation, interference mitigation, sample preparation, sample decomposition, sample analysis, sample storage/archiving/disposal, data analysis and interpretation, and data management/archiving. Standard protocols improve communication between scientists and other parties and achieve common goals. Discussion of some of these methods can be found in Appendix 3. The term *standard operating procedure* is not always appropriate and the term *protocol* can generally be used. Protocols are typically media specific and often are formalized as SOPs. It is always important to document any deviation from these SOPs. Topics that need to be covered in the SOPs would be sampling procedures, including sample containers, volume, interference mitigation, preservation, cleaning and decontamination of equipment and containers, field equipment calibration, maintenance, testing, inspection, and field documentation. Procedures should also be covered on how to perform the task, wherein each step is detailed. Flowcharts are especially helpful. USEPA has a guidance document for preparing SOPs (USEPA 2007) and the Dairy Farm Business (2001) has an excellent guide to writing SOPs.

6.2.3.1 Field Data Collection Information

Specific information needs to be documented for each sample when it is collected in the field. This information includes identification number, county and state, date and time of sampling, name of person collecting sample, sample media, sample type (e.g., composite, grab), sample description, sampling method, sampling equipment, sampling location (e.g., Global Positioning System [GPS], Universal Transverse Mercator or mine coordinates, latitude/longitude from a map and a photo), sample location on study site, depth or elevation at collection site, purpose of collecting sample, observational field information (e.g., temperature, weather conditions, site-specific information, collection problems), field measurements, constituents to be determined, instrument calibration information, and preservation methods (USEPA 2008). Additional site description information could include mining district, mine name, commodities recovered, geochemical overview (main geochemical issues at the site including known data, previous spill of process tailings facility, and discharge of seeps), description of catchment basin and size, landscape/topography, land use, predominant bedrock lithology, vegetation, photos of landscape and site, and a hand-drawn map of the site. It can be quite useful to compile meteorological information (e.g., temperature minimum/maximum, precipitation, and snowfall) at the time of sample collection and to retain that information with the analytical information. Photographs are always useful to document sampling sites and conditions for future consideration. Appendix 5 includes descriptions of field data collected for specific sampling and monitoring case studies.

6.2.3.2 Field Equipment

Field equipment should be adequate for the tasks, and maintained and calibrated to provide accurate measurements. To keep track of each piece of equipment, a unique identification number can be linked to manufacturer maintenance and calibration protocols, and operational instructions. The measurement equipment should be calibrated before being put into service and thereafter according to an established procedure, including maintaining records of all equipment calibrations and maintenance (USEPA 2013).

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6.3 SAMPLING DESIGN AND THE SAMPLING PLAN DOCUMENT

The sampling design should always be focused on what data are needed to answer questions and associated data quality objectives. Chapter 3 expands on the special considerations for development of the sampling plan design for mining and processing sites. A useful reference is USEPA Method QA/G-5S, *Guidance on Choosing a Sampling Design for Environmental Data Collection for Use in Developing a Quality Assurance Project Plan* (USEPA 2002c). Sampling locations and the rationales for the selection should be targeted and recorded on a map and table within the sampling plan. A study area reconnaissance is always advised. The spatial boundaries identified for the investigation should also be delineated on the map. Timing of sampling at each location should be considered and specifically programmed if it could influence the objectives and the reliability of the data.

For example, in one SMP for a mining site, sample locations were selected based on the results of previous sampling efforts at this site and existing data needs in order to maintain data comparability. The purpose was to assess current surface water and sediment conditions and to further determine the nature and extent of the environmental impacts. The data collected were to be used to evaluate the toxicological impacts and provide an ecological risk assessment.

Site locations are often selected to generate a background or reference data set, to determine source contribution to existing levels, and to evaluate potential contributions from other sources (i.e., tributaries). Additional sampling sites may be listed in the sampling plan to be added in the field based on observed conditions. (See Sampling and Analysis of Mountain Waters sidebar.)

To address sampling parameters, the following data might be collected for a typical surface water sampling event for a mine in operation:

- Field water-quality parameters—pH, conductivity, dissolved oxygen, temperature, GPS locations, and photos (as needed)
- Volumetric stream flows—using flowmeters, flumes (where necessary), and observation of stream flow measurement from fixed instrumentation
- Representative surface water samples—including streams, seeps, and all discharges for dissolved metals and total metals
- Sediment—total metals and total mercury
- Macroinvertebrates—assemblage (species identification and count)

For example, based on the conceptual model of the previously mentioned hypothetical operating mine and past sampling verification in the same area, the contaminants and sources that potentially pose a risk to ecological receptors, based on site-specific water quality standards and the watershed pathways, require further characterization. The primary analytes of concern identified for the site are aluminum, cadmium, calcium, copper, lead, manganese, nickel, silver, and zinc. However, the full suite of metals will be analyzed because additional potential contaminants may exist and the additional incremental cost for the complete suite is negligible. The process for modeling ecological risks was acknowledged and the appropriate parameters for each ecological receptor identified. No statistical or quantitative decision rule could be established prior to sampling. When the data are analyzed, if the calculated hazard quotient does not exceed one for acute toxicity, there is no ecologically significant effect observable in direct studies of community structure/function compared to reference, and site-specific toxicity tests do not show significant mortality effects compared to reference and laboratory control, then remedial actions are not likely to be necessary. Tolerable limits on decision errors, which are used to establish performance

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SAMPLING AND ANALYSIS OF MOUNTAIN WATERS—CHALK CREEK

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Chalk Creek in central Colorado drains an area of approximately 250 km^2 (24,864 ha), including the primary source of acid rock drainage (ARD), the Mary Murphy mine. Lode mining developed on the valley slopes producing from gold, silver, and lead-zinc deposits through an extensive system of underground workings on numerous levels along sulfide veins. The Golf Tunnel was constructed to transport ore to the mill and to provide drainage to the 670-m (2,198-ft) (vertical) mine workings of the Mary Murphy mine. Work by the Colorado Division of Minerals and Geology (CDMG), their consultant, and the U.S. Environmental Protection Agency involved the characterization of the ARD groundwater flow systems. Multiple dyes were introduced into the fractured bedrock mountain/mine pool system. Results from the tracing studies indicate four discrete groundwater flow systems: (1) a relatively slow-flow system in the high mountain faulted bedrock very high in zinc; (2) rapid flow through the cavern-type systems of the interconnected mine voids and fractures lower in the mountain; (3) a weathered, shallow fracture system in the upper 8 m (26 ft) of bedrock; and (4) infiltration of precipitation through the tailings repository into the near surface system, also very high in zinc (INSTAAR n.d.). After examining one of these adits, mining specialists from CDMG found that ARD was draining from only a few fractures in the rock. In fact, most of the mine drainage originated from the very back of the Golf Tunnel from a shaft drilled vertically to intercept the mine workings some 245 m (804 ft) above. Very little ARD was draining into the tunnel except from this site, even though many other sulfide vein systems were mined. Mine inflows were segregated into clean water that could be piped out directly without treatment and the one ARD site, which can be readily collected for passive treatment. The major lesson learned is that if uncontaminated water can be intercepted and diverted before it enters mineralized areas of mine workings and becomes ARD, the total loadings of metals will be significantly diminished (Russell 2006).

goals for the data-collection design, need to be established. For this example, the number of samples and station locations are based largely on past investigations. Some sampling locations had been eliminated based on evaluation of their contribution to the data set as a whole.

6.4 CONDUCT THE PILOT STUDY

Conducting a pilot study, pre-study, screening test, feasibility study, or orientation survey consists of a series of preliminary experiments prior to conducting the final plan to work out any difficulties or problems that could arise, in order to improve quality and efficiency. A pilot study or the first step of a phased monitoring program can reveal deficiencies or problems with the procedures or design of a plan before time and resources are expended on large-scale plans. Using a phased approach is suitable for sampling to get a preliminary understanding of the problem, variability in the data and types of materials (waters, sediments rocks), and then allows subsequent phases to build on early phases with the goal of reducing uncertainty filling data gaps and conducting more sampling of those areas with the greatest uncertainty and variability. A pilot study can also address logistical issues, such as evaluating if technicians have the proper skills and training, if the equipment operates correctly under field conditions, if QA/QC procedures are adequate, if statistical applications are sufficient, and identifying any other difficulties under field conditions. A pilot study allows for testing of the hypotheses based on the conceptual model and can lead to

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testing additional hypotheses. After conducting the pilot survey, it is important to consider what worked and modify the plan accordingly, including the protocols, if necessary.

For another sampling effort, the following text describes an example of an operating mineral processing facility. The conceptual model and past monitoring identified plumes of impacted groundwater. The model is being used to analyze and compile new data and generate three-dimensional visualizations of the aquifers and associated plumes. The conceptual model and data collection will be continuously updated until the plume has been fully delineated horizontally and vertically and all data gaps have been filled. The sampling plan consists of borehole screening sampling of groundwater. If possible, concentrations of target analytes (e.g., arsenic and trichloroethylene) in groundwater during screening-level sampling will be determined by an on-site mobile laboratory. Project screening levels estimated from the preliminary human health risk assessment would be used to delineate the plume vertically and horizontally. The screening samples would also be compared to regional action levels. The results of the screening-level analysis will be used to determine offset boring locations and well installation locations. The borehole screening samples to be collected and analyzed would be used for field decisions and well location selection, and these screening results will possibly be used as additional information for the final risk assessment.

6.5 IMPLEMENT THE SAMPLING AND MONITORING PLAN (PROGRAM IMPLEMENTATION)

Three basic steps are involved in implementing the SMP (program implementation): sampling, analysis, and interpretation. These appear to be independent, but in reality, they are interdependent functions. If problems are encountered at the sampling step, all efforts in the succeeding steps may fail. The sampling stage is probably the most important and most difficult to correct once the samples are collected. Therefore, it is imperative that the initial stages previously discussed are well thought out and implemented before samples are collected. Field personnel must adhere to all appropriate protocols.

Implementation of health and safety procedures while sampling is of utmost importance. Knowing the hazards and actions to take if something goes wrong should be identified in the planning stage and carried out in the implementation phase. For instance, sampling with a partner rather than solo sampling has saved numerous lives. Also, knowing the location of the nearest hospital or emergency services can save time as well as lives.

6.6 SUMMARY

Implementation of the SMP consists of tasks for management and leadership of the sampling effort, assembly and training of the monitoring team, identification of field data collection information, including the sampling design, protocols and SOPs, and conducting the pilot and full-scale study. Additional items that could be included in an SMP include a formal statement of the purpose of the sampling, a formal statement of assumptions, a list of the parties responsible for implementing each step of the plan, a signed confirmation of participants and their responsibilities in the plan, a formal health and safety plan, a schedule of activities, a description of the QA/QC procedures, and documentation of arrangements made for access to sampling locations.

6.7 REFERENCES

Dairy Farm Business. 2001. *Standard Operating Procedures: A Writing Guide*. Penn State, College of Agricultural Sciences, Agricultural Research and Cooperative Extension. http://centaur.vri.cz/news/prilohy/pril771.pdf. Accessed 9/11/13.

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INSTAAR (Institute of Arctic and Alpine Research). n.d. *Mary Murphy Mine—Characterizing Acid Mine Drainage Flowpaths and Residence Times Using Isotope Tracers*. http://snobear.colorado.edu/Daniel/mineresearch/mmm.html. Accessed 9/11/13.

Russell, C. 2006. Mine drainage challenges from A to Z in the United States. In *Proceedings, 7th International Conference on Acid Rock Drainage (ICARD 7)*. Lexington, KY: American Society of Mining and Reclamation. pp. 1785–1802. www.imwa.info/docs/imwa_2006/1785-Russell-CO.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2001. *EPA Requirements for Quality Assurance Project Plans (EPA QA/R-5)*. U.S. Environmental Protection Agency, EPA/240/B-01/003. www.epa.gov/quality/qs-docs/r5-final.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2002a. *Guidance for Developing Quality Systems for Environmental Programs (EPA QA/G-1)*. U.S. Environmental Protection Agency. EPA/240/R-02/008. www.epa.gov/quality/qs-docs/g1-final.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2002b. *Overview of the EPA Quality System for Environmental Data and Technology*. U.S. Environmental Protection Agency. EPA/240/R-02/003. www.epa.gov/quality/qs-docs/overview-final.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2002c. *Guidance on Choosing a Sampling Design for Environmental Data Collection for Use in Developing a Quality Assurance Project Plan (EPA QA/G-5S)*. U.S. Environmental Protection Agency. EPA/240/R-02/005. www.epa.gov/quality/qs-docs/g5s-final.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2006. *Guidance on Systematic Planning Using the Data Quality Objectives Process (EPA QA/G-4)*. U.S. Environmental Protection Agency. EPA/240/B-06/001. www.epa.gov/quality/qs-docs/g4-final.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2007. *Guidance for Preparing Standard Operating Procedures (SOPs) (EPA QA/G-6)*. U.S. Environmental Protection Agency. EPA/600/B-07/001. www.epa.gov/quality/qs-docs/g6-final.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2008. *Field Measurement Uncertainty*. U.S. Environmental Protection Agency. SESDPROC-014-R0. www.epa.gov/region4/sesd/fbqstp/Field-Measurement-Uncertainty.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2013. *Field Operations Group, Operational Guidelines for Field Activities*. EPA report. www.epa.gov/region8/qa/FieldOperationsGroupOperationalGuidelinesForFieldActivities.pdf. Accessed 9/11/13.

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CHAPTER 7

Data Management, Assessment, and Analysis for Decision Making

Carol C. Russell, Kathleen S. Smith, and Virginia T. McLemore

7.1 INTRODUCTION

The aim of a sampling and monitoring program is to inform the decision makers about minimizing and mitigating the potential harm, and to enhance the potential benefits of the project based on scientific data. Good quality data managed properly serve as the core to all subsequent design, technical analysis, and responses and need to be conducted under proper quality assurance procedures, well organized, and available for analysis. This chapter describes steps to ensure that data are managed appropriately and provides basic data analysis strategies. Specifically, monitoring helps to determine and implement timely remedial measures to ensure that environmental impacts are prevented or minimized. In addition, monitoring can measure if actions taken have achieved the desired results and that resources have been utilized appropriately, efficiently, and effectively. For monitoring to achieve these expectations, the data must be analyzed, assessed against a goal, managed, and presented in such as way as to inform the decision-making process. Decisions are made not just on the information provided but on other factors regarding values, relationships, and the integrity of the process.

7.2 DATA MANAGEMENT

Data and information management is a process that starts with project design and can extend well beyond the data analysis and information dissemination phases. Management, interpretation, and presentation are highlighted to convey that data have little intrinsic information and must undergo processing to be transformed into information. Data are like letters of the alphabet: taken individually, they reveal very little; put together with a little thought and organization, however, those same letters can tell a complete story.

The amount of data and information collected at mine sites is extensive, thus requiring the need for effective data management to enable the mining company as well as federal, state, and local agencies and other stakeholders to make informed decisions. To make the best use of baseline data collected during the exploration phase, the data need to be available to mine planners and metallurgists, and also need to be communicated to the wastewater treatment managers. Requirements for data management and sharing need to be identified before sampling and monitoring begins.

Data handling and the use of databases is a major undertaking throughout a monitoring program. Six basic components of data management are

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1. Project design and planning,
2. Data management (paper and digital),
3. Capture of metadata,
4. Data archiving,
5. Data manipulation (statistics), and
6. Presentation of information.

Efforts are being made to develop data storage systems that facilitate data exchange among different groups that conduct sampling and monitoring.

A *database* is a system where data are stored, whereas a *relational database* is a database where the data are organized into similar entities that are related to one another by a common, unique identification number (called a primary key). Relational databases facilitate effective management of a large amount of investigation data by eliminating duplication and maintaining consistency of the data. Databases can be developed for a specific resource (e.g., surface water) or as a general clearinghouse of information. One example of a broad database is the U.S. Environmental Protection Agency's (USEPA's) national water and biological data storage and retrieval system, called STORET. A specific set of quality control measures (metadata) is required for any data entered into the system. For more information, see Section 7.8 (USEPA 2011). Databases are best if they are designed or modeled before or during the first stage of the sampling and monitoring program. The typical mining-related data to be incorporated into a database or a set of relational databases include locational data, mineralogy, chemical analyses, geologic data on drillholes and test pits, historical and recent photographs, and other data. It may be useful to coordinate mining and environmental data into one relational database. One of the most effective tools to enter, store, report, and utilize these data are by relational databases such as SQL Server, Access, FoxPro (all by Microsoft, Redmond, Washington); Paradox (Corel, Ottawa, Ontario); GIS (geographic information system) databases; or on other servers (McLemore et al. 2004). Relationships among data are stored in the database along with the data and the definitions of the data, called metadata.

Data can be stored, processed, and retrieved using four major components to transfer and exchange data: (1) database management systems, (2) word processing software, (3) computer-aided drafting or CAD software, and (4) GIS software. Computer technology is such that all of these techniques can be connected or linked together. Within a database management system, the tables can be linked to each other, where appropriate, and a main table can provide the organizational structure to other linked tables. The database is linked to additional data in other file formats outside of the database, such as photographs, reports, laboratory scans, and so on. Database management systems query and manipulate tabular data for further calculation, analysis, reporting, or exchange. Some databases can be designed to display statistical data and graphs automatically.

Geographic or locational data includes both surveyed points and GPS (Global Positioning System) data. CAD software applies computer graphics to manual drafting techniques and includes design, drafting, and display of graphically oriented data. GIS software captures, manages, manipulates, analyzes, models, and displays spatial (locational) data. Once the data are entered into appropriate databases with locations, the data can easily be converted into GIS format for displaying on maps and performing geospatial analyses.

The fields used in the database must be fully explained and documented, which is referred to as metadata. Metadata documents need to contain documentation on methods used, sources of data, data quality, and any pertinent information that documents the data collected, such as

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special handling procedures (see Metadata sidebar). It is important that staff is assigned to input and maintain the database throughout the project.

Everything needs to be documented, from field studies to modeling runs. Computers are ideal to electronically capture, store, distribute, annotate, display, and disseminate the extensive data collected at mine sites. Some mining and consulting companies have developed sophisticated computer programs to handle these data. Other companies use commercially available software. Capability with multiple interrelated systems needs to be considered. Evaluating data by simply inputting it into specially designed software is not a recommended method because the oversight of a competent person is invaluable. Computers are merely tools to aid in evaluation and interpretation of the data.

The most difficult task is maintaining quality control (QC) of the data being entered into the database and the integrity of the database itself. QC for data management is a system of procedures, checks, audits, and corrective actions to ensure that data are collected, stored, and analyzed in an acceptable manner, and that technical aspects and reporting are adequate.

7.3 ANALYSIS AND ASSESSMENT OF DATA

The term *scientific assessment* means interpretation of data based on scientific or technical knowledge from multiple factual inputs, data, models, assumptions, and/or professional judgment to bridge uncertainties in the available information. Different types of assessments are performed based on the objectives of the sampling and monitoring effort. The four general types of assessments include

1. Condition assessments to detect chemical, physical, and biological status;
2. Causal pathway assessments to determine causes and identify their sources;
3. Predictive assessments to estimate environmental, economic, and societal risks and benefits associated with a range of possible management actions (Cormier and Suter 2008); and
4. Outcome assessments to evaluate the results of the decisions made.

Condition assessments ask, Is there a problem? Causal pathway assessments ask, What caused the problem? Predictive assessments ask, What will be the consequences of taking a particular action? Outcome assessments ask, Did the solution work?

In a similar fashion, an environmental assessment undertaken for a mining operation would help to support decision making to protect the environment. The assessment puts a value on something to determine its positive or negative value. At a mining site, assessments of impacts are either predictive, as is the case of environmental impact assessments or after-the-fact assessment of damage, normally called a site assessment. A good reference for abandoned mine assessment is USEPA's *Abandoned Mine Site Characterization and Cleanup Handbook* (USEPA 2000a).

The assessment is broken down into human or environmental risk assessments, as discussed in Chapter 4. Another gauge of impact might be the exceedance of a standard such as a drinking water standard established in the Safe Drinking Water Act or the exceedance of a screening level for soil cleanup utilized under Superfund.

7.3.1 Evaluate Laboratory Results

Quality decisions are based on quality information derived from quality samples (data). However, it is often difficult to obtain quality and reproducible samples. The best "tool" in the production of quality samples is to systematically avoid errors at the beginning rather than trying to identify

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METADATA

The following data qualifiers are the minimum information about a data point that is necessary to ensure consideration for standards and classifications proceedings.

A. **Project description:** Reasons why data was collected.

B. **Data source:** Who supplied the information or from what database was information taken?

C. **Waterbody type:** Type of waterbody sampled (stream, lake, reservoir, wetland, ditch, drain, etc.).

D. **Location:** A geographical location must be supplied with the data. The location description must contain enough information for the sample point to be located on a 1:24,000 quadrangle map. The sample location would best be described by latitude and longitude to at least the nearest second. When using a geopositioning device (e.g., GPS), the geopositioning method and horizontal datum must be noted. Other location descriptors include state, county, and hydrologic unit code.

E. **Date and time:** The date and time of sample collection should be noted. On time-composite samples, the beginning and ending times should be supplied.

F. **Characteristic measured:** This would include the name of the characteristic and its sample fraction (e.g., ammonia as nitrogen, dissolved iron, suspended solids).

G. **Result value measured:** This includes the quantitative or numeric result measured. This could also be a qualitative result such as good, fair, or poor. The quantitative value must have reporting units in metric (International System of Units).

H. **Quantification and/or detection limit:** This is the quantification limit (QL) and/or method detection limit (MDL) as reported by the laboratory for each analysis. The MDL is the smallest amount of a substance that can be detected, although not necessarily quantified, by a particular method. The QL is the smallest amount of a given substance that can be detected and quantified at a level that is statistically significant. The QL is sometimes referred to as the practical quantification limit, or PQL. The PQL is approximately 3 to 10 times the MDL but is typically defined as a quantity that is 5 times the MDL.

I. **Method techniques:** This is information about whether the sample was grab or composite. If composite, additional descriptors such as time, flow, width, depth, or depth weighted should be noted. Instrumentation is used to collect and analyze the sample. Information on handling and transportation of the sample is also required.

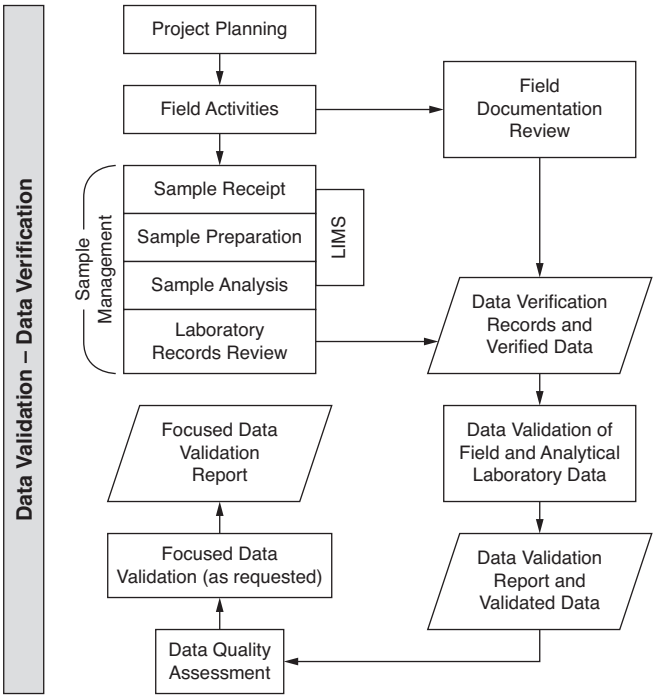
J. **Who measured:** Information should be supplied as to who physically collected the sample and who did laboratory and field analyses. This can be by agency, company, laboratory, or individual.

K. **Lake/Reservoir information:** Most lake and reservoir monitoring plans include collecting depth profiles for temperature, dissolved oxygen, pH, and specific conductance. Complete profile information should be included. Results for each meter depth should be provided for each sampling date. Depth information for water quality samples should include upper and lower depths for composite or integrated water column samples, or as the actual depth for discrete depth grab samples.

L. **Other:** Although not required, it is suggested that quality descriptors such as method precision and bias be included with value or data set. Also, inclusion of reference and method number for the analytical method would add greater specificity as to the chemical form being measured.

Source: Adapted from USEPA 2013

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LIMS = Laboratory Information Management Systems.

Source: USEPA 2002.

FIGURE 7.1 Data verification and data validation components in the project life cycle

problems with the data after the fact. Figure 7.1 illustrates steps in the verification and validation of data.

Nevertheless, laboratory results can be evaluated in a number of ways to determine if the data are useable (Weight and Sonderegger 2001). Tools can be used to minimize uncertainties and maximize confidence in decision making. Comparison between field and laboratory measurements, ionic balances, spikes, sample splits, and additional laboratory results are common indications of precision and also improve QC. However, these approaches do not guarantee accuracy, and, ultimately, the analyses must make geologic sense and compare with the known mineralogy and geologic setting. Different laboratories may use the same analytical methods on the samples, but the results could still be significantly different. Laboratory results should be compared with field data collected, which can identify potential problems of sample stability, preservation methods, poor equipment, or poor technique. Field results may be temperature dependent, so it is important to record the temperature of the sample in the field, especially for water samples. In the laboratory, samples must be analyzed at room temperature, as volumetric glassware is calibrated at room temperature.

Determining ionic balances is another method of evaluating laboratory results for water quality samples. Typically, analyses of water samples include major anions and cations. The sum of the milliequivalents per liter of the major anions should equal the sum of the milliequivalents per liter of the major cations. Solid samples should include major components: SiO_2 (silicon dioxide), TiO_2 (titanium dioxide), Al_2O_3 (aluminum oxide), CaO (calcium oxide), MgO (magnesium oxide), MnO (manganese (II) oxide), K_2O (potassium oxide), Na_2O (sodium oxide), P_2O_5

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(phosphorus pentoxide), and loss on ignition or LOI. The sum of the major components should total 100%. The sum of anions will not always equal the cations and the sum of major oxides will not always equal 100%, and the acceptable deviations must be agreed upon.

Samples can be spiked in the field or in the laboratory to check the accuracy of analytical measurements. A *spike* is a controlled addition to a sample of any media with a known amount of the analyte(s) to be determined.

Sample replicates and splits are recognized and highly recommended methods of determining the variability of the results. In the field, sample replicates include duplicate samples collected at the same site but given different sample numbers. Large samples collected at the site can be homogenized and split to obtain additional samples to test for precision and variability. During the sample preparation, sample splits are critical in determining if the preparation methods are appropriate. During the analysis, sample splits are regularly performed to check procedures and proper functioning of the instrument.

It is good practice to send splits of the same samples to different laboratories for comparison and to include certified samples (with similar sample numbers as the field samples) with the spikes. Major elements should agree within ±5%, and trace elements should agree within ±10% (Weight and Sonderegger 2001).

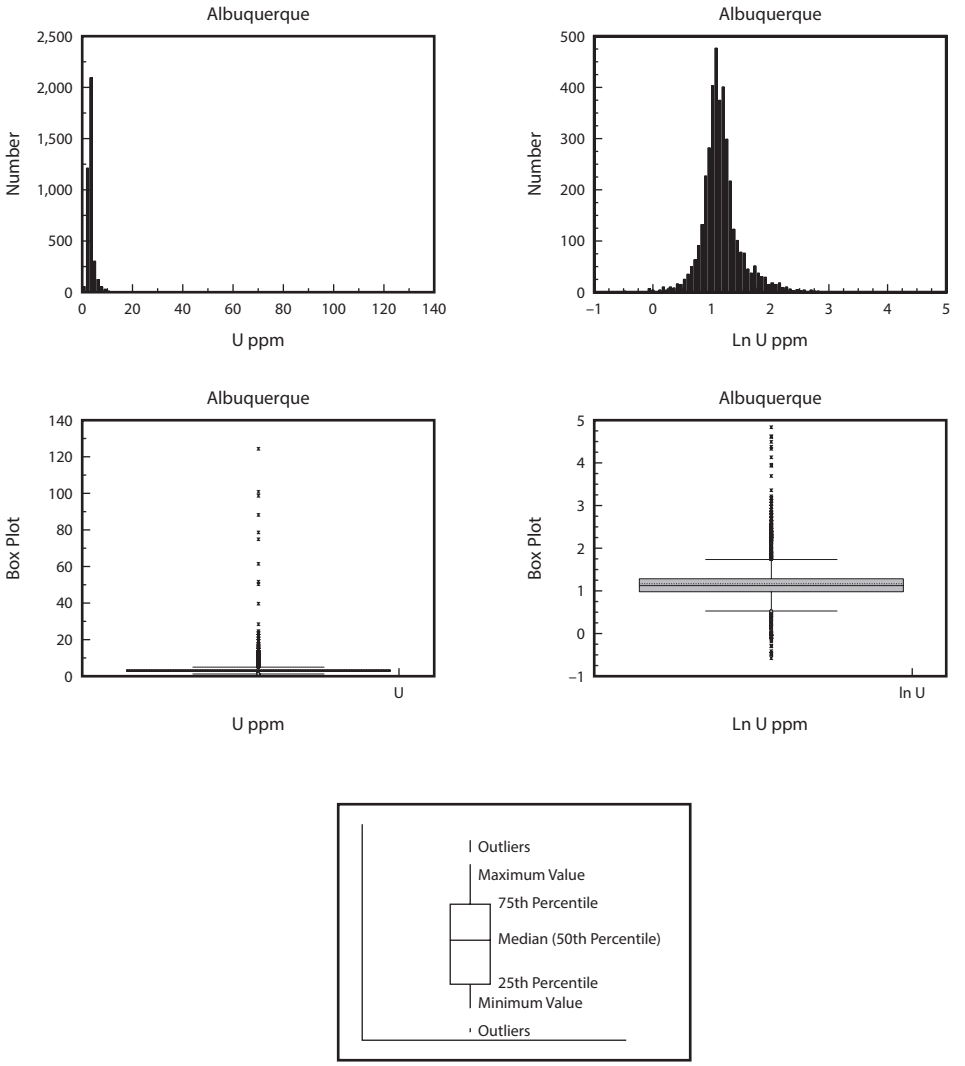
7.4 DATA PRESENTATION

The goal of data presentation is to identify patterns and trends in the data that could go unnoticed using purely numerical methods. In addition, visual tools can be used to effectively convey information to nonscientist decision makers and stakeholders. People tend to understand pictures much more readily than mathematical formulas. Graphs can be used to identify patterns and trends, to quickly confirm or disprove hypotheses, to discover new phenomena, to identify potential problems, and to suggest corrective measures. Graphical representations include displays of individual data points, statistical quantities, temporal data, and spatial data. Because no single graphical representation provides a complete picture of a data set, the analyst should choose different graphical techniques to illuminate various characteristics of the data. At a minimum, the analyst should choose a graphical representation of the individual data points and a graphical representation of basic statistical properties. An example of discontinuous data is the number of samples collected on a specific date. In contrast, an example of continuous data would be measurement of flow as a function of time.

Numerous options for data presentation are described as follows:

- A bar chart has a vertical and horizontal portrayal of information with the rectangular bars being proportional in length to the values they represent. Bar charts are used for plotting discrete (or "discontinuous") data (i.e., data that have discrete values and are not continuous). Comparing the monitoring results to the goals established in the sampling and monitoring plan is very useful. An example is a comparison of concentrations of metals in water with the appropriate water quality criteria.
- A histogram is a graphical representation that looks similar to a bar chart but consists of tabular frequencies or probability distribution of a continuous variable (Figure 7.2). The categories are usually specified as consecutive, non-overlapping intervals of a variable. The categories (intervals) must be adjacent and are often chosen to be of the same size. For example, the number of samples by month that have a pH of less than 5 can be portrayed in a histogram.

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Source: McLemore 2010.

FIGURE 7.2 Histograms and box plots of uranium analyses in stream sediments for the Albuquerque 1:250,000 topographic quadrangle

- A box plot (also known as box-and-whiskers plot) is composed of a central box divided by a line with two lines extending out from the box called whiskers (Figure 7.2). The length of the central box indicates the spread of the bulk of the data (the central 50%), whereas the length of the whiskers show how stretched the tails of the distribution are. The width of the box has no particular meaning, so the plot can be made quite narrow without affecting its visual impact. The sample median is displayed as a line within the box, and the sample mean is displayed using a plus (+) sign. Any unusually small or large data points are displayed by an asterisk (*) on the plot. A box plot can be used to assess the symmetry of the data. If the distribution is symmetrical, then the box is divided in two equal halves by the median; the whiskers will be the same length and the number of extreme data points will be distributed equally on either end of the plot.

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7.5 DATA INTERPRETATION

Although computers are quite helpful in organizing data, deciphering the story behind these facts remains a human task. The overall purpose of data interpretation is to get answers to study design questions—the same questions that originally provoked the start of a monitoring program. The products of risk assessment (interpretation) can be thought of as, among other things, communication products. Their value lies in their contribution to the objectives of the decision-making function, including their effects on the primary decision maker and other interested parties who participate in the decision or otherwise use the information that the products convey. As in any complex design problem, the process of design is intended to find the best solution to achieve multiple simultaneous and competing objectives while satisfying constraints on the process or the end product. One will need to avoid *confirmation bias*. As the term is typically used in the psychological literature, it connotes the seeking or interpreting of evidence in ways that are partial to existing beliefs, expectations, or a hypothesis.

Data are the raw numbers collected from the appropriate sources at the proper times using the correct methods to answer a question. This is true for all data (e.g., geologic, mineralogical, meteorological, financial, chemical, biological). These data are analyzed numerically, spatially, visually, and/or statistically and converted into information. The information received is turned into knowledge based on experience, training, and intuition; then this knowledge is used to make decisions. Without quality data, informed decisions cannot be made. Monitoring data are assessed against benchmarks, data from reference sites, or conditions, as well as by comparison with accepted guidelines for water, sediment, human health, and food quality. The development of regulatory guidelines and standards relies on published data from laboratory monitoring of human and environmental toxicology.

Interpretation of data is the process of analyzing the data in context with other information about the samples, the site, and their environment, and to understand the physical, chemical, and biological processes at work. Too often overlooked is the process of comparing the data with criteria or a standard. The planning sequence of moving from data analysis to information and knowledge is supposed to provide confidence that the sometimes costly actions to address an environmental problem are justified. The scientific method starts with limited data and information from which a tentatively held hypothesis about cause and effect is formed. The hypothesis is tested, and new understandings and new hypotheses can be stated and tested. By definition, science is the process of iterative questioning. This sampling and monitoring handbook recommends policy decisions be based on *the science*. Decisions to pursue some actions must be made, based on a preponderance of evidence, but there may be a need to continue to apply science as a process (data collection and tools of analysis) in order to minimize the likelihood of future errors.

The terms *data* and *information* are often used as synonyms, but data are not the same as information. Unanalyzed data do not constitute information. Data must be interpreted for their meaning through the filter of analytical techniques, and the result of such data analysis is information that can support decision making. Knowing what data are needed and turning those data into information constitutes, in large part, the science behind an environmental management program. Information for decision making should be based on carefully collected and interpreted monitoring data.

The techniques for transforming data into information include statistical inference methods, simulation modeling of complex systems, and, at times, simply the application of the best professional judgment of the analyst. In all these processes, there will always be some uncertainty (and thus some *unreliability*) about whether the resulting information accurately characterizes the environmental problem and the effectiveness of the solutions. Because uncertainty cannot

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be eliminated, determining whether the information generated from data analysis is reliable is a value judgment. Individuals and groups will have different opinions about whether and how to proceed with environmental management given a certain level of uncertainty. An optimal project design balances the uncertainties and associated costs with the resources available from the project.

7.5.1 Statistical Analysis

Statistical analysis is an important tool in summarizing, describing, analyzing, interpreting, and presenting data, and can be used as a predictive tool, but it must be used with caution and complete understanding of the data limitations and the problem to be addressed. Numerous statistical packages are available to summarize, analyze, and interpret the data (USEPA 2006, 2008). Statistical analysis of data is useful for four reasons:

1. Statistics can be used in evaluating the precision and accuracy of the data.
2. Statistics can aid in interpretation of the data and coming to justifiable conclusions.
3. Statistics can help in developing a better approach to the project, before implementing the sampling and monitoring program.
4. Statistics can be used to scientifically justify the conclusions.

It is not practical in this section to describe a rigorous evaluation and description of the advantages and disadvantages of statistical techniques. For most data, relatively simple statistical techniques will result in the most obvious interpretations. Statistics measure central tendencies and dispersion of unbiased samples. It is important to remember that a sample never defines a population perfectly; error always exists.

Once data are in hand, a number of statistical tests can be performed. Testing includes (1) tests of differences between groups (independent samples), used when a comparison is made between samples; (2) tests of differences between variables (dependent samples), used when a comparison is made between variables measured in the same sample; and (3) tests of relationships between variables (e.g., correlation coefficients). Statistical estimates can also be performed.

The choices among space, time, and thematic parameters that are made in designing a monitoring program also affect the choice of statistical analyses. Geostatistical techniques may be desired for analyzing data collected at a large number of sites. The goal of the first step in statistical analysis is to summarize some basic quantitative characteristics of the data set using common statistical quantities. Some statistical quantities useful to the analyst include

- Number of observations;
- Measures of central tendency, such as a mean, median, or mode;
- Measures of dispersion, such as range, variance, standard deviation, coefficient of variation, or interquartile range;
- Measures of relative standing, such as percentiles;
- Measures of distribution symmetry or shape; and
- Measures of association between two or more variables, such as correlation.

Many types of statistics can be used (Isaaks and Srivastava 1989; Rollinson 1993; Helsel and Hirsch 2002). Two of the most frequently used descriptors of environmental data are the mean and standard deviation. Textbook statistics commonly assume that if a parameter is measured many times under the same conditions, then the measurement values will be randomly distributed around the average with more values clustering near the average than farther away. In this

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ideal situation, a graph of the frequency of each measure plotted against its magnitude should yield a bell-shaped, or normal, curve. Although both the mean and the standard deviation are quite useful in describing environmental data, the actual measures often do not fit a normal distribution. Other statistics sometimes come into play to describe the data (USEPA 2006).

Normal distributions of geochemical data should not be assumed (Rollinson 1993; Helsel and Hirsch 2002) and can be determined by histograms or other statistical methods. A normal distribution is an accepted model of the behavior of certain random events and is used to approximate other probability distributions. Classical statistical analysis requires that the data are normal or log-normal and represent one population.

Descriptive statistics use indicators such as the mean, median, mode, standard deviation, coefficient of variation, and so on. The *mean* or *arithmetic average* is the sum of the values divided by the number of values. The *median* is the middle value of a list ranged from lowest to largest. The *mode* is the value of a variable that occurs most frequently, and there can be multiple modes in a set of data that is not normally distributed. The *frequency distribution* or the *frequency of occurrence* is typically illustrated by a histogram (a bar or line graph of values where the length of the line shows the frequency of that value), cumulative frequency plot, or density function plot. A *normal distribution* or *bell curve* is a density function plot where the curve is regularly shaped; other distributions of data are likely (Schreuder et al. 2004). The *standard deviation* is a measure of how much the data deviates from the mean.

Additional statistical tools are useful in sampling and monitoring programs. Regression analysis is the process used to determine the best fit of a line or curve to a set of data consisting of dependent and independent variables; linear regression is used to describe how well the trend fits along a line. Curvilinear regression is used to describe how well the trend fits along a curve. Factor analysis is a statistical tool used to group a set of many observed variables into two or more related factors. A correlation is used to define the linear relationship between two variables. Pierson correlation is obtained by dividing the covariance of the two variables by the product of their standard deviations. A correlation coefficient of 0.8 is generally a good correlation, although in some cases, lower correlation coefficients could be acceptable or indicate significant data relationships. The term *trend* is used when the data indicate a relationship, but not a strong linear or curvilinear correlation.

Outliers are the sample points that appear to be inconsistent with the remainder of the collected data (Iglewicz and Hoaglin 1993) and are important to identify and properly interpret their source. Outliers can be determined by visual inspection of scatter plots and data tables or by values greater than 1 standard deviation. Some outliers can be identified as recording errors by checking the outlier values with the original data set; such errors must then be corrected in the final data set. Any remaining outliers should be identified, and, if appropriate, described on a case-by-case basis in the project reports. In some cases, the tests or measurements can be rerun to confirm the outlier value; appropriate notes should be recorded in the database (USEPA 2000b). See Unusual Measurements sidebar.

It also is important to realize that a correlation does not imply causation or that a correlation cannot always be used to infer a causal relationship between the variables. Correlations need to be examined by defining geologic, geochemical, or geotechnical processes that could be responsible for the correlation. It is important to try to understand variations in the data. In many cases, the variation is sample heterogeneity.

Statistical analyses can be used for description, communication, and to test hypothesis regarding the population from which the data were drawn. The confidence interval estimates the range in which a population parameter falls. A confidence interval is a range of a probability

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UNUSUAL MEASUREMENTS CAN BE VALID—IRON MOUNTAIN MINE

Carol C. Russell, U.S. Environmental Protection Agency

From the 1860s through 1963, the Iron Mountain mine site in northern California was periodically mined for iron, silver, gold, copper, zinc, and pyrite. Many miles of streams, tributaries, and reservoirs within the Sacramento River Basin are impacted by the acid drainage (AD) from the mine with a pH level measured at –3.6. Salmon kills have been noted since 1899. Releases of stored AD into the reservoirs are timed to coincide with the presence of diluting releases of water from Shasta Dam. One option for remediation was to plug the mine portal. Studies predicted that if the mine portals were plugged, a pool of water would develop with extremely acidic pH (near 1) and high metal concentrations (grams of dissolved metals per liter). The flooded mine pool would have a volume of approximately 600,000 m^3 and would present a high risk to the environment and to the local community because of the potential for catastrophic plug failures, faulty plug seals releasing mine water, and acid seeps developing in other locations. Based on this information, plugging of the mine was dropped from consideration. Instead, a combination of remediation activities was chosen with the clear understanding that new technologies are needed. The options chosen were an acid neutralization treatment plant (lime/sulfide high-density sludge treatment process), capping of selected areas, and surface water diversions. An agreement regarding the payment to state and federal governments of close to $1 billion has been structured using a unique funding mechanism of combined financial assurance and insurance (USEPA 2010, 2011).

distribution and does not provide any information about individual values in the population. Hence, confidence intervals cannot be used in applications such as comparison to environmental standards, because the confidence interval estimates a population parameter (e.g., mean) but does not provide information about individual values. The tolerance interval estimates the range that should contain a certain percentage of each individual measurement in the population. Tolerance intervals tell us what the individual values should be and are especially useful in compliance monitoring when one is concerned with maximum contaminant levels.

The two significant sources of uncertainty in any water quality management program are epistemic and aleatory uncertainty (Stewart 2000). *Epistemic uncertainty*, incomplete knowledge or lack of sufficient data to estimate probabilities, is a result of reliance on models relating sources of pollution to human health and biological responses. Our conceptual understanding of the systems under study is incomplete, because models are necessarily simplified representations of the complexity of the natural systems and are derived from limited data for simulating the systems. Thus, the goal should be to increase the availability of data, improve its reliability, and advance the modeling capabilities (NRC 2001).

Complete certainty in support of environmental management decisions cannot be achieved because of *aleatory uncertainty*, the inherent variability of natural systems and processes. Aleatory uncertainty arises in systems characterized by randomness. Not only is the environment characterized by randomness, but it is also an open system in which the boundaries of possible biological outcomes cannot be known in advance.

7.5.2 Spatial Interpretation Methods

Spatial interpretation or analysis includes any technique using topological, geometric, and geographic properties. In mining and environmental science, spatial analysis generally involves

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different techniques that show samples and resulting data on maps. Techniques such as variography, contouring, bubble plots, and kriging are useful in determining optimal sampling patterns for the area and to interpolate values in between sample points. GIS technology has evolved into a powerful tool in evaluating and analyzing large, complex data in the mining, environmental, and geological fields. ESRI ArcMap is a part of ArcGIS and includes a suite of geospatial processing programs that allows the viewer to examine, plot, edit, and analyze geospatial data and aid in establishing relationships between complex sets of data; see Section 7.8). Integrating numerous data sets is an important tool in mine reclamation sites (Grunsky 1997; McLemore 2010). Pattern recognition is a key concept in data analysis, and a variety of techniques are available to evaluate the geographic representation of the data. The R Project for statistical computing (see Section 7.8) provides another technique for evaluating spatial data. In many instances, statistical analysis of the data is the only way to interpret the large quantity of data collected. However, special problems may be encountered when statistical techniques are applied to geological and geochemical data because of the heterogeneity of the earth's crust. Geostatisticians are constantly refining statistical techniques to better account for these heterogeneities.

Geostatistics is a branch of applied statistics concerned with spatial data and can be very useful in interpreting environmental data. In geostatistics, each data value is associated with a location in space. *Semivariance* is a measure of the degree of spatial dependence between samples. The plot of the semivariances as a function of distance from a point is referred to as a semivariogram. Use of geostatistics in data analysis impacts sampling methods. In designing a sampling plan intended for geostatistical interpretation, there is no need to avoid autocorrelations; hence, sampling becomes less restrictive. Use of geostatistics changes the emphasis from estimation of averages to mapping spatially distributed populations. *Kriging* is a geostatistical technique to interpolate the value at an unobserved location from observations of its value at nearby locations.

7.5.3 Models

Models can be very useful tools for data interpretation. For example, the conceptual model described in Chapter 5 can be used to test a hypothesis that is later confirmed, or not, through data collection. A predictive model is broadly defined to include both mathematical expressions and expert scientific judgment. Mathematical models can be characterized as *empirical* (also known as statistical) or *mechanistic* (process-oriented), but most useful models have elements of both types. An empirical model is based on a statistical fit to data as a way to statistically identify relationships between stressor and response variables. A mechanistic model is a mathematical characterization of the scientific understanding of the critical biogeochemical processes in the natural system; the only data input is in the selection of model parameters and initial and boundary conditions.

Data availability is one source of uncertainty in the development of models for decision support. Although there are other sources of uncertainty as well, models should be selected (simple versus complex) in part based on the data available to support their use. The use of complex mechanistic models is warranted if it helps promote the understanding of complex systems and as long as uncertainties in the results are reported and incorporated into decision making. However, there may be a tendency to use complex mechanistic models to conduct water quality assessments in situations with little useful water quality data and/or involving major remediation expenditures or legal actions.

7.6 DECISION-MAKING PROCESS

In general, decision making follows a six-step format:

1. Formulate the problem (question) to be answered,

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2. Analyze the risks,
3. Define the options or alternatives,
4. Make decisions based on available information and risk analyses,
5. Take actions to implement the decisions, and
6. Perform an evaluation of the effectiveness of the actions taken.

The first step of problem formulation corresponds to the first component of a sampling and monitoring program: *defining the purpose, questions, and objectives of the sampling and monitoring program*. At this step, ask if the correct questions were asked and if the data are of sufficient quantity and quality to answer the questions. If not, what are the data gaps or does more data need to be collected. Each case study in Appendix 5 discusses the purpose, questions, and objectives of the study.

The second step, analyzing the risks, was covered in Chapter 4. For the purposes of this handbook, *risk* is defined as the measure of the probability that damage to life, health, property, and/or the environment will occur as a result of a given hazard or condition.

For step three, defining the options or alternatives, it is recommended that the framework for decision making has the capacity to engage a comprehensive range of stakeholders and address the interdependence and cumulative effects of various options or alternatives.

Step four, making sound decisions, appears to be easy, but it is often the most difficult part of a sampling and monitoring program, especially if decision makers or the public were not involved early in the process of defining information needs. Sound environmental decision making is dependent on effective working relationships among scientists, policymakers, and the public based on reliable and timely *translations* of information and views between the communities. To facilitate communication between these groups, it is helpful to understand the general perspectives and potential biases of the groups. From the scientists' perspective, many management decisions are not made based on complete scientific data. From the policymakers' perspective, all scientists want to do is to collect more data. From the public's standpoint, risks that are voluntarily undertaken are downplayed. The public fears what it doesn't know and what it has no control over. The public tends to believe the first reports no matter the sources, even if they are preliminary and change with new data. Risks considered most serious by the general public can be different from those considered most serious by the technical professionals and policymakers charged with reducing environmental risk (see Education and Research Opportunities sidebar).

For example, some chemicals associated with the mining industry, such as cyanide, are not well understood by the public, have an automatic negative connotation in the media and public consciousness, and may need to be considered prominently in a sampling and monitoring program simply to improve public perception, understanding, and to allay concerns. The public process is doomed if the company is perceived to be reactive rather than proactive.

Step five, taking actions to implement the decisions, is considered self-explanatory. The action plan needs to be readily available to those implementing, monitoring, and analyzing the plan.

Step six is the step seldom taken in a sampling and monitoring program, but it is one of the most important steps: performing an evaluation of the appropriateness and effectiveness of the actions taken. Determine from all involved parties (the decision makers, stakeholders, and interested parties) if the information was adequate and useful. Documentation of lessons learned can provide amazing insights for your next sampling program. Each case study in Appendix 5 discusses the lessons learned from the selected study.

When in doubt regarding the final decision, one should use the precautionary principle as recommended by Morgan et al. (1990) as follows: "Increasingly, environmental decision-making

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EDUCATION AND RESEARCH OPPORTUNITIES AT THE SLIPPERY ROCK WATERSHED, PENNSYLVANIA

Virginia T. McLemore, New Mexico Bureau of Geology and Mineral Resources

The Slippery Rock Watershed Coalition was created to restore a portion of the Slippery Rock Watershed in northwestern Pennsylvania that is impacted by acid mine drainage from abandoned coal mine complexes. The coalition has successfully utilized local college students from Grove City College in providing background data for passive treatment systems. In 1998, a scale model of a vertical flow system and an aerobic wetland was constructed in part by the college students to improve the quality of the water in that portion of the watershed. A monitoring program was developed and the students collected samples weekly as part of this program. Current results indicate that the system is viable for improving the water quality. This is one of many examples where the local community can be successfully involved in sampling and monitoring programs. In this case, the educational opportunities offered to the college students were invaluable (Brenner 2002), and these efforts lower the cost of the reclamation (Taylor et al. 2002).

A similar coalition was developed elsewhere in the Slippery Rock Watershed called the Jennings Water Quality Improvement Coalition. Elimination or abatement of degraded acid drainage from nearby coal mines has been attempted four times over the past 30 years, with little effect on the water quality. In 1997, the coalition, including government agencies, mining and environmental companies, Girl Scouts, college and home-school students, and other volunteers, constructed a vertical flow system. Monitoring of the system was by coalition volunteers from September 1997 through June 1999, and results indicate that the system continues to discharge alkaline water with low concentrations of metals (Taylor et al. 2002).

is scrutinized with respect to a precautionary principle. This principle asserts that where uncertainty and doubt make it impossible to be sure about a correct decision, any errors should favor the long-term sustainability of the environment" (Morgan and Henrion 1990; Raiffa 1997). The academic discipline of decision analysis under uncertainty, among others, has a rich literature on which to draw for methods and findings.

7.6.1 The Decision-Making Environment and the Importance of Process

The importance of attention to *process* is entirely compatible with the theory of the management sciences that defines a *good decision* under uncertainty as one that uses the most appropriate processes and methods to assemble and interpret evidence, to apply the decision makers' values properly, and to make timely choices with available resources, rather than defining a good decision only according to its (apparent) outcomes. Most decision-making situations involving matters of public health and environmental risk have four common elements:

1. The desire to use the best scientific methods and evidence to inform decisions
2. Uncertainty that limits the ability to characterize the magnitude of the problem and the corresponding benefits of proposed actions
3. The need for timely decision making that precludes resolving important uncertainties before the decisions are required
4. The presence of some sort of trade-off among adverse outcomes (which may be health, ecologic, or economic outcomes, each affecting a different set of stakeholders)

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Most decisions that are based on data from sampling and monitoring programs are made to avoid risks, reduce risks, reduce the consequences of events, or ensure against the financial consequences of risks, as described in Chapter 4. Finally, the data, interpretation, and results need to be presented to the decision makers in a form that is useful to them such as in reports and presentations.

If monitoring data are limited, and when the decisions are under significant uncertainty, the precautionary principle comes into play. Where a course of action poses a risk that cannot be fully quantified, the precautionary principle provides conservative protection in the case of potential harm even if the probability cannot be determined accurately by the existing science (Crawford-Brown and Crawford-Brown 2011). Adaptive management refers to an iterative, systematic monitoring process to support robust decision making in the face of uncertainty. The U.S. Council on Environmental Quality issued a guidance memorandum regarding mitigation and monitoring that encourages the use of adaptive management (CEQ 2011):

> The guidance encourages agencies to develop internal processes for post-decision monitoring to ensure the implementation and effectiveness of the mitigation. It also states that agencies may use adaptive management as part of an agency's action. Adaptive management, when included in the NEPA [National Environmental Policy Act] analysis, allows for the agency to take alternate mitigation actions if mitigation commitments originally made in the NEPA and decision documents fail to achieve projected environmental outcomes.

A critical dimension of scope is the explicit inclusion of the various possible mitigation options that might be considered to reduce the risk that is being assessed. The scope may need to be expanded so that the assessment will provide not only estimates of existing risk, but estimates of risk reduction associated with changes in the risk-generating mining operation.

7.6.2 Decision Makers and Stakeholders

Interaction with the stakeholders (i.e., all interested parties), especially those making decisions, is an imperative, integral, and iterative part of a sampling and monitoring program. The public stakeholders should be involved throughout the project to maintain transparency and manage potential conflict caused by misconceptions or false data (see Stillwater Mining sidebar). The classic regulatory requirements of public participation should not be the goal, but the decision makers must be involved from the beginning of the process through to the end. Monitoring for monitoring's sake is worthless. Sampling and monitoring should be conducted to answer questions, and who better to define what is needed than those people asking the questions and making the decisions.

Legally required participation methods in decision making in the United States, such as public hearings, review and comment procedures in particular, are not always the best procedure. Not only do they not always meet most basic goals for public participation, but they can be counterproductive, causing anger, outrage, and mistrust. Research shows that collaborative participation can solve complex, contentious problems (Innes and Booher 2003). Participation in a sampling and monitoring program by the public can take on a variety of components. For example, local communities can have access to information and data unavailable to those conducting a monitoring program. They can also have questions that need to be answered as part of the sampling and analysis of data. Moreover, active participation in a sampling and monitoring effort tends to eliminate questions of data validity because participants have ownership in the process. This collaborative dialogue is a multi-way interaction in which citizens and other players work and talk in

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STILLWATER MINING'S GOOD NEIGHBOR AGREEMENT

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More than 10 years ago, Stillwater Mining Company, Northern Plains Resource Council, Stillwater Protective Association, and Cottonwood Resource Council established an innovative agreement that resolved disputes, protected the environment, and encouraged responsible economic development. The good neighbor agreement (GNA) provided solid criteria for the implementation of water monitoring plans, aggressive water discharge limits (that exceed normal regulatory standards), biological monitoring, reclamation, and closure plans with an emphasis on "best management practices" for all mining activities. A unique component of the GNA is that it provided the councils a voice in the decision-making process and an opportunity to review and comment on plans, procedures, and proposed solutions up front in the planning stage where issues can be addressed before development. This process minimizes the potential for conflicts that can slow or stall development and, at the same time, ensures that important environmental issues are addressed and factored into mining activities. The most recent environmental impact statement was processed in just a few months with the councils providing information to the regulatory agencies in support of the proposed amendment to the Stillwater long-term water management plans.

formal and informal ways to influence action in the public arena before it is virtually a foregone conclusion. The process is one of give and take, and joint problem solving, possibly in a social network cyberspace.

A common framework that allows for interacting and influencing one another and all are acting independently in the world results in a much better program. This is not one-way communication from citizens to government, or government to citizens, or company to government, or company to citizens. It is a multidimensional model where communication, learning, and action are joined together and where the policy, interests, and citizenry co-evolve. Innes and Booher (2003) describe such a model as follows:

> When an inclusive set of citizens can engage in authentic dialogue where all are equally empowered and informed and where they listen and are heard respectfully and when they are working on a task of interest to all, following their own agendas, everyone is changed. They learn new ideas and they often come to recognize that others' views are legitimate. They can work through issues and create shared meanings as well as the possibility of joint action.

The following tips are suggested for involving communities and stakeholders (USEPA 2000a):

- Know and respect the community.
- Establish an ongoing company presence.
- Maintain communication at all times.
- Devote special attention to historical preservation; concerns deserve special attention.
- Utilize the local community expertise.
- Involve the community in planning and implementing the sampling and monitoring program.

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Mitigation and enhancement measures are best identified and developed in cooperation with local stakeholders. In 2013, the National Academy of Sciences (NAS 2013) suggested that "Early communication facilitates incorporation of stakeholder perspectives in the process and helps to identify uncertainties for consideration in the final decision." Furthermore, the International Council on Mining and Metals (ICMM 2010) produced *Good Practice Guidance on Health Impact Assessment in the Mining and Metals Sector*, an international reference on providing partnerships in decision making for local communities potentially affected by mining. When decisions must be made that affect groups of people or large segments of society, the process should be conducted within a structured framework that helps the decision makers take into account multiple objectives, needs, preferences, and values as well as vulnerabilities, risks, and uncertainties. The structured decision making is applied to break complex decisions into smaller, more tractable parts that are not prone to error and bias and are internally consistent.

7.7 SUMMARY

In order for data to be useful for decision making, it must be transformed into information by the process of assessment and analysis. Often the questions that decision makers would like to answer relate to compliance: what caused the problem, could the problem be prevented, what is the cost, and will it work? In addition, decision makers want to know the reliability of the data. Sometimes the scientific data is valid but the process used to make the decision is flawed. Ultimately, if data are not used to make decisions, they are of limited value.

7.8 INTERNET RESOURCES

ESRI's ArcGIS resources: http://resources.arcgis.com/content/web-based-help

R Project for statistical computing: www.r-project.org/

USEPA's STORET database: www.epa.gov/storet/

7.9 REFERENCES

Brenner, F.J. 2002. Public private partnerships: Education and research opportunities. SME Preprint 02-144. Littleton, CO: SME.

CEQ (U.S. Council on Environmental Quality). 2011. Memorandum on Appropriate Use of Mitigation and Monitoring and Clarifying the Appropriate Use of Mitigated Findings of No Significant Impact, dated January 14, 2011. Washington, DC: CEQ.

Cormier, S.M., and Suter, G.W. 2008 A framework for fully integrating environmental assessment. *Environ. Manage*. 42:543–556.

Crawford-Brown, D., and Crawford-Brown, S. 2011. The precautionary principle in environmental regulations for drinking water. *Environ. Sci. Policy* 14:370–387.

Grunsky, E.C. 1997. Strategies and methods for the interpretation of geochemical data. In *Current Topics in GIS and Integration of Exploration Datasets*. Short Course, Exploration '97 Workshop. Ottawa, ON: Geological Survey of Canada.

Helsel, D.R., and Hirsch, R.M. 2002. *Statistical Methods in Water Resources*. Techniques of Water Resources Investigations, Book 4. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/twri/twri4a3/. Accessed 9/11/13.

ICMM (International Council on Mining and Metals). 2010. *Good Practice Guidance on Health Impact Assessment in the Mining and Metals Sector*. ICMM report. www.icmm.org. Accessed 9/11/13.

Iglewicz, B., and Hoaglin, D.C. 1993. *How to Detect and Handle Outliers*. Milwaukee, WI: American Society for Quality Control.

Innes, J.E., and Booher, D.E. 2003. Collaborative policymaking through dialogue. In *Deliberative Policy Analysis: Understanding Governance in the Network Society*. Edited by M.A. Hajer and H. Wagenaar. New York: Cambridge University Press.

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Isaaks, E.H., and Srivastava, R.M. 1989. *An Introduction to Applied Geostatistics*. New York: Oxford University Press.

McLemore, V.T. 2010. Use of the New Mexico Mines Database and ArcMap in uranium reclamation studies. SME Preprint 10-125. Littleton, CO: SME. http://geoinfo.nmt.edu/staff/mclemore/documents/11-139.pdf. Accessed 9/11/13.

McLemore, V.T., Hoffman, G.K., Wilks, M., Raugust, J.S., and Jones, G.R. 2004. Use of databases in characterization at mine sites. In *Proceedings, 2004 National Meeting of the American Society of Mining and Reclamation*. Lexington, KY: American Society of Mining and Reclamation.

Morgan, G.M., and Henrion, M.H. 1990. *Uncertainty: A Guide to Dealing with Uncertainty in Quantitative Risk and Policy Analysis*. New York: Cambridge University Press.

NAS (National Academy of Sciences). 2013. *Environmental Decisions in the Face of Uncertainty*: Washington D.C. National Academies Press. www.nap.edu/catalog.php?record_id=12568. Accessed 9/11/13.

NRC (National Research Council). 2001. *Assessing the TMDL Approach to Water Quality Management*. Washington, DC: National Academies Press.

Raiffa, H. 1997. *Decision Analysis: Introductory Readings on Choices Under Uncertainty*. New York: McGraw-Hill.

Rollinson, H.R. 1993. *Using Geochemical Data: Evaluation, Presentation, Interpretation*. Harlow, England: Pearson Education, Prentice Hall, Longman Group.

Schreuder, H.T., Ernst, R., and Ramirez-Maldonado, H. 2004. *Statistical Techniques for Sampling and Monitoring Natural Resources*. General Technical Report-RMRS-GTR-126. Fort Collins, CO: U.S. Department of Agriculture, Rocky Mountain Research Station. www.fs.fed.us/rm/pubs/rmrs_gtr126.pdf. Accessed 9/11/13.

Stewart, T.R. 2000. Uncertainty, judgment, and error in prediction. In *Prediction: Science, Decision Making, and the Future of Nature*. Edited by D. Sarewitz, R.A. Pielke, and R. Byerly. Washington, DC: Island Press. pp. 41–57.

Taylor, W., Johnson, D., Dunn, M., and Hedin, R. 2002. A public-private partnership success story: Treating abandoned mine drainage at Jennings Environmental Education Center. SME Preprint 02-144. Littleton, CO: SME.

USEPA (U.S. Environmental Protection Agency). 2000a. *Abandoned Mine Site Characterization and Cleanup Handbook*. U.S. Environmental Protection Agency. EPA /910-B-00/-001. www.epa.gov/superfund/policy/remedy/pdfs/amscch.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2000b. *Guidance for Data Quality Assessment—Practical Methods for Data Analysis* (EPA QA/G-9 QA00 update). U.S. Environmental Protection Agency. EPA/600/R-96/084. www.epa.gov/region6/qa/qadevtools/mod4references/secondaryguidance/g9-final.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2002. *Guidance on Environmental Data Verification and Data Validation*: U.S. Environmental Protection Agency. EPA QA/G-8 240/R-02/004. www.epa.gov/QUALITY/qs-docs/g8-final.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2006. *Data Quality Assessment: Statistical Methods for Practitioners* (EPA QA/G-9S). U.S. Environmental Protection Agency. EPA/240/B-06/003. www.epa.gov/QUALITY/qs-docs/g9s-final.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2008. *Scout 2008 Version 1.0–Quick Overview: A Statistical Software Package for the Classical and Robust Analysis of Univariate and Multivariate Data Sets with and Without Non-Detect Observations*: U.S. Environmental Protection Agency. EPA/600/F-08/016. www.epa.gov/esd/databases/scout/Scout%202008%20v.1.00.01/Scout%20Resources/Scout%202008%20Guides/Quick%20Overview%20Scout%202008%20v.%201.0.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2010. *Integrating Ecological Assessment and Decision-Making at EPA: A Path Forward Results of a Colloquium in Response to Science Advisory Board and National Research Council Recommendations*. EPA/100/R-10/004. www.epa.gov/raf/publications/pdfs/integrating-ecolog-assess-decision-making.pdf. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2011. STORET database. www.epa.gov/storet/. Accessed 9/11/13.

USEPA (U.S. Environmental Protection Agency). 2013. Metadata: Water Quality Observation. http://water.epa.gov/scitech/datait/models/basins/wqobs.cfm. Accessed 12/20/13.

Weight, W.D., and Sonderegger, J.L. 2001. *Manual of Applied Field Hydrogeology*. New York: McGraw-Hill.

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CHAPTER 8

Additional Key Issues and Future Research Needs

Virginia T. McLemore

8.1 INTRODUCTION

Structural problems associated with mines, process facilities, mine structures, and waste management units must be considered to ensure the safety of workers and alleviate environmental impacts that could result from structural failure and a possible release of contaminants into the local drainage. Even avalanches could affect the best sampling and monitoring plans (see The Effect of Avalanches sidebar). Most of these features should have a monitoring plan to examine them periodically during the mine life cycle and even after operations. The following sections briefly describe some of these stability issues and their effect on water quality. This chapter concludes with some comments on research needs and topics.

8.2 SLOPE STABILITY AND FAILURE

A slope failure is the uncontrolled or unscheduled release of the slope material beyond the confines of the original topography (Robertson and Skermer 1988; Neuendorf et al. 2005). Most slope failures are caused by disruptive actions that can be described in one of two ways:

1. Sudden, intense, or extreme events such as floods that cause liquefaction (Hutchinson 1988), earthquakes, volcanic action, avalanches, and glaciation, which apply forces exceeding the values for which the slopes were originally designed.
2. Slow, but perpetual actions of wind and water erosion, including frost action, lubrication, other forms of weathering and decomposition, chemical reaction, and biological actions such as intrusion by roots, animals, and humans.

The most common slope failures at mine sites are from slopes forming the open pit walls, mine waste piles, and process tailings impoundments, although any slope that has been disturbed by mining or construction activities can fail under the right conditions, including slopes adjacent to roads and railroads.

Two types of slopes are associated with mine sites: cut slopes and manufactured or filled slopes. The excavation of overburden and/or ore creates cut slopes, also called benches or highwalls. Manufactured or filled slopes are formed by the dumping or piling of overburden, process tailings, waste rock, or other materials; these features can be high in metals, acid forming, or reactive. Slope failure as a result of gravitational forces or by saturation of the material by water can result in direct release or direct exposure of these materials to the surrounding weathering environment. Changes to an existing slope can create environmental problems, such as increased erosion, rapid runoff, sedimentation, changes in wildlife patterns, and the exposure of potentially

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THE EFFECT OF AVALANCHES AT THE OPHIR MINE, COLORADO

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Ophir, a small mining town located 12 km (7.5 miles) from Telluride, Colorado, was heavily damaged by snow avalanches in the early 1900s during the mining boom. Active treatment of acid rock drainage (ARD) in this area would be very expensive and would be hampered by liability provisions of the Clean Water Act. Likewise, more passive-treatment strategies, such as diverting ARD through constructed wetlands and bioreactors, must still be regarded as experimental because of the cold temperatures at this elevation. In addition, the site is potentially hazardous because of the risk of destruction by snow-slides. The U.S. Environmental Protection Agency recommends strategies to intercept and divert water away from mineralized zones, thereby reducing metal loading to the Howard Fork.

reactive natural materials. In many instances, slope failures result in degradation of the drainage quality of the receiving stream.

Slope failures and breaches in tailings piles are more common, whereas sudden slope failures of mine rock piles are not as common. However, when any slope failure occurs, the resulting publicity feeds public perceptions that these mine features are hazardous.

Four types of sudden, intense failures of mine-waste rock piles are (Pernichele and Kahle 1971; Whiting 1981)

1. Debris flow (also known as flow slide),
2. Foundation failure,
3. Edge slump, and
4. Blowout (internally generated by elevated hydraulic pressures that accumulate in some layers resulting in decreasing internal effective stress and stability).

Many investigators have studied the mechanisms that result in failed rock piles, which are summarized by McLemore et al. (2009). Any disturbances should be routinely sampled and monitored throughout the mine life cycle.

8.3 TAILINGS IMPOUNDMENTS

Tailings are the fine-grained residue of mineral processing typically found at mill sites. Some other terms applied to tailings include slimes, tails, leach residue, or slickens. The volume of tailings generally exceeds the volume of the recovered minerals by several orders of magnitude. Most tailings contain water and are stored in impoundments, typically called tailings storage facilities (or ponds), where the water is reused in the mill or allowed to evaporate and the solid particles settle to the bottom. The tailings are contained by liners, natural rock formations, and human-made steep slopes called tailings dams and generally consist predominantly of sand-size and smaller fragments.

Selected types of tailings release and damage to the environment at tailings storage facilities include the following (USCOLD 1994; Klohn 1995; Davies 2002; Blight and Fourie 2003; Mayoral and Romo 2008; Azam and Li 2010):

- Unusual weather
- Overtopping

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- Static instability or structural defect (liner failure)
- Seismic instability (earthquakes)
- Internal erosion or piping
- Wind-blown dust
- Seepage or drainage of liquid wastes, such as acid drainage
- Mine subsidence
- Weak foundation
- Excessive water level rise

Not only can water quality be affected, but the entire downstream ecosystem can be adversely impacted. Flooding can contaminate surface water, groundwater, and soil, and it can adversely affect wildlife and humans.

Although engineering of tailings dams has improved since the 1990s, tailings dam failures still occur nearly every year somewhere in the world (Table 8.1; Davies et al. 2002; McLemore et al. 2009; Azam and Li 2010) and cost millions of dollars to remediate. These tailings dam failures can have serious impacts downstream. In Europe, the most common cause of tailings dam failures is related to unexpected rainfall above the engineered strength of the tailings dam (Rico et al. 2008). Elsewhere in the world, other causes of tailings dam failure are seismic liquefaction (Rico et al. 2008), inadequate management, errors in site selection, poor foundation, and lack of stability of downstream slopes. Most of the dam failings occur at active mine sites (Davies et al. 2002; Azam and Li 2010), where sampling and monitoring plans are generally in place. Sampling and monitoring of water within and surrounding tailings facilities and reclaimed water are typically routine.

The design, construction, operation, and decommissioning of tailings dams must be carefully planned in order to ensure their safe performance. Safe tailings dams can be built by utilizing sound engineering practices and understanding climatic and tectonic conditions. Lessons learned from previous incidents should be incorporated into any future tailings design.

8.4 SUBSIDENCE

Mine subsidence is the collapse of overlying geologic formations into mine voids, and the potential for subsidence exists for all underground mining. Effects of subsidence are not always visible at the ground surface. Subsidence can manifest itself in the form of sinkholes or troughs at the surface. Sinkholes are usually formed by the collapse of a portion of a mine void (such as a room in room-and-pillar mining or block caving techniques). The extent of the surface disturbance is usually limited in size. Natural sinkholes and caves are common in terrains underlain by limestone or other carbonate or evaporate rock, and mining can enlarge these natural features. Many types of underground mining can result in subsidence at the surface. Troughs are formed from the subsidence of large portions of the underground void and typically occur over areas where most of the resource has been removed. Sinkholes or depressions in the landscape interrupt surface-water drainage patterns; ponds and streams may be drained or channels can be redirected. In developed areas, subsidence has the potential to affect cultural features such as building foundations and walls, highways, and pipelines. Subsidence can contribute to increased infiltration to underground mines, potentially resulting in increased acid rock drainage (ARD) generation. Groundwater flow can be interrupted as impermeable strata break down and could result in flooding of the mine and drying up of water wells. Impacts to groundwater include changes in water quality, permeability, and drainage flow patterns (including surface-water recharge). Monitoring

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TABLE 8.1 Recent tailings dam incidents

Date	Location	Company	Type of Ore	Description	Release	Impacts	Reference
July 21, 2011	Mianyang City, Songpan County, Sichuan Province, China	Xichuan Minjiang Electrolytic Manganese Plant	Mn	Tailings dam damaged from landslides caused from heavy rains	?	Damaged roads, houses, and local water supply	WISE (2013)
October 4, 2010	Kolontár, Hungary	MAL Magyar, Aluminum	Bauxite	Tailings dam failed	700,000 m^3 of red mud	Several towns (8 km^2) flooded; 10 people killed	Boston.com (2010)
June 25, 2010	Huancavelica, Peru	United Minera Caudalosa Chica	Ag, Cu, Pb, Zn ore	Tailings dam failed	21,420 m^3 of tailings	Contamination of Rio Escalera and Rio Opamyo 110 km downstream	Guerra (2010)
August 29, 2009	Karamken, Russia	—	Gold	Tailings dam failed after heavy rain	—	11 homes destroyed; 1 person killed	WISE (2013)
December 22, 2008	Kingston Fossil Plant, Harriman, Tennessee	—	Coal ash	Retention wall failed	4.1 m^3	Covered 400 acres; damaged 12 homes	Dewan (2008)

NOTE: Most of these failures occurred outside of the United States. Other incidents are summarized at www.tailings.info/accidents.htm.

of the surface topography throughout the mine life cycle and even after closure can predict subsidence issues at most facilities.

8.5 MINE OPENINGS

Horizontal and vertical mine openings can be physical hazards and potential threats to the general population if not properly secured. Adventurous individuals want to enter them to explore, and can possibly be injured or killed as a result of unstable ground or bad air (e.g., insufficient oxygen or presence of poisonous gases such as carbon monoxide). Mine openings can also be the source of drainage of mining influenced water (MIW) that eventually enters the local drainage system, possibly causing contamination. Draining adits or tunnels are a particular concern, because these adits were excavated beneath the ore horizons with the sole purpose of draining the ore body and typically contain a variety of metals, which can enter the local drainage system. Detailed sampling and monitoring of these features can determine if they pose an environmental threat. The Argo Tunnel near Idaho Springs, Colorado (Figure 8.1)—an adit used to drain upper mine workings—typically produces water flow rates ranging from 200 to 300 gpm, depending on the season (see sidebar in Chapter 2). The pH ranges between 2.7 and 3.2, and the water contains high concentrations of zinc, iron, sulfate, cadmium, copper, and manganese.

8.6 CLIMATE CHANGE

Climate changes are natural processes and found throughout the geologic past. It is not as important to identify the source of potential climatic changes (i.e., emissions from humans, volcanic eruptions, deep sea methane release, etc.) as it is to recognize that climate changes are inevitable. Potential changes include, but are not limited to, the following (Trenberth 1999; Arctic North Consulting 2009; Geyer and Wasilewski 2009):

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Courtesy of Virginia T. McLemore.

FIGURE 8.1 Argo Tunnel near Idaho Springs, Colorado

- Temperature changes, over the course of a year and during the seasons
- Changes in precipitation and in relative amounts of snowfall versus rainfall
- Changes in timing of precipitation events
- Rising sea level due to melting of ice caps
- Air quality changes
- Increases in wind, floods, heat waves, and storms (cyclones, tornadoes, hurricanes, ice storms)

Future climate changes and extremes can impact the mining industry in different ways (OCCIAR, n.d.; Nelson and Schuchard, n.d.). Two major effects of climate change should be considered: how the mine operations may impact the future climate and how the mine company will adapt to a changing climate. A changing climate can result in modifications of existing sampling and monitoring plans.

Very large mines and process facilities have the potential to impact local and even global climate change by increasing the amount of carbon dioxide (CO_2) due to acid neutralization by limestone, kiln and roaster process operations, and other greenhouse gases in the atmosphere. Most operations in the United States will be required to evaluate the carbon impact from their operations. As forests and other vegetation are cleared, CO_2 is no longer taken up by the vegetation and remains in the atmosphere. CO_2 and other gases can be released into the atmosphere by mining equipment and processing facilities (Norgate and Rankin 2000) or through liberation of methane from coal and other sedimentary units during mining. Environmental Law Alliance Worldwide (2010) estimates that more than 1 kg of greenhouse gas is emitted into the atmosphere for every 1 kg of metal produced (Norgate and Rankin 2000). Studies of climates in geologic times suggest that an increase in CO_2 levels can result in a more humid climate in some areas that are higher in precipitation and temperature (Retallack 2009). Other areas can experience drier conditions. Some mining companies are taking steps to improve the energy efficiency of their mining and processing operations. More research is needed to understand what, if any, effects mining and processing can have on climate and how to mitigate such effects.

Mining companies that plan to accommodate changes in climate, including the proposed CO_2 regulations, and identify potential benefits that can be achieved could be perceived as being proactive and be received more favorably by the local communities. Mining companies will also be the source for raw materials needed to produce alternative forms of energy (e.g., solar panels,

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USE OF BIOCHAR TO REDUCE ACID ROCK DRAINAGE AND STORE CO_2

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Biochar is charcoal created by pyrolysis (heating plant and other organic material in the absence of oxygen) of biomass and can be used as a soil amendment and fertilizer at mine sites. The biomass can be dead wood from forest fires or insect damage, or wastes from poultry farms. Biochar adds carbon and other nutrients to the soil, improves the soil's ability to hold water, and is suited for low-pH, low-humus soils. Biochar added to mine wastes can reduce acid drainage, improve water quality, and increase plant growth. In addition, biochar may actually store CO_2, N_2O, and CH_4 (i.e., greenhouse gases). Field tests are under way at mine sites in the Rocky Mountains and in West Virginia to determine the effectiveness of biochar as a soil amendment in mine reclamation (West Virginia University 2011; IBI 2013; Harley 2010).

wind turbines, electric/hybrid cars). Nuclear power, which requires the mining of uranium, could be a larger component of the local and national power supply in the future. Process tailings facilities in New Mexico and Arizona are currently the sites of large solar panel arrays that supply electricity to the surrounding communities, thereby providing for a sustainable postmining use. Mine wastes and process tailings could be potentially effective in absorbing CO_2 and storing carbon; the use of biochar is one example (see Biochar sidebar; Harley 2010). As research progresses, other benefits are likely to be realized.

The effects of changing climate are being researched throughout the world (Lemmen et al. 2008; Arctic North Consulting 2009; Geyer and Wasilewski 2009; Nelson and Schuchard, n.d.). However, mining operations today are designed in respect to historical climate changes instead of projected future climate changes.

Mines may see changes in both weather and climate (Nelson and Schuchard, n.d.). Some areas could see more frequent weather changes than the mine is now facing or even new changes in the weather, such as excessive rainfall. Excessive rainfall could lead to increases and decreases in ARD and metals concentrations in waters, in both MIW and waters from adjacent mineralized but unmined areas. Sedimentation could increase as rainfall increases. After prolonged droughts, sudden increases in ARD and metals concentrations would occur during the early part of storm events, sometimes called *first flush* (Nordstrom 2008). However, as weather changes in other areas, mines could face events not always foreseen nor engineered to withstand, such as flooding, erosion (both wind and water), landslides, heat waves, overflowing of impacted water containment ponds, debris flows, increased snowfall and freezing rain, avalanches, increased wildfires, and amplified winds that can result in increased dust emissions. Landslides, mudslides, and flooding can occur after wildfires. Changes can be dramatic even between adjacent water basins (Hanson et al. 2006). Increased temperatures could result in increased evaporation from tailings facilities. Heat waves could stress workers and equipment, possibly resulting in production delays. Hotter and drier climates can increase wildfires that could threaten mines and processing sites. Existing mining infrastructures are not always designed for extreme changes in weather conditions. All of these effects could impact water quality. Water supplies could be affected by short-term weather and long-term climate changes. New technologies are needed to increase reuse and reduce water use in mines and processing facilities. Also, weather and ultimately climate change could affect migration of animals. All of these factors should be considered in the mine and processing plans; the mine plans should at least be flexible enough to incorporate changes if these factors play a role.

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8.7 SAMPLING AND MONITORING IN OTHER COUNTRIES

Water quality impacts by mining activities is a major concern in most countries in the world and has resulted in various regulatory programs by government agencies. Sampling and monitoring plans, although not always specifically required by the regulatory agencies throughout the world, are fundamental to achieving desired results. Most international mining companies use similar sampling and monitoring programs in their mines and processing facilities regardless of their location, which results in environmental programs exceeding national requirements at many international mines.

Stakeholder engagement and respect for indigenous cultures are becoming increasingly important throughout the world (Mineral Resources Group 2009). International organizations such as the World Bank, International Finance Corporation, and World Health Organization can provide additional guidance.

A description of the regulatory process and sampling and monitoring plans throughout the world is beyond the scope of this handbook. However, a summary of the regulatory requirements for the major countries can be found in the *Global Acid Rock Drainage Guide* (INAP 2009). Some common themes worldwide include the following (INAP 2009):

- Environmental management plans are typically formed as part of the authorization process and can describe how the effects will be managed during the mining operational phase.
- The prediction of conditions and planning for mine closure should be determined, which could require numerous sampling and monitoring plans.
- Ongoing monitoring, review, and continuous improvement, particularly for ARD, are recommended.

Many areas of the world have summarized requirements for mine reclamation and avoiding water quality risks. Australia recently enacted new regulations. Price and Errington (1998) and Mine Environment Neutral Drainage (MEND 2005) provide a comprehensive list of the information requirements and factors to consider regarding ARD management in mines operating in Canada, and Campbell (2004) discusses regulations in Africa. The European Commission is responsible for developing policies and proposals for regulations of mines, under which the European Union member states will operate (INAP 2009). In Australia, a mining and rehabilitation program (MARP) must be prepared that documents risks and provides specific and detailed information on management and control measures for those risks (Mineral Resources Group 2009, 2010). The MARP includes a complete description of the mine site and planned operations, documentation of consultation with communities and indigenous people, description and documentation of risks, and management of those risks.

8.8 FUTURE RESEARCH NEEDS AND TOPICS

As in most studies, more questions are realized:

- How do we better measure quantitative mineralogy?
- How can supergene minerals originally in the mined rock be differentiated from secondary minerals originating from weathering in a mine-waste storage facility?
- What are the impacts from ARD or MIW from industrial minerals mines? (An important step would be to perform a literature search of existing data to identify sources of information needed to quantify impacts on water from industrial minerals mines in the United States in terms of stream miles, watershed acreage, number of sites, and groundwater acreage. To accomplish this, a consensus-reviewed report could be produced that can be used

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as the basis for a request for proposal to carry out the study on the impacts of historical mined lands on water resources.)

- What are the risks of mine storage facilities to produce ARD? (Sampling and testing techniques need to be further developed to address the hydrologic and geochemical characterization of mine storage facilities.)
- What do differences in stable isotopes in different types of waters mean, and can isotopes characterize mixing and mechanisms of mixing?
- What types, formats, content, and inventories of database management are needed, and how should they be managed?
- What is the effect of MIW from sand and gravel/aggregate pits?
- What is the geochemistry/mineralogy of cast-off products from industrial activities and how do they affect MIW (i.e., cement, dredging, mine wastes, organic wastes, fines)?
- How do MIW and ARD affect sediments, sediment transport, and contamination?
- How do mine and forest fires affect water quality at mine and processing sites?
- How do blowouts affect water quality?
- How do we deal with advancements in technologies, new technologies, and integration of technologies involving water quality at mine sites?

In addition, the following research items reflect a future need:

- Development of improved selective/partial extraction techniques to identify and quantify mineralogy and its effect on water quality.
- Better methods for the long-term monitoring of remediation sites.
- Case studies of remediation and models of drainage quality at mine sites.
- Development of improved methods to model mine hydrology.
- Use of stable and radiometric isotopic data in drainage systems, including groundwater.

8.9 REFERENCES

Arctic North Consulting. 2009. *Climate Change and Canadian Mining: Opportunities for Adaptation*. Report prepared for the David Suzuki Foundation. www.davidsuzuki.org/publications/downloads/2009/Climate_Change_And_Canadian_Mining.pdf. Accessed 8/23/13.

Azam, S., and Li, Q. 2010. *Tailings Dam Failures: A Review of the Last One Hundred Years*. Waste Geotechnics. www.infomine.com/publications/docs/Azam2010.pdf. Accessed 8/23/13.

Blight, G.E., and Fourie, A.B. 2003. *A Review of Catastrophic Flow Failures of Deposits of Mine Waste and Municipal Refuse*. Report. ww.unina2.it/flows2003/flows2003/articoli/G.E.%20BLIGHT%20&%20A.B.%20FOURIE.pdf. Accessed 11/20/13.

Boston.com. 2010. The big picture: A flood of toxic sludge. October 6. www.boston.com/bigpicture/2010/10/a_flood_of_toxic_sludge.html. Accessed 9/16/13.

Campbell, B., ed. 2004. Regulating mining in Africa: For whose benefit? Nordiska Afrikainstitutet, Discussion Paper 26. http://nai.diva-portal.org/smash/record.jsf?searchId=1&pid=diva2:240515. Accessed 8/23/13.

Davies, M.P. 2002. *Tailings Impoundment Failures: Are Geotechnical Engineers Listening?* Infomine report. www.infomine.com/publications/docs/Davies2002.pdf. Accessed 8/23/13.

Davies, M., Martin, T., and Lighthall, P. 2002. *Mine Tailings Dams: When Things Go Wrong*. Infomine report. www.infomine.com/publications/docs/Davies2002d.pdf. Accessed 8/23/13.

Dewan, S. 2008. Tennessee ash flood larger than initial estimate. *New York Times*. December 26. www.nytimes.com/2008/12/27/us/27sludge.html. Accessed 9/16/13.

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Environmental Law Alliance Worldwide. 2010. *Guidebook for Evaluating Mining Project EIAs*. Eugene, OR: Environmental Law Alliance Worldwide. www.elaw.org/files/mining-eia-guidebook/Full-Guidebook.pdf. Accessed 8/23/13.

Geyer, T., and Wasilewski, C. 2009. Climate change risk and impact assessment for global diversified mining group. SME Preprint 09-015. Littleton, CO: SME.

Guerra, I. 2010. Tailings dam collapses in Huancavelica, Peru. *Mines and Communities*. July 4. www.minesandcommunities.org/article.php?a=10212. Accessed 9/16/13.

Hanson, R.T., Dettinger, M.D., and Newhouse, M.W. 2006. Relations between climatic variability and hydrologic time series from four alluvial basins across the southwestern United States. *Hydrogeol. J.* 14:1122–1146.

Harley, A.D. 2010. Biochar activities in the Rocky Mountains with respect to mine reclamation (abstract). Geological Society of America, *Abstr. Prog*. 42(5):158.

Hutchinson, J.N. 1988. General Report. Morphological and geotechnical parameters of landslides in relation to geology and hydrogeology. 5th International Symposium on Landslides, Switzerland, pp. 3–35.

IBI (International Biochar Initiative). 2013. Learn more about biochar. www.biochar-international.org/. Accessed 9/16/13.

INAP (International Network for Acid Prevention). 2009. *Global Acid Rock Drainage Guide* (GARD Guide). www.gardguide.com/index.php/Main_Page. Accessed 8/23/13.

Klohn, E.J. 1995. Tailings dams: Their evolution to major hydraulic structures and the importance of their safety. Keynote address to Canadian Dam Safety Association Conference, Banff, Alberta, October.

Lemmen, D.S., Warren, F.J., Lacroix, J., and Bush, E., eds. 2008. *From Impacts to Adaptation: Canada in a Changing Climate 2007*. Ottawa, ON: Government of Canada. www.nrcan.gc.ca/earth-sciences/climate-change/community-adaptation/assessments/132. Accessed on 9/16/13.

Mayoral, J.M., and Romo, M.P. 2008. Geo-science environmental aspects affecting tailings dams failures. *Am. J. Environ. Sci.* 4:212–222.

McLemore, V.T., Fakhimi, A., van Zyl, D., Ayakwah, G.F., Boakye, K., Anim, K., Ennin, F., Felli, P., Fredlund, D., Gutierrez, L.A.F., Nunoo, S., Tachie-Menson, S., and Viterbo, V.C. 2009. *Literature Review of Other Rock Piles: Characterization, Weathering, and Stability*. Open-File Report 517. New Mexico Bureau of Geology and Mineral Resources. http://geoinfo.nmt.edu/publications/openfile/details.cfml?Volume=517. Accessed 9/16/13

MEND (Mine Environment Neutral Drainage). 2005. *List of Potential Information Requirements in Metal Leaching/Acid Rock Drainage (ML/ARD) Assessment and Mitigation Work*. MEND Report 5.10E. www.mend-nedem.org/reports/files/5.10E.pdf. Accessed 9/16/13.

Mineral Resources Group. 2009. *Guidelines for Miners: Mining Approval Processes in South Australia*. Government of South Australia, Minerals Regulatory Guidelines MG1, version 1.9. www.pir.sa.gov.au/__data/assets/pdf_file/0011/94952/mg01_mining_approvals_v1.9.pdf.

Mineral Resources Group. 2010. *Guidelines for Miners: Preparation of a Mining and Rehabilitation Program (MARP) for Extractive Mineral Operations in South Australia*. Government of South Australia. Minerals Regulatory Guidelines MG1, version 1.9. www.pir.sa.gov.au/__data/assets/pdf_file/0006/71673/MG06_prepn_marp_extractives_v1.9.pdf. Accessed 9/16/13.

Nelson, J., and Schuchard, R. n.d. Adapting to climate change: A guide for the mining industry. BSR industry series. www.bsr.org/reports/BSR_Climate_Adaptation_Issue_Brief_Mining.pdf. Accessed 8/20/13.

Neuendorf, K.K.E., Mehl, J.P., Jr., and Jackson, J.A. 2005. *Glossary of Geology*, 5th ed. Alexandria, VA: American Geological Institute.

Nordstrom, D.K. 2008. Acid rock drainage and climate change. *J. Geochem. Explor*. 100:97–104.

Norgate, T.E., and Rankin W.J. 2000. Life cycle assessment of copper and nickel production. In *Proceedings, Minprex 2000, International Conference on Minerals Processing and Extractive Metallurgy*. pp. 133–138. www.minerals.csiro.au/sd/CSIRO_Paper_LCA_CuNi.htm. Accessed 9/16/13.

OCCIAR (Ontario Centre for Climate Impacts and Adaptation Resources). n.d. Mining: In a changing climate. Fact sheet. www.climateontario.ca/doc/factsheets/Mining%20Factsheet%20--%20Final.pdf. Accessed 8/20/13.

Pernichele, A.D., and Kahle, M.B. 1971. Stability of waste dumps at Kennecott's Bingham Canyon mine. *AIME Trans*. 250:363–367.

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Price, W.A., and J.C. Errington. 1998. *Guidelines for Metal Leaching and Acid Rock Drainage at Mine Sites in British Columbia.* Ministry of Energy and Mines, August.

Retallack, G.J. 2009. Greenhouse crises of the past 300 million years: *Geolog. Soc. Am. Bull*. 121:1441–1445.

Rico, M., Benito, G., Salgueiro, A.R., Diez-Herrero, A., and Pereira, H.G. 2008. Reported tailings dam failures: A review of the European incidents in the worldwide context. *J. Hazard. Mater*. 152:846–852.

Robertson, A., and Skermer, N.A. 1988. Design considerations for the long term stability of mine wastes. First International Environmental Workshop, Darwin.

Trenberth, K.E. 1999. Conceptual framework for changes of extremes of the hydrological cycle with climate change. *Climate Change* 42:327–339.

USCOLD (U.S. National Committee on Large Dams). 1994. *Tailings Dam Incidents*. Report prepared by the USCOLD Committee on Tailings Dams, November.

West Virginia University. 2011. Research highlights. http://research.wvu.edu/wvu_research_highlights/2011/7/25/from-farm-to-fuel-to-fields. Accessed 7/9/11.

Whiting, D.L. 1981. Surface and groundwater pollution potential. In M.K. McCarter, ed. *Design of Non-impounding Mine Waste Dumps*. New York: AIME. pp. 90–98.

WISE (World Information Service on Energy). 2013. Uranium project: Chronology of major tailings dam failures. www.wise-uranium.org/mdaf.html. Accessed 9/16/13.

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APPENDIX 1—SELECTED ONLINE RESOURCES FOR SAMPLING, MONITORING, AND ANALYTICAL CHEMISTRY METHODS

Compiled by Kathleen S. Smith, Carol C. Russell, and Virginia T. McLemore

Reviewed by Mary P. Goldade and Thomas Moyer

Series Title: Management Technologies for Metal Mining Influenced Water

Volume 6: Sampling and Monitoring for the Mine Life Cycle

Editors: Virginia T. McLemore, Kathleen S. Smith, and Carol C. Russell

Publisher: Society for Mining, Metallurgy & Exploration Inc., Englewood, Colorado

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Any use of trade, firm, or product names is for descriptive purposes only and does not imply endorsement by the U.S. Government. The links in this appendix were active as of May 2013. This appendix lists selected online resources in approximate order of relevance and is not an exhaustive list of online resources. To the extent this appendix mentions or discusses statutory or regulatory authorities, it does so for informational purposes only. Please note that regulatory requirements may be more specific than those implied in these websites, and the reader should consult the appropriate statutes or regulations for complete information. Please note that ASTM methods are listed in a separate appendix.

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GENERAL WEBSITES (all accessed 9/17/13)

Acid Drainage Technology Initiative Metal-Mining Sector (ADTI-MMS):
http://ese.mines.edu/adti/

Acid Drainage Technology Initiative Coal-Mining Sector (ADTI-CMS):
www.aciddrainage.com/

Mine Environment Neutral Drainage (MEND) Program:
www.mend-nedem.org

International Network for Acid Prevention (INAP) Global Acid Rock Drainage (GARD) Guide:
www.gardguide.com/
The GARD Guide deals with the prediction, prevention, and management of drainage produced from sulfide mineral oxidation. It is intended as a state-of-the-art summary of the best practices and technology to assist mine operators and regulators to address issues related to sulfide mineral oxidation.

EnviroMine: Environmental Technology in Mining:
http://technology.infomine.com/enviromine/
EnviroMine provides information on environmental issues related to mining, such as acid mine drainage, water and soil contamination, responsible and sustainable mining, and mine closures. Access to some information requires membership.
http://technology.infomine.com/enviromine/ard/home.htm
This InfoMine Acid Rock Drainage website provides a technical resource for information about acid rock drainage fundamentals, prediction, treatment and management, and case studies.

National Environmental Methods Index (NEMI):
www.nemi.gov
NEMI is maintained under the direction of the Methods and Data Comparability Board, a partnership of water-quality experts from Federal agencies, States, municipalities, industry, and private organizations. The Methods Board is chartered under the National Water Quality Monitoring Council, whose mission since its charter in May 1997 is to coordinate and provide guidance on implementation of a voluntary, integrated, nationwide monitoring strategy. The site allows the user to search for a variety of chemical, microbiological, biological, toxicological, and physical methods.

Environmental Monitoring and Measurement Advisor (EMMA):
www.emma-expertsystem.com/
EMMA is an expert system (interactive software) for project managers, administrators, and others who use or procure laboratory services for environmental analyses. It is used to plan improved and cost-effective environmental monitoring projects. It is also a highly effective teaching aid for instructors and students and was funded in part by the National Science Foundation (NSF). EMMA allows the user to tailor plans to meet specific project needs by

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considering the physical and chemical characteristics of the sampling site and target analytes, desired data quality, available budget, and objectives.

National Water Quality Monitoring Council (NWQMC):
http://acwi.gov/monitoring/
The NWQMC provides a national forum for coordination of consistent and scientifically defensible methods and strategies to improve water quality monitoring, assessment and reporting. The NWQMC Methods and Data Comparability Board provides a forum for exploring, evaluating, and promoting methods that facilitate collaboration and further comparability between water monitoring programs.

North American Land Cover Monitoring System (NALCMS):
www.cec.org/Page.asp?PageID=924&SiteNodeID=565
The NALCMS was developed to depict information about land cover and land cover change in a seamless, consistent, and automated way across North America at regular intervals. The NALCMS is a collaborative effort of government agencies in Canada, Mexico and the United States coordinated by the Commission for Environmental Cooperation's North American Environmental Atlas initiative. Based upon the analysis of images collected remotely by satellites, the NALCMS seeks to be a dynamic land cover change monitoring system and a vital long-term information source for users of land cover change products across North America and can be used for a variety of applications including carbon sequestration analysis, wildlife habitat mapping, ecosystem monitoring, environmental planning, weather forecasting, water quality assessments, and biofuels production potential.

New Mexico Bureau of Geology and Mineral Resources Decision Makers Field Guides:
http://geoinfo.nmt.edu/publications/guides/decisionmakers/home.cfm
The purpose of these field guides is to present decision makers with the opportunity to learn first-hand about geological problems, opportunities, and potential solutions from top experts.

Mining and the Environment Information, Laurentian University:
http://142.51.14.1/Laurentian/Home/Departments/Library/Finding_information/Mining_and_the_Environment_Information/Mining+and+the+Environment+Information.htm?Laurentian_Lang=en-CA
The Mining Environment Database provides references and abstracts to over 20,000 journal articles, books, and government reports dealing with abandoned mines, acid mine drainage, and land reclamation.

National Institute of Standards and Technology (NIST) Standard Reference Materials:
http://ts.nist.gov/measurementservices/referencematerials/

National Institute for Occupational Safety and Health (NIOSH) Office of Mine Safety and Health Research:
www.cdc.gov/niosh/mining/topics/index.html

Mine Safety and Health Administration (MSHA):
www.msha.gov/

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Office of Surface Mining:
www.osm.gov/

Cleanup/Screening Levels For Hazardous Waste Sites:
www.cleanuplevels.com/

U.S. ENVIRONMENTAL PROTECTION AGENCY WEBSITES (all accessed 9/16/13)

Framework for Metals Risk Assessment:
www.epa.gov/raf/metalsframework/
The Framework for Metals Risk Assessment, released on March 8, 2007, outlines key principles about metals and describes how they should be considered in conducting human health and ecological risk assessments. The Framework seeks to advance our understanding of the impact of metals in a consistent manner across the USEPA's programs since inorganic metals and metal compounds present unique issues for risk assessors.

Abandoned Mine Site Characterization and Cleanup Handbook:
www.epa.gov/superfund/policy/remedy/pdfs/amscch.pdf

USEPA and Hardrock Mining: A Source Book for Industry in the Northwest and Alaska:
http://yosemite.epa.gov/R10/WATER.NSF/Sole+Source+Aquifers/hardrockmining

USEPA Region 4, Science and Ecosystem Support Division (SESD) Website:
www2.epa.gov/aboutepa/about-region-4s-science-and-ecosystem-support-division-sesd
The tabs on this website link to a number of information sources about a variety of topics.

Measurement and Monitoring Technologies for the 21st Century (21M2; CLU-IN):
www.clu-in.org/programs/21m2/litsearch.cfm
The Measurement and Monitoring Initiative Literature Search System is updated quarterly and incorporates citations and descriptions from literature related to the identified USEPA Office of Solid Waste and Emergency Response (OSWER) needs areas and includes commercially available monitoring and measurement material plus government-sponsored small business research projects (SBIR) and other awards.

USEPA Target Compounds and Analytes:
www.epa.gov/superfund/programs/clp/target.htm
The USEPA Contract Laboratory Program (CLP) Target Compound and Target Analyte Lists were originally derived from the USEPA Priority Pollutant List. In the years since the inception of the CLP, compounds and analytes have been added to, and deleted from this list based on advances in analytical methods, evaluation of method performance data, and the needs of the Superfund program. The target compounds and analytes, along with their corresponding Contract Required Quantitation Limits are listed.

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USEPA Test Method Collections:
www.epa.gov/osa/fem/methcollectns.htm
This site provides a compilation of test methods, which are approved procedures for measuring the presence and concentration of physical and chemical pollutants, evaluating properties such as toxic properties of chemical substances or measuring the effects of substances under various conditions.

USEPA Hazardous Waste Test Methods (SW-846 On-line):
www.epa.gov/waste/hazard/testmethods/sw846/online/index.htm
This site provides an online version of the USEPA "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," also known as SW-846.

USEPA Field-based Analytical Methods:
www.epa.gov/superfund/programs/dfa/fldmeth.htm
Field-based analytical methods are one of the key tools of dynamic field activities because they provide the data that is used for on-site decision making. They are a broad category of analytical methods that can be applied on the site of sample collection activities. They include methods that can be used outdoors with handheld equipment, as well as more rigorous methods that require the controlled environments of a mobile laboratory. Some of the most common methods are portable gas chromatography (GC), immunoassay test kits (IA), X-ray fluorescence (XRF), and gas chromatography/mass spectrometry (GC/MS).

USEPA Clean Water Act Analytical Methods:
www.epa.gov/waterscience/methods/
USEPA publishes laboratory analytical methods that are used by industries and municipalities to analyze the chemical, physical and biological components of wastewater and other environmental samples that are required by regulations under the authority of the Clean Water Act (CWA). Most of these methods are published as regulations in the Code of Federal Regulations (CFR) in Title 40 Part 136.

USEPA Drinking Water Act Analytical Methods:
www.epa.gov/safewater/methods/analyticalmethods.html
Water systems must use USEPA approved analytical methods when analyzing samples to meet federal monitoring requirements or to demonstrate compliance with drinking-water regulations. Approved methods are listed in the Code of Federal Regulations after publication in a final rule or as part of an expedited approval. Approved methods are developed by USEPA, other government agencies, universities, consensus methods organizations, water laboratories, and instrument manufacturers.

USEPA Benthic Macroinvertebrate Protocols:
http://water.epa.gov/scitech/monitoring/rsl/bioassessment/ch07main.cfm

USEPA Agency-wide Quality System Documents:
www.epa.gov/QUALITY/qa_docs.html

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USEPA Water Quality Standards Database:
http://water.epa.gov/scitech/swguidance/standards/wqshome_index.cfm
The USEPA National Water Quality Standards Database (WQSDB) provides access to U.S. EPA and state water-quality standards information in text, tables, and maps.

USEPA Drinking Water Standards and Risk Management:
http://water.epa.gov/drink/standardsriskmanagement.cfm

USEPA Aquatic Life Criteria:
www.epa.gov/waterscience/criteria/aqlife/

USEPA ECOTOX Database:
http://cfpub.epa.gov/ecotox/
The USEPA ECOTOX (ECOTOXicology) database provides single chemical toxicity information for aquatic and terrestrial life.

USEPA Monitoring and Assessing Water Quality:
http://water.epa.gov/type/watersheds/monitoring/index.cfm
This website provides a variety of resources that relate to monitoring and assessing water quality.

USEPA Ecological Risk Characterization, documents and links:
www.epa.gov/oswer/riskassessment/eco_characterization.htm

USEPA Superfund Risk Assessment:
www.epa.gov/oswer/riskassessment/risk_superfund.htm
This website is intended primarily to be a resource for USEPA, State, other federal agency, and contractor risk assessors and toxicologists. It provides guidance, tools and databases that might be useful in preparing human health and ecological risk assessments.

USEPA Abandoned Mine Lands (AMLs):
www.epa.gov/aml/
This website identifies USEPA's resources related to the range of environmental risks and challenges from AMLs as well as risk management approaches.

Triad Resource Center:
www.triadcentral.org/
Triad is a Federal/State interagency partnership. The Triad is an innovative approach to decision-making for hazardous waste site characterization and remediation. The Triad approach proactively exploits new characterization and treatment tools, using work strategies developed by innovative and successful site professionals. The Triad Resource Center provides the information hazardous waste site managers and cleanup practitioners need to implement the Triad effectively.

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U.S. GEOLOGICAL SURVEY WEBSITES
(all accessed 9/16/13)

USGS National Field Manual for the Collection of Water-Quality Data:
http://water.usgs.gov/owq/FieldManual/
The USGS National Field Manual is designed to achieve consistency in the scientific methods and procedures used, to document the methods and procedures, and to maintain technical expertise. USGS field personnel use this manual to ensure that the data collected are of the quality required to fulfill the USGS mission. These data are used for environmental and resource assessments by the USGS, other government agencies and scientific organizations, and the general public.

USGS Techniques of Water-Resources Investigations Reports:
http://pubs.usgs.gov/twri/

USGS Publications Warehouse:
http://pubs.er.usgs.gov/
The *Publications Warehouse* site allows searches for USGS publications by author, title, year, or product type. Advanced search options include searching for text in title. Eventually, all products by USGS authors will be available through the *Publications Warehouse*.

USGS Water Data for the Nation:
http://waterdata.usgs.gov/nwis

USGS Analytical Methods for Chemical Analysis of Geologic and Other Materials:
http://pubs.usgs.gov/of/2002/ofr-02-0223/
This manual presents validated analytical methods used by the USGS Mineral Resources Program. The manual is intended primarily for use by laboratory scientists. The analytical methods are written in a step-by-step approach so that they may be used as a training tool and provide detailed documentation of the procedures for quality assurance.

USGS Standard Reference Materials:
Powdered standards:
http://minerals.cr.usgs.gov/geo_chem_stand/
Liquid standards:
http://bqs.usgs.gov/srs/
These sites contain lists of available reference materials, analysis information, certificates, and useful links.

USGS National Water-Quality Assessment Program (NAWQA):
http://water.usgs.gov/nawqa/
http://water.usgs.gov/nawqa/protocols/ (Detailed protocols)
The USGS National Water-Quality Assessment Program (NAWQA) provides an understanding of water-quality conditions; whether conditions are getting better or worse over time; and how natural features and human activities affect those conditions. Regional and national assessments are possible because of a consistent study design and uniform methods of data collection and

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analysis. Monitoring data are integrated with geographic information on hydrological characteristics, land use, and other landscape features in models to extend water-quality understanding to unmonitored areas.

USGS Toxic Substances Hydrology Program:
http://toxics.usgs.gov/
The USGS Toxic Substances Hydrology Program provides objective scientific information on environmental contamination to improve characterization and management of contaminated sites, to protect human and environmental health, and to reduce potential future contamination problems.

USGS Mineral Resources Program:
http://minerals.usgs.gov/
The USGS Mineral Resources Program (MRP) provides scientific information for objective resource assessments and unbiased research results on mineral potential, production, consumption, and environmental effects.

USGS Geophysical Technology Transfer:
http://water.usgs.gov/ogw/bgas/g2t.html
A primary goal of the USGS Office of Groundwater, Branch of Geophysics (OGW–BG) mission is to transfer geophysical knowledge and experience. The goal of this Geophysical Technology Transfer (G2T) section is to provide USGS scientists with web-based resources that will support the development and implementation of Earth science studies.

USGS Abandoned Mine Lands Initiative:
http://amli.usgs.gov/
This website describes the USGS Abandoned Mine Lands Initiative work.

USGS Mine Drainage Activities:
http://mine-drainage.usgs.gov/

INTERNATIONAL WEBSITES (all accessed 9/16/13)

Mine Environment Neutral Drainage (MEND):
www.mend-nedem.org

Natural Resources Canada Minerals and Metals:
www.nrcan.gc.ca/minerals-metals/home

Natural Resources Canada Certified Reference Materials:
www.nrcan.gc.ca/mms-smm/tect-tech/crm-mrc-eng.htm
Natural Resources Canada's Canadian Certified Reference Materials Project prepares and certifies reference materials principally for analytical laboratories in the mineral, metallurgical, Earth science, and environmental industries.

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National Orphaned/Abandoned Mines Initiative (NOAMI) – Canada:
www.abandoned-mines.org/home-e.htm

International Network for Acid Prevention (INAP):
www.inap.com.au/
The International Network for Acid Prevention is an international organization of mineral resource companies dedicated to the responsible management of reducing liabilities associated with sulfide mine materials.

International Council on Mining and Metals (ICMM):
www.icmm.com/

International Mine Water Association (IMWA):
www.imwa.info/
The International Mine Water Association (IMWA) is a multidisciplinary expert organization that links experts of various branches of natural sciences and technology.

Sustainable Minerals Institute (SMI):
http://www.smi.uq.edu.au/
www.jktech.com.au/professional-development

Australian Mineral Industries Research Association (AMIRA):
www.amira.com.au/

Euromines: European Association of Mining Industries:
www.euromines.org/
Euromines provides an interface between the European extractive industry and the European authorities and international or intergovernmental bodies.

Partnership for Acid Drainage Remediation in Europe (PADRE):
www.padre.imwa.info/

British Columbia Ministry of Energy, Mines, and Natural Gas, Mineral Exploration and Mining:
www.empr.gov.bc.ca/Mining/Pages/default.aspx

Mitigating the environmental impact of mining waste (MiMi):
http://www.mistra.org/en/mistra/research/completed-research/mimi.html

Chinese Network for Acid Mine Drainage (CNAMD):
www.cnamd.net/en/

South American Network for Acid Prevention (SANAP):
http://ingenieria.uchile.cl/constituci_n_del_cap_tulo_chileno_de_sanap_south_american_network_for_acid_prevention

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South African Water Research Commission:
www.wrc.org.za

South African Department of Water Affairs:
www.dwaf.gov.za

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APPENDIX 2—SUMMARY OF SELECTED ASTM METHODS

Compiled by Samuel Nunoo, Frederick Ennin, Kojo Anim, and Virginia T. McLemore

Reviewed by Jeffrey A. Farrar and William Lipps

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DISCLAIMER:
This purpose of this appendix is to summarize selected ASTM methods related to sampling and monitoring of MIW. The ASTM methods can be obtained from www.astm.org (accessed May 2013). When using ASTM methods it is recommended that the number and date of the reference be cited. ASTM methods are periodically updated to reflect current changes. Please note that the method descriptions do not purport to address safety concerns, if any, associated with the method. It is the responsibility of the user of the method to establish appropriate health and safety practices and to determine the applicability of regulatory limitations prior to use. Please also note that regulatory requirements may be more specific than those implied in these method summaries. Any use of trade, firm, or product names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

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ASTM C117: Standard Test Method for Materials Finer than 75-µm (No. 200) Sieve in Mineral Aggregates by Washing
This test method covers the determination of the amount of material finer than a 75-µm (No. 200) sieve in aggregate by washing. Clay particles and other aggregate particles that are dispersed by the wash water, as well as water-soluble materials, will be removed from the aggregate during the test.

ASTM D420: Standard Guide to Site Characterization for Engineering, Design, and Construction Purposes
This guide refers to ASTM methods by which soil, rock, and groundwater conditions may be determined. The objective of the investigation should be to identify and locate, both horizontally and vertically, significant soil and rock types and groundwater conditions present within a given site area and to establish the characteristics of the subsurface materials by sampling or in situ testing, or both.

ASTM D422: Standard Test Method for Particle-Size Analysis of Soils
This test method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75 µm (retained on the No. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75 µm is determined by a sedimentation process, using a hydrometer to secure the necessary data (Note 1 and Note 2).

ASTM D421: Standard Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants
This practice covers the dry preparation of soil samples as received from the field for particle-size analysis and the determination of the soil constants.
This practice can be used to prepare samples for particle-size and plasticity tests where it is desired to determine test values on air-dried samples, or where it is known that air drying does not have an effect on test results relative to samples prepared in accordance with Practice D 2217.

ASTM D425: Standard Test Method for Centrifuge Moisture Equivalent of Soils
This test method covers the determination of the moisture equivalent of soil in the laboratory by means of a centrifuge technique.

ASTM D427: Test Method for Shrinkage Factors of Soils by the Mercury Method
This test method provides a procedure for obtaining the data which are used to calculate the shrinkage limit and the shrinkage ratio.

ASTM D558: Standard Test Methods for Moisture-Density Relations of Soil-Cement Mixtures
These methods cover the determination of the relationship between the water content and the density of soil-cement mixtures when compacted before cement hydration as prescribed.

ASTM D559: Standard Test Methods for Wetting and Drying Compacted Soil-Cement Mixtures

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These test methods cover procedures for determining the soil-cement losses, water content changes, and volume changes (swell and shrinkage) produced by repeated wetting and drying of hardened soil-cement specimens. The specimens are compacted in a mold, before cement hydration, to maximum density at optimum moisture content using the compaction procedure described in Test Methods D 558.

ASTM D560: Standard Test Methods for Freezing and Thawing Compacted Soil-Cement Mixtures

These test methods cover procedures for determining the soil-cement losses, moisture changes, and volume changes (swell and shrinkage) produced by repeated freezing and thawing of hardened soil-cement specimens. The specimens are compacted in a mold, before cement hydration, to maximum density at optimum moisture content using the compaction procedure described in Test Methods D 558.

ASTM D653: Standard Terminology Relating to Soil, Rock, and Contained Fluids

These definitions apply to many terms found in the Terminology section of standards of ASTM Committee D18.

ASTM D698: Standard Test Methods for Laboratory Compaction Characteristics of Soil Using Standard Effort (12,400 ft-lbf/ft^3 (600 kN-m/m^3))

This test method covers laboratory compaction procedures used to determine the relationship between water content and dry unit weight of soils (compaction curve) compacted in a 4 or 6-in. (101.6 or 152.4-mm) diameter mold with a 5.5-lbf (24.4-N) rammer dropped from a height of 12 in. (305 mm) producing a compactive effort of 12,400 ft-lbf/ft^3 (600 kN-m/m^3).

ASTM D806: Standard Test Method for Cement Content of Hardened Soil-Cement Mixtures

This test method covers the determination by chemical analysis of cement content of hardened soil-cement mixtures.

This test method is based on determination by chemical analysis of the calcium oxide (CaO) content of the sample. The method may not be applicable to soil-cement materials containing soils or aggregates which yield significant amounts of dissolved calcium oxide (CaO) under the conditions of the test.

ASTM D854: Standard Test Methods for Specific Gravity of Soil Solids by Water Pycnometer

These test methods cover the determination of the specific gravity of soil solids that pass the 4.75-mm (No. 4) sieve, by means of a water pycnometer. When the soil contains particles larger than the 4.75-mm sieve, Test Method C 127 shall be used for the soil solids retained on the 4.75-mm sieve and these test methods shall be used for the soil solids passing the 4.75-mm sieve.

ASTM D933: Standard Practice for Reporting Results of Examination and Analysis of Water-Formed Deposits

This practice covers the manner in which the results of examination and analysis for inorganic constituents of deposits formed from waters are to be reported. While various practices of reporting the analysis of water-formed deposits are in use, this practice is intended as a rational

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and comprehensive practice for general application. For use in specific industries or individual cases, molecular combinations may be useful and desirable.

ASTM D934: Standard Practices for Identification of Crystalline Compounds in Water-Formed Deposits by X-Ray Diffraction

These practices provide for X-ray diffraction analysis of powdered crystalline compounds in water-formed deposits. Two are given as follows:

	Sections
Practice A—Camera	12 to 21
Practice B—Diffractometer	22 to 30

Both practices yield qualitative identification of crystalline components of water-formed deposits for which X-ray diffraction data are available or can be obtained. Greater difficulty is encountered in identification when the number of crystalline components increases. Amorphous phases cannot be identified without special treatment. Oils, greases, and most organic decomposition products are not identifiable. The sensitivity for a given component varies with a combination of such factors as density, degree of crystallization, particle size, coincidence of strong lines of components and the kind and arrangement of the atoms of the components. Minimum percentages for identification may therefore range from 1 to 40 %.

ASTM D1140: Standard Test Methods for Amount of Material in Soils Finer than No. 200 (75-µm) Sieve

These test methods cover determination of the amount of material finer than a 75-m (No. 200) sieve by washing. Material finer than the 75-µm (No. 200) sieve can be separated from larger particles much more efficiently and completely by wet sieving than with dry sieving. Therefore, when accurate determinations of material finer than 75-µm sieve in soil are desired, this test method is used on the test specimen prior to dry sieving.

ASTM D1411: Standard Test Methods for Water-Soluble Chlorides Present as Admixtures in Graded Aggregate Road Mixes

These test methods are applicable to the determination of water-soluble calcium, magnesium, and sodium chlorides used as admixes in the preparation of graded aggregate road materials.

ASTM D1143: Standard Test Method for Piles under Static Axial Compressive Load (Withdrawn 2006)

This test method covers procedures for testing vertical or batter piles individually or groups of vertical piles to determine response of the pile or pile group to a static compressive load applied axially to the pile or piles within the group. This test method is applicable to all deep foundation units that function in a manner similar to piles regardless of their method of installation. This test method is divided into the following sections:

ASTM D1194: Standard Test Method for Bearing Capacity of Soil for Static Load and Spread Footings (Withdrawn 2003)

This test method covers estimation of the bearing capacity of soil in place by means of field loading tests. This test method can be used as part of a procedure for soil investigation for

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foundation design. It gives information on the soil only to a depth equal to about two diameters of the bearing plate, and takes into account only part of the effect of time.

ASTM D1241: Standard Specification for Materials for Soil-Aggregate Subbase, Base, and Surface Courses

This specification covers the quality and grading of the following materials for use in the construction of subbase, base, and surface courses: sand-clay mixtures; gravel; stone or slag screenings; sand; crusher-run coarse aggregate consisting of gravel, crushed stone, or slag combined with soil mortar; or any combination of these materials. The requirements are intended to cover only materials having normal or average specific gravity, absorption, and gradation characteristics. Where other materials are to be used, appropriate limits suitable to their use must be specified.

ASTM D1245: Standard Practice for Examination of Water-Formed Deposits by Chemical Microscopy

This practice describes a procedure for the examination of water-formed deposits by means of chemical microscopy. This practice may be used to complement other methods of examination of water-formed deposits as recommended in Practices D 2331 or it may be used alone when no other instrumentation is available or when the sample size is very small. The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

ASTM D1452: Standard Practice for Soil Investigation and Sampling by Auger Borings

This practice covers equipment and procedures for the use of earth augers in shallow geotechnical exploration. This practice does not apply to sectional continuous flight augers.

ASTM D1556: Standard Test Method for Density and Unit Weight of Soil in Place by the Sand-Cone Method

This test method may be used to determine the in-place density and unit weight of soils using a sand cone apparatus.

ASTM D1557: Standard Test Methods for Laboratory Compaction Characteristics of Soil Using Modified Effort (56,000 ft-lbf/ft^3(2,700 kN-m/m^3))

These test methods cover laboratory compaction methods used to determine the relationship between water content and dry unit weight of soils (compaction curve) compacted in a 4- or 6-in. (101.6 or 152.4 mm) diameter mold with a 10-lbf. (44.5-N) rammer dropped from a height of 18 in. (457 mm) producing a compactive effort of 56,000 ft-lbf/ft^3 (2,700 kN-m/m^3).

ASTM D1558: Standard Test Method for Moisture Content Penetration Resistance Relationships of Fine-Grained Soils

This test method is for establishing the moisture-penetration resistance relationships of fine-grained soils as determined by the soil penetrometer.

ASTM D1586: Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils

This test method describes the procedure, generally known as the Standard Penetration Test (SPT), for driving a split-barrel sampler to obtain a representative disturbed soil sample for

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identification purposes, and measure the resistance of the soil to penetration of the sampler. Another method (Test Method D 3550) to drive a split-barrel sampler to obtain a representative soil sample is available but the hammer energy is not standardized.

ASTM D1587: Standard Practice for Thin-Walled Tube Sampling of Soils for Geotechnical Purposes

This practice covers a procedure for using a thin-walled metal tube to recover relatively intact soil samples suitable for laboratory tests of engineering properties, such as strength, compressibility, permeability, and density. Thin-walled tubes used in piston, plug, or rotary-type samplers should comply with Section 6.3 of this practice which describes the thin-walled tubes.
Note 1—This practice does not apply to liners used within the samplers.

ASTM D1632: Standard Practice for Making and Curing Soil-Cement Compression and Flexure Test Specimens in the Laboratory (Withdrawn 2005)

This practice covers the procedure for making and curing compression and flexure test specimens of soil-cement in the laboratory under accurate control of quantities of materials and test conditions.

ASTM D1633: Standard Test Methods for Compressive Strength of Molded Soil-Cement Cylinders

This test method covers the determination of the compressive strength of soil-cement using molded cylinders as test specimens.

ASTM D1634: Standard Test Method for Compressive Strength of Soil-Cement Using Portions of Beams Broken in Flexure (Modified Cube Method)

This test method covers the determination of the compressive strength of soil-cement, using for test specimens portions of beams broken in flexure in accordance with Test Method D 1635.

ASTM D1883: Standard Test Method for CBR (California Bearing Ratio) of Laboratory-Compacted Soils

This test method covers the determination of the CBR (California Bearing Ratio) of pavement subgrade, subbase, and base/course materials from laboratory compacted specimens. The test method is primarily intended for but not limited to, evaluating the strength of cohesive materials having maximum particle sizes less than 3/4 in. (19 mm).

ASTM D1997: Standard Test Method for Laboratory Determination of the Fiber Content of Peat Samples by Dry Mass

This test method covers the laboratory determination of the fiber content of samples of peat (as defined in Classification D 4427) by dry mass. It also may be used for non-peat organic soil materials. Because this test method is simple and requires no sophisticated equipment to perform, it is especially recommended for routine reconnaissance work, where large numbers of samples need to be tested and mineral contents are low.

ASTM D2113: Standard Practice for Rock Core Drilling and Sampling of Rock for Site Investigation

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This practice covers the guidelines, requirements, and procedures for core drilling, coring, and sampling of rock for the purposes of site investigation. The borehole could be vertical, horizontal, or angled. This practice is described in the context of obtaining data for the design, construction, or maintenance of structures, and applies to surface drilling and drilling from adits and exploratory tunnels.

ASTM D2166: Standard Test Method for Unconfined Compressive Strength of Cohesive Soil

This test method covers the determination of the unconfined compressive strength of cohesive soil in the intact, remolded, or reconstituted condition, using strain-controlled application of the axial load. This test method provides an approximate value of the strength of cohesive soils in terms of total stresses.

ASTM D2167: Standard Test Method for Density and Unit Weight of Soil in Place by the Rubber Balloon Method

This test method covers the determination of the in-place density and unit weight of compacted or firmly bonded soil using a rubber balloon apparatus. This test method is suitable for use as a means of acceptance for compacted fill or embankments constructed of fine-grained soils or granular soils without appreciable amounts of rock or coarse material.

ASTM D2168: Standard Test Methods for Calibration of Laboratory Mechanical-Rammer Soil Compactors

These test methods for the calibration of mechanical soil compactors are for use in checking and adjusting mechanical devices used in laboratory compacting of soil and soil-aggregate in accordance with Test Methods D 698, D 1557 and other methods of a similar nature which might specify this method. Calibration for use with one method does not qualify the equipment for use with another method.

The weight of the mechanical rammer is adjusted as described in and in order to provide that the mechanical compactor will produce the same result as the manual compactor.

ASTM D2216: Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

These test methods cover the laboratory determination of the water (moisture) content by mass of soil, rock, and similar materials where the reduction in mass by drying is due to loss of water

ASTM D2217: Standard Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants (Withdrawn 2007)

This practice covers the wet preparation of soil samples as received from the field for particle-size analysis and determination of soil constants.

ASTM D2325: Standard Test Method for Capillary-Moisture Relationships for Coarse- and Medium-Textured Soils by Porous-Plate Apparatus (Withdrawn 2007)

This test method covers the determination of capillary-moisture relationships for coarse- and medium-textured soils as indicated by the soil-moisture tension relations for tensions between 10 and 101 kPa (0.1 and 1 atm). Under equilibrium conditions, moisture tension is defined as the equivalent negative gage pressure, or suction, corresponding to a soil moisture content. This test

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method determines the equilibrium moisture content retained in a soil subjected to a given soil-water tension. This test method is not suitable for very fine-textured soils.

ASTM D2332: Standard Practice for Analysis of Water-Formed Deposits by Wavelength-Dispersive X-Ray Fluorescence
This practice covers X-ray spectrochemical analysis of water-formed deposits. The practice is applicable to the determination of elements of atomic number 11 or higher that are present in significant quantity in the sample (usually above 0.1 %).

ASTM D2434: Standard Test Method for Permeability of Granular Soils (Constant Head)
This test method covers the determination of the coefficient of permeability by a constant-head method for the laminar flow of water through granular soils. The procedure is to establish representative values of the coefficient of permeability of granular soils that may occur in natural deposits as placed in embankments, or when used as base courses under pavements. In order to limit consolidation influences during testing, this procedure is limited to disturbed granular soils containing not more than 10% soil passing the 75-[mu]m (No. 200) sieve.

ASTM D2435: Standard Test Methods for One-Dimensional Consolidation Properties of Soils Using Incremental Loading
This test method covers procedures for determining the magnitude and rate of consolidation of soil when it is restrained laterally and drained axially while subjected to incrementally applied controlled-stress loading.

ASTM D2487: Standard Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)
This practice describes a system for classifying mineral and organo-mineral soils for engineering purposes based on laboratory determination of particle-size characteristics, liquid limit, and plasticity index and shall be used when precise classification is required.

ASTM D2488: Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)
This practice covers procedures for the description of soils for engineering purposes. This practice also describes a procedure for identifying soils, at the option of the user, based on the classification system described in Test Method D 2487. The identification is based on visual examination and manual tests. It must be clearly stated in reporting an identification that it is based on visual-manual procedures.

ASTM D2573: Standard Test Method for Field Vane Shear Test in Cohesive Soil
This test method covers the field vane test in saturated clay and silt soils for determination of undrained shear strength. Knowledge of the nature of the soil in which each vane test is to be made is necessary for assessment of the applicability and interpretation of the test. The test is not applicable for sandy soils which may allow drainage during the test. This test method addresses testing on land and for testing in drill holes or by self drilling or continuous push methods from the ground surface. This method does not address specifically marine testing where special test requirements or variations in equipment may be required. The user is referred to ASTM STP 1014 for additional information on in-place vane shear testing.

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ASTM D2664: Standard Test Method for Triaxial Compressive Strength of Undrained Rock Core Specimens without Pore Pressure Measurements
This test method covers the determination of the strength of cylindrical rock core specimens in an undrained state under triaxial compression loading. The test provides data useful in determining the strength and elastic properties of rock, namely: shear strengths at various lateral pressures, angle of internal friction, (angle of shearing resistance), cohesion intercept, and Young's modulus. It should be observed that this method makes no provision for pore pressure measurements. Thus the strength values determined are in terms of total stress that is not corrected for pore pressures.

ASTM D2844: Standard Test Method for Resistance *R*-Value and Expansion Pressure of Compacted Soils
This test method covers the procedure for testing both treated and untreated laboratory compacted soils or aggregates with the stabilometer and expansion pressure devices to obtain results indicative of performance when placed in the base, subbase, or subgrade of a road subjected to traffic. The values stated in SI units are to be regarded as the standard. However, use of equipment manufactured to be readable in inch-pound units only should not be considered as non-conformance to the test method. Many stabilometers and expansion pressure devices are manufactured to be readable in inch-pound units only.

ASTM D2845: Standard Test Method for Laboratory Determination of Pulse Velocities and Ultrasonic Elastic Constants of Rock
This test method describes equipment and procedures for laboratory measurements of the pulse velocities of compression waves and shear waves in rock and the determination of ultrasonic elastic constants (Note 1) of an isotropic rock or one exhibiting slight anisotropy. Note —The elastic constants determined by this test method are termed ultrasonic since the pulse frequencies used are above the audible range. The terms sonic and dynamic are sometimes applied to these constants but do not describe them precisely. It is possible that the ultrasonic elastic constants may differ from those determined by other dynamic methods.

ASTM D2850: Standard Test Method for Unconsolidated-Undrained Triaxial Compression Test on Cohesive Soils
This test method covers determination of the strength and stress-strain relationships of a cylindrical specimen of either undisturbed or remolded cohesive soil. Specimens are subjected to a confining fluid pressure in a triaxial chamber. No drainage of the specimen is permitted during the test. The specimen is sheared in compression without drainage at a constant rate of axial deformation (strain controlled).

ASTM D2901: Standard Test Method for Cement Content of Freshly Mixed Soil-Cement (Withdrawn 2006)
This test method covers determination of the cement content of samples of freshly-mixed soil-cement.

ASTM D2922: Standard Test Methods for Density of Soil and Soil-Aggregate in Place by Nuclear Methods (Shallow Depth)

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This test method covers the determination of the total or wet density of soil and soil-rock mixtures by the attenuation of gamma radiation where the source and detector(s) remain on the surface (Backscatter Method) or the source or detector is placed at a known depth up to 300 mm (12 in.) while the detector(s) or source remains on the surface (Direct Transmission Method).

ASTM D2936: Standard Test Method for Direct Tensile Strength of Intact Rock Core Specimens
This test method covers the determination of the direct tensile strength of intact cylindrical rock specimens.

ASTM D2937: Standard Test Method for Density of Soil in Place by the Drive-Cylinder Method
This test method covers the determination of in-place density of soil by the drive-cylinder method. The test method involves obtaining a relatively undisturbed soil sample by driving a thin-walled cylinder and the subsequent activities for the determination of in-place density. When sampling or in-place density is required at depth, Test Method D 1587 should be used.

ASTM D2938: Standard Test Method for Unconfined Compressive Strength of Intact Rock Core Specimens
This test method specifies the apparatus, instrumentation, and procedures for determining unconfined compressive strength of intact rock core specimens.

ASTM D2944: Standard Test Method of Sampling Processed Peat Materials
This test method covers procedures for obtaining samples for use in the determination of moisture, ash, and organic matter; volume; pH; volume weights, water-holding capacity, and air capacity of water-saturated peat materials; total nitrogen; particle size range; and sand content of processed peat materials.

ASTM D2973: Standard Test Method for Total Nitrogen in Peat Materials
This is a chemical test method for the determination of the weight percent of nitrogen in peat material.

ASTM D2974: Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils
These test methods cover the measurement of moisture content, ash content, and organic matter in peats and other organic soils, such as organic clays, silts, and mucks. This test method should be used for geotechnical and general classification. In addition, the test method should be used when peats are being evaluated for use as a fuel.

ASTM D2976: Standard Test Method for pH of Peat Materials
This test method covers the electrometric measurement of the pH of peat materials. It is a means of expressing the degree of acidity or alkalinity of peat material suspended in water and 0.01 M calcium chloride solution.

ASTM D2977: Standard Test Method for Particle Size Range of Peat Materials for Horticultural Purposes

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This test method covers measurement of the weight percentage of fractions of a peat material defined in terms of selected ranges of screen sizes.

ASTM D2978: Standard Test Method for Volume of Processed Peat Materials
This test method covers the measurement of the volume of loose and baled processed peat expressed as cubic feet.

ASTM D2980: Standard Test Method for Volume Weights, Water-Holding Capacity, and Air Capacity of Water-Saturated Peat Materials
This test method was designed to evaluate the aeration, water penetration, and water retention properties of peat under field conditions of water saturation.

ASTM D3017: Standard Test Method for Water Content of Soil and Rock in Place by Nuclear Methods (Shallow Depth)
This test method covers the determination of water content of soil and rock by the thermalization or slowing of fast neutrons where the neutron source and the thermal neutron detector both remain at the surface. The water content in mass per unit volume of the material under test is determined by comparing the detection rate of thermalized or slow neutrons with previously established calibration data.

ASTM D3080: Standard Test Method for Direct Shear Test of Soils Under Consolidated Drained Conditions
This test method covers the determination of the consolidated drained shear strength of a soil material in direct shear. The test is performed by deforming a specimen at a controlled strain rate on or near a single shear plane determined by the configuration of the apparatus. Generally, three or more specimens are tested, each under a different normal load, to determine the effects upon shear resistance and displacement, and strength properties such as Mohr strength envelopes. Shear stresses and displacements are nonuniformly distributed within the specimen. An appropriate height cannot be defined for calculation of shear strains. Therefore, stress-strain relationships or any associated quantity such as modulus, cannot be determined from this test.

ASTM D3087: Standard Test Method for Operating Performance of Anion-Exchange Materials for Strong Acid Removal
This test method covers the determination of the operating capacity of anion-exchange materials when used for the removal of hydrochloric and sulfuric acid from water. It is designed to simulate operating conditions for strong acid removal and is intended for use in testing both new and used materials. The values stated in SI units are to be regarded as the standard. The inch-pound units given in parentheses are for information only.

ASTM D3148: Standard Test Method for Elastic Moduli of Intact Rock Core Specimens in Uniaxial Compression (Withdrawn 2005)
This test method covers the determination of elastic moduli of intact rock core specimens in uniaxial compression. It specifies the apparatus, instrumentation, and procedures for determining the stress-axial strain and the stress-lateral strain curves, as well as Young's modulus, E, and Poisson's ratio, v

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Note —This test method does not include the procedures necessary to obtain a stress-strain curve beyond the ultimate strength.

ASTM D3152: Standard Test Method for Capillary-Moisture Relationships for Fine-Textured Soils by Pressure-Membrane Apparatus (Withdrawn 2007)
This test method covers the determination of capillary-moisture properties of fine-textured soils as indicated by the moisture content - moisture tension relationships determined by pressure-membrane apparatus using tensions between 1 and 15 atm (101 and 1520 kPa). Moisture tension (matrix suction) is defined as the equivalent negative gage pressure, or suction, in soil moisture. The test result is a moisture content which is a measure of the water retained in the soil subjected to a given soil - water tension (or at an approximately equivalent height above the water table).
Note 1--For determination of capillary-moisture relationships of coarse- and medium-textured soils, refer to Test Method D2325.

ASTM D3155: Standard Test Method for Lime Content of Uncured Soil-Lime Mixtures
This test method covers the determination of the lime content of soil-lime mixtures sampled from a project under construction or at the pug-mill, or both. In soils with highly variable amounts of $CaCO_3$ (such as caliche), it may be difficult to obtain a representative sample.

ASTM D3213: Standard Practices for Handling, Storing, and Preparing Soft Undisturbed Marine Soil
These practices cover methods for project/cruise reporting, and handling, transporting and storing soft cohesive undisturbed marine soil. Procedures for preparing soil specimens for triaxial strength, and consolidation testing are also presented. These practices may include the handling and transporting of sediment specimens contaminated with hazardous materials and samples subject to quarantine regulations.

ASTM D3282: Standard Practice for Classification of Soils and Soil-Aggregate Mixtures for Highway Construction Purposes
This practice covers a procedure for classifying mineral and organomineral soils into seven groups based on laboratory determination of particle-size distribution, liquid limit, and plasticity index. It may be used when a precise engineering classification is required, especially for highway construction purposes. Evaluation of soils within each group is made by means of a *group index*, which is a value calculated from an empirical formula. Note—The group classification, including the group index, should be useful in determining the relative quality of the soil material for use in earthwork structures, particularly embankments, subgrades, subbases, and bases. However, for the detailed design of important structures, additional data concerning strength or performance characteristics of the soil under field conditions will usually be required.

ASTM D3370: Standard Practices for Sampling Water from Closed Conduits
These practices cover the equipment and methods for sampling water from closed conduits such as process streams at power stations for chemical, physical, microbiological, and radiological analyses. It does not cover specialized equipment required for and unique to a specific test or method of analysis

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ASTM D3385: Standard Test Method for Infiltration Rate of Soils in Field Using Double-Ring Infiltrometer
This test method describes a procedure for field measurement of the rate of infiltration of liquid (typically water) into soils using double-ring infiltrometer. Soils should be regarded as naturally occurring fine or coarse-grained soils or processed materials or mixtures of natural soils and processed materials, or other porous materials, and which are basically insoluble.

ASTM D3404: Standard Guide for Measuring Matric Potential in Vadose Zone Using Tensiometers
This guide covers the measurement of matric potential in the vadose zone using tensiometers. The theoretical and practical considerations pertaining to successful onsite use of commercial and fabricated tensiometers are described. Measurement theory and onsite objectives are used to develop guidelines for tensiometer selection, installation, and operation.

ASTM D3441: Standard Test Method for Mechanical Cone Penetration Tests of Soil
This test method covers the determination of end bearing and side friction, the components of penetration resistance that are developed during the steady slow penetration of a pointed rod into soil. This test method is sometimes referred to as the Dutch Cone Test or Cone Penetration Test and is often abbreviated as CPT.
This test method includes the use of mechanical cone and friction-cone penetrometers. It does not include the use of electric and electronic cones or data interpretation.
The use of electric and electronic cones is covered in Test Method D 5778.

ASTM D3550: Standard Practice for Thick Wall, Ring-Lined, Split Barrel, Drive Sampling of Soils
This practice covers procedure for thick wall, split barrel drive sampling of soil to obtain representative samples of soil for classification and laboratory testing. The sampler is considered to be a thick wall sampler with sharpened cutting shoe and ball check vent. The middle barrel section is often of split barrel design, but a solid barrel can be used and both may contain ring liners. The sampler is often driven, but can also be pushed in softer deposits. Penetration resistance data may be recorded. This standard uses procedures similar to Test Method D 1586 on Penetration Resistance and Split Barrel Sampling of Soils. However, in this practice, differing hammer weights, drop heights, and different size samplers are used, so the data must not be reported as conforming to Test Method D 1586 and cannot be used to determine Normalized penetration resistance data for sands in accordance with Practice D 6066.

ASTM D3558: Standard Test Methods for Cobalt in Water
These test methods cover the determination of dissolved and total recoverable cobalt in water and wastewater by atomic absorption spectrophotometry. Three test methods are included as follows:

Test Method	Concentration Range	Sections
Test Method A—Atomic Absorption, Direct	0.1 to 10 mg/L	7 to 16
Test Method B—Atomic Absorption, Chelation-Extraction	10 to 1000 µg/L	17 to 26
Test Method C—Atomic Absorption, Graphite Furnace	5 to 100 µg/L	27 to 36

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ASTM D3559: Standard Test Methods for Lead in Water

The test for lead is necessary because it is a toxicant and because there is a limit specified for lead in potable water in the National Interim Primary Drinking Water Regulations. This test serves to determine whether the lead content of potable water is above or below the acceptable limit. These test methods cover the determination of dissolved and total recoverable lead in water and wastewater by atomic-absorption spectrophotometry and differential pulse anodic stripping voltammetry. Four test methods are included as follows:

Test Method	Concentration Range	Sections
Test Method A—Atomic Absorption, Direct	1.0 to 10 mg/L	7 to 15
Test Method B—Atomic Absorption, Chelation-Extraction	100 to 1000 µg/L	16 to 24
Test Method C—Differential Pulse Anodic Stripping Voltammetry	1 to 100 µg/L	25 to 35
Test Method D—Atomic Absorption, Graphite Furnace	5 to 100 µg/L	36 to 44

ASTM D3668: Standard Test Method for Bearing Ratio of Laboratory Compacted Soil-Lime Mixtures (Withdrawn 1997)

This test method covers the determination of the bearing ratio of soil-lime mixtures when compacted and tested in the laboratory by comparing the penetration load of the soil-lime mixture to that of a standard material.

ASTM D3689: Standard Test Method for Individual Piles Under Static Axial Tensile Load (Withdrawn 2005)

This test method covers procedures for testing vertical or batter piles, individually or in groups, to determine response of the pile or pile group to a static tensile load applied axially to the pile or pile group. This test method is applicable to all deep foundation units that function in a manner similar to piles, regardless of their method of installation. This test method is divided into the following sections:

Section Referenced Documents 2 Significance and Use 3 Apparatus for Applying Loads 4 Apparatus for Measuring Movements 5 Safety Precautions 6 Loading Procedures 7 Procedures for Measuring Movements 8 Report 9 Precision and Bias 10

ASTM D3740: Standard Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

This practice establishes minimum qualifications for agencies engaged in the testing and inspection of soil and rock. Minimum requirements for field and laboratory personnel are defined. The practice also covers the establishment and maintenance of a quality system. Criteria are provided for evaluating the capability of an agency to properly perform designated tests on soil and rock, and for establishing essential characteristics pertaining to an agency's organization, personnel, facilities, and quality system. This practice may be supplemented by more specific criteria and requirements for particular projects.

ASTM D3858: Standard Test Method for Open-Channel Flow Measurement of Water by Velocity-Area Method

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This test method is used to measure the volume rate of flow of water moving in rivers and streams and moving over or through large man-made structures. It can also be used to calibrate such measuring structures as dams and flumes. Measurements may be made from bridges, cableways, or boats; by wading; or through holes cut in an ice cover. This test method is used in conjunction with determinations of physical, chemical, and biological quality and sediment loadings where the flow rate is a required parameter.

ASTM D3864: Standard Guide for Continual On-Line Monitoring Systems for Water Analysis

This guide covers the selection, establishment, application, and validation and verification of monitoring systems for determining water characteristics by continual sampling, automatic analysis, and recording or otherwise signaling of output data. This guide provides a unified approach to the use of on-line monitoring systems for water quality analysis. Safety precautions, system design and installation, calibration techniques, operating procedures, and validation and verification procedures shall be in accordance with the specified requirements.

ASTM D3877: Standard Test Methods for One-Dimensional Expansion, Shrinkage, and Uplift Pressure of Soil-Lime Mixtures

These test methods provide procedures for conducting expansion, shrinkage, and uplift pressure tests on compacted soil-lime mixtures and can be used to determine the lime content required to achieve desired control of volume changes caused by increases or decreases of moisture. The tests can be used to determine (a) the magnitude of volume changes under varying load conditions, (b) the rate of volume change, and (c) the magnitude of pressure change as moisture changes of the soil-lime mixture take place. The permeability of soil-lime mixture can also, if desired, be determined at the various load conditions. Note—Changes in field conditions can have major effects on the expansion and shrinkage characteristics of expansive soils. Therefore, to the greatest extent possible, initial and anticipated future field conditions should be duplicated, particularly with respect to moisture and density.

ASTM D3966: Standard Test Methods for Deep Foundations under Lateral Load

The test methods described in this standard measure the lateral deflection of a vertical or inclined deep foundation when subjected to lateral loading. These methods apply to all deep foundations, referred to herein as "pile(s)," that function in a manner similar to driven piles or cast in place piles, regardless of their method of installation, and may be used for testing single piles or pile groups. The test results may not represent the long-term performance of a deep foundation.

ASTM D3967: Standard Test Method for Splitting Tensile Strength of Intact Rock Core Specimens

This test method covers testing apparatus, specimen preparation, and testing procedures for determining the splitting tensile strength of rock by diametral line compression of a disk. Note—The tensile strength of rock determined by tests other than the straight pull test is designated as the “indirect” tensile strength and, specifically, the value obtained in Section 9 of this test is termed the “splitting” tensile strength.

ASTM D3999: Standard Test Methods for the Determination of the Modulus and Damping Properties of Soils Using the Cyclic Triaxial Apparatus

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These test methods cover the determination of the modulus and damping properties of soils in either undisturbed or reconstituted states by either load or stroke controlled cyclic triaxial techniques. The cyclic triaxial properties of soil are evaluated relative to a number of factors including: strain level, density, number of cycles, material type, saturation, and effective stress. These test methods are applicable to both fine-grained and coarse-grained soils as defined by the unified soil classification system or by Classification D 2487. Test specimens may be undisturbed or reconstituted by compaction in the laboratory.

ASTM D4015: Standard Test Methods for Modulus and Damping of Soils by Resonant-Column Method

These test methods cover the determination of shear modulus, shear damping, rod modulus (commonly referred to as Young's modulus), and rod damping for solid cylindrical specimens of soil in the undisturbed and remolded conditions by vibration using the resonant column. The vibration of the specimen may be superposed on a controlled ambient state of stress in the specimen. The vibration apparatus and specimen may be enclosed in a triaxial chamber and subjected to an all-around pressure and axial load. In addition, the specimen may be subjected to other controlled conditions (for example, pore-water pressure, degree of saturation, temperature). These test methods of modulus and damping determination are considered nondestructive when the strain amplitudes of vibration are less than 10^4 rad (10^4 in./in.), and many measurements may be made on the same specimen and with various states of ambient stress.

ASTM D4016: Standard Test Method for Viscosity of Chemical Grouts by Brookfield Viscometer (Laboratory Method)

This test method covers the determination of viscosity of catalysed chemical grouts with the Brookfield viscometer (laboratory method), over the range from 1.0 to 1000 cP (0.001 to 1 Pa·s).

ASTM D4043: Standard Guide for Selection of Aquifer Test Method in Determining Hydraulic Properties by Well Techniques

This guide covers an integral part of a series of standards that are being prepared on the in situ determination of hydraulic properties of aquifer systems by single- or multiple-well tests. This guide provides guidance for development of a conceptual model of a field site and selection of an analytical test method for determination of hydraulic properties. This guide does not establish a fixed procedure for determination of hydrologic properties.

ASTM D4044: Standard Test Method for (Field Procedure) for Instantaneous Change in Head (Slug) Tests for Determining Hydraulic Properties of Aquifers

This test method covers the field procedure for performing an in situ instantaneous change in head (slug) test. This test method is used in conjunction with an analytical procedure such as Test Method D 4104 to determine aquifer properties.

ASTM D4050: Standard Test Method for (Field Procedure) for Withdrawal and Injection Well Tests for Determining Hydraulic Properties of Aquifer Systems

This test method covers the field procedure for selecting well locations, controlling discharge or injection rates, and measuring water levels used to analyze the hydraulic properties of an aquifer or aquifers and adjacent confining beds. This test method is used in conjunction with an

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analytical procedure such as Test Methods D 4105 or D 4106 to determine aquifer properties. The appropriate field and analytical procedures are selected as described in Guide D 4043.

ASTM D4083: Standard Practice for Description of Frozen Soils (Visual-Manual Procedure)
This practice presents a procedure for the description of frozen soils based on visual examination and simple manual tests. It is intended to be used in conjunction with Test Method D 2487 and Practice D 2488, which describe and classify soils, but do not cover their frozen state. This procedure is based on "Guide to Field Description of Permafrost for Engineering Purposes," National Research Council of Canada, 1963, and MIL-STD-619.

ASTM D4104: Standard Test Method (Analytical Procedure) for Determining Transmissivity of Nonleaky Confined Aquifers by Overdamped Well Response to Instantaneous Change in Head (Slug Tests)
This test method covers the determination of transmissivity from the measurement of force-free (overdamped) response of a well-aquifer system to a sudden change of water level in a well. Force-free response of water level in a well to a sudden change in water level is characterized by recovery to initial water level in an approximate exponential manner with negligible inertial effects. The analytical procedure in this test method is used in conjunction with the field procedure in Test Method D 4044 for collection of test data.

ASTM D4105: Standard Test Method (Analytical Procedure) for Determining Transmissivity and Storage Coefficient of Nonleaky Confined Aquifers by the Modified Theis Nonequilibrium Method
This test method covers an analytical procedure for determining transmissivity and storage coefficient of a nonleaky confined aquifer under conditions of radial flow to a fully penetrating well of constant flux. This test method is a shortcut procedure used to apply the Theis nonequilibrium method. The Theis method is described in Test Method D 4106. This test method is used in conjunction with the field procedure given in Test Method D 4050.

ASTM D4106: Standard Test Method for (Analytical Procedure) for Determining Transmissivity and Storage Coefficient of Nonleaky Confined Aquifers by the Theis Nonequilibrium Method
This test method covers an analytical procedure for determining the transmissivity and storage coefficient of a nonleaky confined aquifer. It is used to analyze data on water-level response collected during radial flow to or from a well of constant discharge or injection. This analytical procedure is used in conjunction with the field procedure given in Test Method D 4050.

ASTM D4127: Standard Terminology Used with Ion-Selective Electrodes
This terminology covers those terms recommended by the International Union of Pure and Applied Chemistry (IUPAC), and is intended to provide guidance in the use of ion-selective electrodes for analytical measurement of species in water, wastewater, and brines.

ASTM D4186: Standard Test Method for One-Dimensional Consolidation Properties of Saturated Cohesive Soils Using Controlled-Strain Loading

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This test method is for the determination of the magnitude and rate-of-consolidation of saturated cohesive soils using continuous controlled-strain axial compression. The specimen is restrained laterally and drained axially to one surface. The axial force and base excess pressure are measured during the deformation process. Controlled strain compression is typically referred to as constant rate-of-strain (CRS) testing. This test method provides for the calculation of total and effective axial stresses, and axial strain from the measurement of axial force, axial deformation, and base excess pressure. The effective stress is computed using steady state equations.

ASTM D4189: Standard Test Method for Silt Density Index (SDI) of Water
This test method covers the determination of the silt density index (SDI) of water. This test method can be used to indicate the quantity of particulate matter in water and is applicable to relatively low (1.0 NTU) turbidity waters such as well water, filtered water, or clarified effluent samples. Since the size, shape, and nature of particulate matter in water may vary, this test method is not an absolute measurement of the quantity of particulate matter.

ASTM D4219: Standard Test Method for Unconfined Compressive Strength Index of Chemical- Grouted Soils
This test method covers the determination of the short-term unconfined compressive strength index of chemically grouted soils, using strain-controlled application of test load.

ASTM D4220: Standard Practices for Preserving and Transporting Soil Samples
These practices cover procedures for preserving soil samples immediately after they are obtained in the field and accompanying procedures for transporting and handling the samples. Limitations - These practices are not intended to address requirements applicable to transporting of soil samples known or suspected to contain hazardous materials.

ASTM D4221: Standard Test Method for Dispersive Characteristics of Clay Soil by Double Hydrometer
This test method, when used in conjunction with a test performed by Test Method D 422 on a duplicate soil sample, provides an indication of the natural dispersive characteristics of clay soils. This test method is applicable only to soils with a plasticity index greater than 4 as determined in accordance with Test Method D 4318 and more than 12 % of the soil fraction finer than 5-m as determined in accordance with Test Method D 422

ASTM D4223: Standard Practice for Preparation of Test Specimens of Asphalt-Stabilized Soils
This practice covers the selection and proportioning of soils and emulsified or cutback asphalts and the fabrication of 4-in. (102-mm) diameter by 2.5-in. (54-mm) high test specimens. All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D 6026.

ASTM D4253: Standard Test Methods for Maximum Index Density and Unit Weight of Soils Using a Vibratory Table
These test methods cover the determination of the maximum-index dry density/unit weight of cohesionless, free-draining soils using a vertically vibrating table. The adjective "dry" before

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density or unit weight is omitted in the title and remaining portions of this standard to be consistent with the applicable definition given in Section 3 on Terminology.

ASTM D4254: Standard Test Methods for Minimum Index Density and Unit Weight of Soils and Calculation of Relative Density

These test methods cover the determination of the minimum-index dry density/unit weight of cohesionless, free-draining soils. The adjective "dry" before density or unit weight is omitted in the title and remaining portions of this standard to be consistent with the applicable definitions given in Section 3 on Terminology.

ASTM D4318: Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils

These test methods cover the determination of the liquid limit, plastic limit, and the plasticity index of soils as defined in Section on Terminology. Two methods for preparing test specimens are provided as follows: *Wet preparation method* and *Dry preparation method*. The method to be used shall be specified by the requesting authority. If no method is specified, use the wet preparation method. The liquid and plastic limits of many soils that have been allowed to dry before testing may be considerably different from values obtained on non-dried samples. If the liquid and plastic limits of soils are used to correlate or estimate the engineering behavior of soils in their natural moist state, samples should not be permitted to dry before testing unless data on dried samples are specifically desired.

ASTM D4319: Standard Test Method for Distribution Ratios by the Short-Term Batch Method

This test method covers the determination of distribution ratios of chemical species for site-specific geological media by a batch sorption technique. It is a short-term laboratory method primarily intended for ionic species subject to migration in granular porous material, and the application of the results to long-term field behavior is not known. Distribution ratios for radionuclides in selected geomedia are commonly determined for the purpose of assessing potential migratory behavior at waste repositories. This test method is also applicable to studies of intrusion waters and for parametric studies of the effects of variables and of mechanisms which determine the measured distribution ratios.

ASTM D4320: Standard Practice for Laboratory Preparation of Chemically Grouted Soil Specimens for Obtaining Design Strength Parameters

This practice covers the laboratory preparation of chemically grouted soil specimens for use in laboratory tests to determine design strength parameters. Note 1—This practice may not be applicable to grout mixtures with gel times shorter than the time required to saturate the specimen with grout. The specimens are intended for both strength and modulus determination in unconfined and confined compression testing. Note 2—Preparation methods for specimens to be used for other purposes are described in Test Methods D 4219 and D 5202.

ASTM D4341: Standard Test Method for Creep of Cylindrical Hard Rock Core Specimens in Uniaxial Compression

This test method covers the creep behavior of intact cylindrical hard rock core specimens in uniaxial compression. It specifies the apparatus, instrumentation, and procedures for determining

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the strain as a function of time under sustained load. Hard rocks are those with maximum strain at failure of less than 2%. Note -Most hard brittle rocks fail in uniaxial compression at strain levels of less than 2%.

ASTM D4373: Standard Test Method for Rapid Determination of Carbonate Content of Soils

This test method covers the determination of carbonate content of soils and soft rock which can be readily broken down by mechanical effort. It is a gasometric method that utilizes a simple portable apparatus. Results should be clearly stated as the calcite equivalent in percent because different carbonate species cover a wide range of percent calcite equivalent as shown below for a number of carbonates: For example, a 100% dolomite would be expected to yield 108.6% calcite equivalent while 100% siderite would yield only 86.4% calcite equivalent. Calcite and aragonite reactions will typically complete within about 10 min. This method does not distinguish between the carbonate species and such determination must be made using quantitative chemical analysis methods such as atomic absorption. The method used to specify how data are collected, calculated, or recorded in this standard is not directly related to the accuracy to which the data can be applied in design or other uses, or both. How one applies the results obtained using this standard is beyond its scope.

ASTM D4380: Standard Test Method for Density of Bentonitic Slurries

This test method covers the determination of the density of slurries used in slurry construction techniques, such as are used for barriers to control the horizontal movement of liquids. This test method is modified from API Recommended Practice 13B.

ASTM D4381: Standard Test Method for Sand Content by Volume of Bentonitic Slurries

This test method covers the determination of the sand content of bentonitic slurries used in slurry construction techniques. This test method has been modified from API Recommended Practice 13B.

ASTM D4394: Standard Test Method for Determining In Situ Modulus of Deformation of Rock Mass Using Rigid Plate Loading Method

This test method covers the preparation, equipment, test procedure, and data reduction for determining in situ modulus of deformation of a rock mass using the rigid plate loading method. This test method is designed to be conducted in an adit or small underground chamber; however, with suitable modifications it could be conducted at the surface.

ASTM D4395: Standard Test Method for Determining In Situ Modulus of Deformation of Rock Mass Using Flexible Plate Loading Method

This test method covers the preparation, equipment, test procedure, and data reduction for determining in situ modulus of deformation of a rock mass using the flexible plate loading method. This test method is designed to be conducted in an adit or small underground chamber; however, with suitable modifications it could be conducted at the surface. This test method is usually conducted parallel or perpendicular to the anticipated axis of thrust, as dictated by the design load.

ASTM D4403: Standard Practice for Extensometers Used in Rock

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This practice covers the description, application, selection, installation, data collecting, and data reduction of the various types of extensometers used in the field of rock mechanics. Limitations of each type of extensometer system are covered.

ASTM D4404: Standard Test Method for Determination of Pore Volume and Pore Volume Distribution of Soil and Rock by Mercury Intrusion Porosimetry
This test method covers the determination of the pore volume and the pore volume distributions of soil and rock by the mercury intrusion porosimetry method. The range of apparent diameters of pores for which this test method is applicable is fixed by the operant pressure range of the testing instrument. This range is typically between apparent pore entrance diameters of about 100 [mu]m and 2.5 nm (0.0025 [mu]m). Larger pores must be measured by another method. Mercury intrusion porosimetry is useful only for measuring pores open to the outside of a soil or rock fragment; mercury intrusion porosimetry will not give the volume of any pores completely enclosed by surrounding solids. This test method will give only the volume of intrudable pores that have an apparent diameter corresponding to a pressure within the pressurizing range of the testing instrument.

ASTM D4405: Standard Test Method for Creep of Cylindrical Soft Rock Core Specimens in Uniaxial Compressions
This test method covers the creep behavior of intact cylindrical soft rock core specimens in uniaxial compression. It specifies the apparatus, instrumentation, and procedures for determining the strain as a function of time under sustained load. Soft rocks include such materials as salt and potash, which often exhibit very large strain at failure.

ASTM D4406: Standard Test Method for Creep of Cylindrical Rock Core Specimens in Triaxial Compression
This test method covers the creep behavior of intact cylindrical rock core specimens' in triaxial compression. It specifies the apparatus, instrumentation, and procedures for determining the strain as a function of time under sustained load.

ASTM D4409: Standard Test Method for Velocity Measurements of Water in Open Channels with Rotating Element Current Meters
This test method describes the design and use of various types of current meters. These current meters are commonly used to measure the velocity at a point in an open channel cross section as part of a velocity-area traverse to determine the flow rate of water. To this end it should be used in conjunction with Test Method D 3858. This test method describes the design and use of cup-type or vane-type vertical axis current meters and propeller-type horizontal axis current meters for measuring water velocities in open channels.

ASTM D4410: Terminology for Fluvial Sediment
These terms are to be used by persons involved in collecting, reporting, and interpreting information pertaining to sedimentation and hydrologic processes as they apply in the development, use, control, and conservation of water and land resources.

ASTM D4427: Standard Classification of Peat Samples by Laboratory Testing

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This classification is a system for subdividing and assigning nomenclature to peat samples through laboratory tests.

ASTM D4428 / D4428M: Standard Test Methods for Crosshole Seismic Testing
These test methods are limited to the determination of horizontally traveling compression (*P*) and shear (*S*) seismic waves at test sites consisting primarily of soil materials (as opposed to rock). A preferred test method intended for use on critical projects where the highest quality data must be obtained is included. Also included is an optional method intended for use on projects which do not require measurements of a high degree of precision. Various applications of the data will be addressed and acceptable interpretation procedures and equipment, such as seismic sources, receivers, and recording systems will be discussed. Other items addressed include borehole spacing, drilling, casing, grouting, deviation surveys, and actual test conduct. Data reduction and interpretation is limited to the identification of various seismic wave types, apparent velocity relation to true velocity, example computations, effective borehole spacing, use of Snell's law of refraction, assumptions, and computer programs.

ASTM D4429: Standard Test Method for CBR (California Bearing Ratio) of Soils in Place
This test method covers the determination of the California Bearing Ratio (CBR) of soil tested in place by comparing the penetration load of the soil to that of a standard material. This test method covers the evaluation of the relative quality of subgrade soils, but is applicable to subbase and some base-course materials. This test method is designed to test in-situ materials and corresponds to Test Method D 1883.

ASTM D4435: Standard Test Method for Rock Bolt Anchor Pull Test
The objective of this test method is to measure the working and ultimate capacities of a rock bolt anchor. This method does not measure the entire roof support system. This method also does not include tests for pretensioned bolts or mine roof support system evaluation. This test method is applicable to mechanical, cement grout, resin, (epoxy, polyester, and the like), or other similar anchor systems.

ASTM D4436: Standard Test Method for Rock Bolt Long-Term Load Retention Test
The objective of this test method is to determine the time over which rock bolt tension decreases from the installed value to a designated minimum value. This test method is applicable to any anchor system which is not fully encapsulated immediately upon installation, including mechanical, cement grout, resin (epoxy, polyester, and the like) or other similar systems.

ASTM D4452: Standard Practice for X-Ray Radiography of Soil Samples
This practice covers the determination of the quality of soil samples in thin wall tubes or of extruded cores by X-ray radiography. This practice enables the user to determine the effects of sampling and natural variations within samples as identified by the extent of the relative penetration of X-rays through soil samples.
This practice can be used to X-ray cores (or observe their features on a fluoroscope) in thin wall tubes or liners ranging from approximately 2 to 6 in. (51 to 152 mm) in diameter. X-rays of samples in the larger diameter tubes provide a radiograph of major features of soils and disturbances, such as large scale bending of edges of varved clays, shear planes, the presence of large concretions, silt and sand seams thicker than 1/4 in. (6.4 mm), large lumps of organic

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matter, and voids or other types of intrusions. X-rays of the smaller diameter cores provide higher resolution of soil features and disturbances, such as small concretions (1/8 in. (3.2 mm) diameter or larger), solution channels, slight bending of edges of varved clays, thin silt or sand seams, narrow solution channels, plant root structures, and organic matter. The X-raying of samples in thin wall tubes or liners requires minimal preparation

ASTM D4506: Standard Test Method for Determining In Situ Modulus of Deformation of Rock Mass Using Radial Jacking Test

This test method is used to determine the in situ modulus of deformation of rock mass by subjecting a test chamber of circular cross section to uniformly distributed radial loading; the consequent rock displacements are measured, from which elastic or deformation moduli may be calculated. The anisotropic deformability of the rock can also be measured and information on time-dependent deformation may be obtained. This test method is based upon the procedures developed by the U.S. Bureau of Reclamation featuring long extensometers. An alternative procedure is also available and is based on a reference bar

ASTM D4511: Standard Test Method for Hydraulic Conductivity of Essentially Saturated Peat

This test method covers the determination of the hydraulic conductivity (permeability) of essentially saturated, intact cylindrical specimens of peat when the hydraulic conductivity is greater than $1\ 10^{-7}$ m/s ($1\ 10^{-5}$ cm/s). During the test, the specimens are contained in the core holder, or in right, regular cylindrical sections cut from the sampling tube in which they were originally obtained in the field. Hydraulic conductivity is calculated on the basis of the measured constant flow rate through the specimen under constant head. For verification, flow rate determinations may be made at two or more values of constant head with corresponding calculations of hydraulic conductivity.

ASTM D4525: Standard Test Method for Permeability of Rocks by Flowing Air

This test method covers the determination of the coefficient of specific permeability for the flow of air through rocks. The procedure establishes representative values of the coefficient of permeability of rocks or well-indurated soils. This test method is limited to permeability values greater than 0.9869 pm^2 (1.0 picodarcy), and is limited to rocks free of oil or unctuous matter.

ASTM D4531: Standard Test Methods for Bulk Density of Peat and Peat Products

These test methods cover the determination of the bulk density of both peat in its natural state and peat products (Note 1). These test methods consist of defining a volume of peat and determining the mass of that specific volume. The difference in the respective methods is in the procedures employed to determine the peat volume. Note—Test Method D 2978 is used for specifying the volume represented by a quantity of loose processed peat or the volume of a peat bale.

ASTM D4535: Standard Test Methods for Measurement of Thermal Expansion of Rock Using Dilatometer

These test methods cover the laboratory measurement of the linear (one-dimensional) thermal expansion of rocks using a dilatometer. These test methods are applicable between temperatures of 25°C to 300°C. Both bench top and confined measurement techniques are presented. Rocks of

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varying moisture content can be tested. For satisfactory results in conformance with these test methods, the principles governing the size, construction, and use of the apparatus described in these methods should be followed. If the results are to be reported as having been obtained by this method, then all pertinent requirements prescribed in this method shall be met.

ASTM D4542: Standard Test Method for Pore Water Extraction and Determination of the Soluble Salt Content of Soils by Refractometer

This test method covers a rapid procedure for squeezing pore water from fine-grained soils for the purpose of determining the amount of soluble salts present in the extracted pore water. This test method was developed for soils having a water content equal to or greater than approximately 14 %, for example, marine soils. An extensive summary of procedures for extracting pore water from soils has been presented by Kriukov and Manheim. This test method is not generally applicable for determining the soluble salt content of the pore water extracted from coarse-grained soils, such as clean sands and gravels.

ASTM D4543: Standard Practices for Preparing Rock Core as Cylindrical Test Specimens and Verifying Conformance to Dimensional and Shape Tolerances

These practices specify procedures for laboratory rock core test specimen preparation of rock core from drill core and block samples for strength and deformation testing and for determining the conformance of the test specimen dimensions with tolerances established by this practice. Cubical, rectangular, or other shapes are not covered by this practice. However, some of the information contained within this practice and in standard Test Method C 170 may still be of use to preparing other test specimen shapes.

ASTM D4544: Standard Practice for Estimating Peat Deposit Thickness

This practice uses a technique of probing to estimate the thickness of surficial peat deposits overlying mineral soil or bedrock. These estimates may be needed for energy, horticultural, or geotechnical purposes.

ASTM D4546: Standard Test Methods for One-Dimensional Swell or Settlement Potential of Cohesive Soils

These test methods cover three alternative laboratory methods for measuring free swell, swell pressure, and the magnitude of one-dimensional swell or collapse of compacted or intact cohesive soils.

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The test methods can be used to measure the magnitude of one-dimensional wetting-induced swell or collapse (hydrocompression) under different vertical (axial) pressures, as well as the magnitude of swell pressure and the magnitude of free swell. It can also be used to obtain data for stress-induced compression following wetting-induced swell or collapse.

ASTM D4553: Standard Test Method for Determining In Situ Creep Characteristics of Rock

This test method covers the preparation, equipment, test procedure, and data documentation for determining in situ creep characteristics of a rock mass using a rigid plate subjected to controlled loading. This test method is designed to be conducted in an underground opening; however, with suitable modifications, this test could be conducted at the surface. The test is usually conducted

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parallel or perpendicular to the anticipated axis of thrust, as dictated by the design load or other orientations, based upon the application.

ASTM D4555: Standard Test Method for Determining Deformability and Strength of Weak Rock by an In Situ Uniaxial Compressive Test

This test method covers the measurement of the deformability and strength of large in situ specimens of weak rock by a uniaxial - compressive test. The test results take into account the effect of both intact material behavior and the behavior of discontinuities contained within the specimen block.

ASTM D4554: Standard Test Method for In Situ Determination of Direct Shear Strength of Rock Discontinuities

This test method covers the measurement of peak and residual direct shear strength of in situ rock discontinuities as a function of stress normal to the sheared plane. This sheared plane is usually a significant discontinuity which may or may not be filled with gouge or soil-like material. The measured shear properties are affected by scale factors. The severity of the effect of these factors must be assessed and applied to the specific problems on an individual basis.

ASTM D4564: Standard Test Method for Density and Unit Weight of Soil in Place by the Sleeve Method

This test method covers the determination of the density of soil in place by the sleeve method.
The sleeve method of determining the density of soil in place is used for cohesionless, granular soils for which other methods of determining the density (sand cone, test pit, and the like) may not be practical. Typically, the sleeve method is applicable for soils that are predominantly fine gravel size, with a maximum of 5 % fines, and a maximum particle size of ¾ in. (19.0 mm). Note —There have been other methods developed for testing cohesionless soils. Compared to other methods, this procedure is convenient for field construction control testing because smaller and lighter equipment is used and the test can be performed in a smaller area.

ASTM D4609: Standard Guide for Evaluating Effectiveness of Admixtures for Soil Stabilization

This guide describes laboratory techniques for evaluating the effectiveness of admixtures for improving the engineering properties of fine-grained soils. Effectiveness is assessed by comparing the unconfined compressive strength (UCS), moisture susceptibility, and moisture-density relationships (MD) of treated and untreated soils.

ASTM D4611: Standard Test Method for Specific Heat of Rock and Soil

This test method covers the determination of instantaneous and mean specific heat of rock and soil. This test method employs the classical method of mixtures. This provides procedures and apparatus simpler than those generally used in scientific calorimetry, an accuracy that is adequate for most rocks and soils, and a degree of precision that is reproducible by laboratory technicians of average skill. While this test method was developed for testing rock and soil, it is easily adaptable to measuring the specific heat of other materials. The testing procedure provides an instantaneous specific heat over the temperature 25 to 300°C or a mean specific heat in that temperature range. The test procedure is limited to dry samples.

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ASTM D4612: Standard Practice for Calculating Thermal Diffusivity of Rocks
This practice involves calculation of the thermal diffusivity from measured values of the mass density, thermal conductivity, and specific heat at constant pressure. It is applicable for any materials where these data can be determined. The temperature range covered by this practice is 20 to 300°C. Note 1—The diffusivity, as determined by this practice, is intended to be a volume average value, with the averaging volume being $\geq 2 \times 10^{-5}$ m^3 (20 cm^3). This requirement necessitates the use of specimens with volumes greater than the minimum averaging volume and precludes use of flash methods of measuring thermal diffusivity, such as the laser pulse technique. Note 2—This practice is closely linked to the overall test procedure used in obtaining the primary data on density, specific heat, and conductivity. It cannot be used as a "stand alone" practice because the thermal diffusivity values calculated by this practice are dependent on the nature of the primary data base. The practice furnishes general guidelines but cannot be considered to be all-inclusive.

ASTM D4622: Standard Test Method for Rock Mass Monitoring Using Inclinometers (Withdrawn 2000)
This test method describes the use of inclinometers for rock mass monitoring, lists some available instruments, outlines operating techniques and maintenance requirements, and presents data reduction formulas. This standard may involve hazardous materials, operations, and equipment.

ASTM D4623: Standard Test Method for Determination of In Situ Stress in Rock Mass by Overcoring Method—USBM Borehole Deformation Gauge
This test method covers the determination of the ambient local stresses in a rock mass and the equipment required to perform in situ stress tests using a three-component borehole deformation gauge (BDG). The test procedure and method of data reduction are described, including the theoretical basis and assumptions involved in the calculations. A section is included on troubleshooting equipment malfunctions. Note —The gauge used in this test method is commonly referred to as a USBM gauge (U.S. Bureau of Mines three-component borehole deformation gauge).

ASTM D4630: Standard Test Method for Determining Transmissivity and Storage Coefficient of Low-Permeability Rocks by In Situ Measurements Using the Constant Head Injection Test
This test method covers a field procedure for determining the transmissivity and storativity of geological formations having permeabilities lower than 10^{-3} μm^2 (1 millidarcy) using constant head injection. The transmissivity and storativity values determined by this test method provide a good approximation of the capacity of the zone of interest to transmit water, if the test intervals are representative of the entire zone and the surrounding rock is fully water-saturated.

ASTM D4631: Standard Test Method for Determining Transmissivity and Storativity of Low Permeability Rocks by In Situ Measurements Using Pressure Pulse Technique
This test method covers a field procedure for determining the transmissivity and storativity of geological formations having permeabilities lower than 10^{-3} μm^2 (1 millidarcy) using the pressure pulse technique. The transmissivity and storativity values determined by this test method provide a good approximation of the capacity of the zone of interest to transmit water, if

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the test intervals are representative of the entire zone and the surrounding rock is fully water saturated.

ASTM D4643: Standard Test Method for Determination of Water (Moisture) Content of Soil by Microwave Oven Heating

This test method outlines procedures for determining the water (moisture) content of soils by incrementally drying soil in a microwave oven. This test method can be used as a substitute for Test Method D 2216 when more rapid results are desired to expedite other phases of testing and slightly less accurate results are acceptable. When questions of accuracy between this test method and Test Method D 2216 arise, Test Method D 2216 shall be the referee method. This test method is applicable for most soil types. For some soils, such as those containing significant amounts of halloysite, mica, montmorillonite, gypsum or other hydrated materials, highly organic soils, or soils in which the pore water contains dissolved solids (such as salt in the case of marine deposits), this test method may not yield reliable water content values.

ASTM D4644: Standard Test Method for Slake Durability of Shales and Similar Weak Rocks

This test method covers the determination of the slake durability index of a shale or other similar rock after two drying and wetting cycles with abrasion.

ASTM D4645: Standard Test Method for Determination of In-Situ Stress in Rock Using Hydraulic Fracturing Method

This test method covers the determination of the in-situ state of stress in rock by hydraulic fracturing. Note —Hydraulic fracturing for stress determination is also referred to as hydrofracturing, and sometimes as minifracing. Hydraulic fracturing and hydrofracturing may also refer to fracturing of the rock by fluid pressure for the purpose of altering rock properties, such as permeability and porosity. Hydraulic fracturing is the widely accepted field method available for in situ stress measurements at depths greater than 50 m. It can be used in drill holes of any diameter. Hydraulic fracturing can also be used in short holes for which other stress measuring methods, such as overcoring, are also available. The advantage of hydraulic fracturing is that it yields stresses averaged over a few square meters (the size of the induced hydraulic fracture) rather than over grain size areas, as in the case of overcoring techniques.

ASTM D4647: Standard Test Method for Identification and Classification of Dispersive Clay Soils by the Pinhole Test

This test method presents a direct, qualitative measurement of the dispersibility and consequent colloidal erodibility of clay soils by causing water to flow through a small hole punched in a specimen. This test method is complemented by Test Method D 4221. This test method and the criteria for evaluating test data are based upon results of several hundred tests on samples collected from embankments, channels, and other areas where clay soils have eroded or resisted erosion in nature. Three alternative procedures for classifying the dispersibility of clay soils are provided .

ASTM D4648: Standard Test Method for Laboratory Miniature Vane Shear Test for Saturated Fine-Grained Clayey Soil

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This test method covers the miniature vane test in very soft to stiff saturated fine-grained clayey soils ($\Phi = 0$). Knowledge of the nature of the soil in which each vane test is to be made is necessary for assessment of the applicability and interpretation of the test results. Note—It is recommended that the miniature vane test be conducted in fine-grained, predominantly clay soils with an undrained shear strength less than 1.0 tsf (100 kPa). Vane failure conditions in higher strength clay and predominantly silty soils may deviate from the assumed cylindrical failure surface, thereby causing error in the measured strength.

ASTM D4696: Standard Guide for Pore-Liquid Sampling from the Vadose Zone
This guide covers the equipment and procedures used for sampling pore-liquid from the vadose zone (unsaturated zone). The guide is limited to in situ techniques and does not include soil core collection and extraction methods for obtaining samples. The term "pore-liquid" is applicable to any liquid from aqueous pore-liquid to oil. However, all of the samplers described in this guide were designed, and are used to sample aqueous pore-liquids only. The abilities of these samplers to collect other pore-liquids may be quite different than those described. Some of the samplers described in this guide are not currently commercially available. These samplers are presented because they may have been available in the past, and may be encountered at sites with established vadose zone monitoring programs. In addition, some of these designs are particularly suited to specific situations. If needed, these samplers could be fabricated.

ASTM D4700: Standard Guide for Soil Sampling from the Vadose Zone
This guide covers procedures that may be used for obtaining soil samples from the vadose zone (unsaturated zone). Samples can be collected for a variety of reasons including the following:
1. Stratigraphic description,
2. Hydraulic conductivity testing,
3. Moisture content measurement,
4. Moisture release curve construction,
5. Geotechnical testing,
6. Soil gas analyses,
7. Microorganism extraction, or
8. Pore liquid and soils chemical analyses.

This guide focuses on methods that provide soil samples for chemical analyses of the soil or contained liquids or contaminants. However, comments on how methods may be modified for other objectives are included.

ASTM D4718: Standard Practice for Correction of Unit Weight and Water Content for Soils Containing Oversize Particles
This practice presents a procedure for calculating the unit weights and water contents of soils containing oversize particles when the data are known for the soil fraction with the oversize particles removed. The practice also can be used to calculate the unit weights and water contents of soil fractions when the data are known for the total soil sample containing oversize particles.

ASTM D4719: Standard Test Method for Prebored Pressuremeter Testing in Soils
This test method covers pressuremeter testing of soils. A pressuremeter test is an in situ stress-strain test performed on the wall of a borehole using a cylindrical probe that is expanded radially. To obtain viable test results, disturbance to the borehole wall must be minimized. This test

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method includes the procedure for drilling the borehole, inserting the probe, and conducting pressuremeter tests in both granular and cohesive soils, but does not include high pressure testing in rock. Knowledge of the type of soil in which each pressuremeter test is to be made is necessary for assessment of (1) the method of boring or probe placement, or both, (2) the interpretation of the test data, and (3) the reasonableness of the test results. This test method does not cover the self-boring pressuremeter, for which the hole is drilled by a mechanical or jetting tool inside the hollow core of the probe. This test method is limited to the pressuremeter which is inserted into predrilled boreholes or, under certain circumstances, is inserted by driving.

ASTM D4729: Standard Test Method for In Situ Stress and Modulus of Deformation Using Flatjack Method

The flatjack test measures stress at a rock surface. The modulus of deformation and the long-term deformational properties (creep) may also be evaluated. Limitation—The flatjack test measures the average stress normal to the surface of the test chamber. Undisturbed stress levels must be determined by theoretical interpretations of these data. Assumptions and Factors Influencing the Data:

1. The stress relief is assumed to be an elastic, reversible process. In nonhomogeneous or highly fractured materials, this may not be completely true.
2. The equations assume that the rock mass is isotropic and homogeneous. Anisotropic effects may be estimated by testing in different orientations.
3. The flatjack is assumed to be 100 % efficient. The design and size requirements of 7.1 were determined to satisfy this requirement to within a few percent.
4. The jack is assumed to be aligned with the principal stresses on the surface of the opening. Shear stresses are not canceled by jack pressure. Orientating the tests in three directions in each plane tested prevents the misalignment from being excessive for at least one of the tests.

ASTM D4750: Standard Test Method for Determining Subsurface Liquid Levels in a Borehole or Monitoring Well (Observation Well)

This test method describes the procedures for measuring the level of liquid in a borehole or well and determining the stabilized level of liquid in a borehole. The test method applies to boreholes (cased or uncased) and monitoring wells (observation wells) that are vertical or sufficiently vertical so a flexible measuring device can be lowered into the hole. Borehole liquid-level measurements obtained using this test method will not necessarily correspond to the level of the liquid in the vicinity of the borehole unless sufficient time has been allowed for the level to reach equilibrium position. This test method generally is not applicable for the determination of pore-pressure changes due to changes in stress conditions of the earth material. This test method is not applicable for the concurrent determination of multiple liquid levels in a borehole.

ASTM D4753: Standard Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

This guide provides minimum requirements for general-purpose scales, balances, and standard masses used in testing soil, rock, and related construction materials.

ASTM D4759: Standard Practice for Determining the Specification Conformance of Geosynthetics

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This practice covers a procedure for determining the conformance of geosynthetic properties to standard specifications. This practice provides a means by which the purchaser can determine the conformance of geosynthetic properties as applied to the acceptance or rejection of a material in regards to a material specification.

ASTM D4767: Standard Test Method for Consolidated Undrained Triaxial Compression Test for Cohesive Soils
This test method covers the determination of strength and stress-strain relationships of a cylindrical specimen of either an undisturbed or remolded saturated cohesive soil. Specimens are isotropically consolidated and sheared in compression without drainage at a constant rate of axial deformation (strain controlled). This test method provides for the calculation of total and effective stresses, and axial compression by measurement of axial load, axial deformation, and pore-water pressure.

ASTM D4829: Standard Test Method for Expansion Index of Soils
This test method allows for determination of expansion potential of compacted soils when inundated with distilled water. This test method provides a simple yet sensitive method for determination of expansion potential of compacted soils for practical engineering applications using an index parameter.

ASTM D4832: Standard Test Method for Preparation and Testing of Controlled Low Strength Material (CLSM) Test Cylinders
This test method covers procedures for the preparation, curing, transporting and testing of cylindrical test specimens of controlled low strength material (CLSM) for the determination of compressive strength. This test method also may be used to prepare and test specimens of other mixtures of soil and cementitious materials, such as self-cementing fly ashes. CLSM is also known as flowable fill, controlled density fill, soil-cement slurry, soil-cement grout, unshrinkable fill, K-Krete, and other similar names.

ASTM D4840: Standard Guide for Sampling Chain-of-Custody Procedures
This guide contains a comprehensive discussion of potential requirements for a sample chain-of-custody program and describes the procedures involved in sample chain-of-custody. The purpose of these procedures is to provide accountability for and documentation of sample integrity from the time samples are collected until sample disposal. These procedures are intended to document sample possession during each stage of a sample's life cycle, that is, during collection, shipment, storage, and the process of analysis.

ASTM D4841: Standard Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents
This practice covers the means of estimating the period of time during which a water sample can be stored after collection and preservation without significantly affecting the accuracy of analysis. The maximum holding time is dependent upon the matrix used and the specific analyte of interest. Therefore, water samples from a specific source must be tested to determine the period of time that sample integrity is maintained by standard preservation practices.

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ASTM D4879: Standard Guide for Geotechnical Mapping of Large Underground Openings in Rock
This guide recommends procedures for mapping large subsurface openings made for either civil or mining purposes. The mapping provides characterization and documentation of the condition of the rock mass at the excavation surface.

ASTM D4914: Standard Test Methods for Density and Unit Weight of Soil and Rock in Place by the Sand Replacement Method in a Test Pit
These test methods cover the determination of the in-place density and unit weight of soil and rock using a pouring device and calibrated sand to determine the volume of a test pit. The word "rock" in these test methods is used to imply that the material being tested will typically contain particles larger than 3 in. (75 mm).

ASTM D4943: Standard Test Method for Shrinkage Factors of Soils by the Wax Method
This test method covers the procedure for determining the shrinkage limit of soils.
The data obtained using this test method may also be used to calculate shrinkage ratio, volumetric shrinkage, and linear shrinkage. This test method is applicable only for cohesive soils.

ASTM D4944: Standard Test Method for Field Determination of Water (Moisture) Content of Soil by the Calcium Carbide Gas Pressure Tester
This test method outlines procedures for determining the water (moisture) content of soil by chemical reaction using calcium carbide as a reagent to react with the available water in the soil producing a gas. A measurement is made of the gas pressure produced when a specified mass of wet or moist soil is placed in a testing device with an appropriate volume of reagent and mixed.

ASTM D4945: Standard Test Method for High-Strain Dynamic Testing of Piles
This dynamic test method covers the procedure for applying an axial impact force with a pile driving hammer or a large drop weight that will cause a relatively high strain at the top of an individual vertical or inclined deep foundation unit, and for measuring the subsequent force and velocity response of that deep foundation unit. High-strain dynamic testing applies to any deep foundation unit, also referred to herein as a "pile," which functions in a manner similar to a driven pile or a cast-in-place pile regardless of the method of installation, and which conforms with the requirements of this test method.

ASTM D4959: Standard Test Method for Determination of Water (Moisture) Content of Soil By Direct Heating
This test method covers procedures for determining the water (moisture) content of soils by drying with direct heat, such as using a hotplate, stove, blowtorch, etc. This test method is applicable for most soil types. For some soils, such as those containing significant amounts of halloysite, mica, montmorillonite, gypsum, or other hydrated materials, highly organic soils or soils that contain dissolved solids, (such as salt in the case of marine deposits), this test method may not yield reliable water content values.

ASTM D4971: Standard Test Method for Determining In Situ Modulus of Deformation of Rock Using Diametrically Loaded 76-mm (3-in.) Borehole Jack

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This test method covers the estimation of in situ modulus of a rock mass at various depths and orientations. Information on time-dependent deformation may also be obtained.

ASTM D4972: Standard Test Method for pH of Soils
This test method covers the measurement of the pH of soils for uses other than for corrosion testing. Such measurements are used in the agricultural, environmental, and natural resources fields. This measurement determines the degree of acidity or alkalinity in soil materials suspended in water and a 0.01 *M* calcium chloride solution. Measurements in both liquids are necessary to fully define the soil's pH. This variable is useful in determining the solubility of soil minerals and the mobility of ions in the soil and assessing the viability of the soil-plant environment. A more detailed discussion of the usefulness of this parameter is not warranted here; however, it can be found in many discussions of the subject.

ASTM D4992: Standard Practice for Evaluation of Rock to be Used for Erosion Control
This practice covers the evaluation of rock to be used for erosion control. The complexity and extent of this evaluation will be governed by the size and design requirements of the individual project, the quantity and quality of rock required, and the potential risk for property damage or loss of human life.

ASTM D5030: Standard Test Method for Density of Soil and Rock in Place by the Water Replacement Method in a Test Pit
This test method covers the determination of the in-place density and unit weight of soil and rock using water to fill a lined test pit to determine the volume of the test pit. The use of the word "rock" in this test method is used to imply that the material being tested will typically contain particles larger than 3 in. (75 mm).

ASTM D5079: Standard Practices for Preserving and Transporting Rock Core Samples
These practices cover the preservation, transportation, storage, cataloging, retrieval, and post-test disposition of rock core samples obtained for testing purposes and geologic study. These practices apply to both hard and soft rock, but exclude ice and permafrost. These practices do not apply to those situations in which changes in volatile gas components, contamination of the pore fluids, or mechanical stress relaxation affect the intended use for the core.

ASTM D5080: Standard Test Method for Rapid Determination of Percent Compaction
This test method describes the procedure for rapidly determining the percent compaction and the variation from optimum moisture content of an in-place soil for use in controlling construction of compacted earth. These values are obtained by developing a three-point compaction curve at the same moisture content as the in-place soil without knowing the value of the moisture content. The soil used for the compaction curve is normally the same soil removed from the in-place density test. For the remainder of this designation, this test method will be referred to as the rapid method. This test method is normally performed for soils containing more than 15 % fines (minus No. 200 sieve size).

ASTM D5084: Standard Test Methods for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter

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These test methods cover laboratory measurement of the hydraulic conductivity (also referred to as *coefficient of permeability*) of water-saturated porous materials with a flexible wall permeameter at temperatures between about 15 and 30°C (59 and 86°F). Temperatures outside this range may be used, however, the user would have to determine the specific gravity of mercury and R_T (see 10.3) at those temperatures using data from *Handbook of Chemistry and Physics*. There are six alternate methods or hydraulic systems that may be used to measure the hydraulic conductivity. These hydraulic systems are as follows:
Method A—Constant Head
Method B—Falling Head, constant tailwater elevation
Method C—Falling Head, rising tailwater elevation
Method D—Constant Rate of Flow
Method E—Constant Volume-Constant Head (by mercury)
Method F—Constant Volume-Falling Head (by mercury), rising tailwater elevation
These test methods may be utilized on all specimen types (undisturbed, reconstituted, remolded, compacted, etc.) that have a hydraulic conductivity less than about 1×10^{-6} m/s (1×10^{-4} cm/s), providing the head loss requirements of are met. For the constant-volume methods, the hydraulic conductivity typically has to be less than about 1×10^{-7} m/s.

ASTM D5088: Standard Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites
This practice covers the decontamination of field equipment used in the sampling of soils, soil gas, sludges, surface water, and groundwater at waste sites which are to undergo both physical and chemical analyses.

ASTM D5089: Standard Test Method for Velocity Measurements of Water in Open Channels with Electromagnetic Current Meters
This test method is particularly used for measuring the velocity at a point in an open channel as part of a velocity-area traverse to determine the flow rate of water. To this end it should be used in conjunction with Test Method D 3858. A single axis probe with cosine response will suffice for most of these applications. **This test method is also useful in applications where the velocity itself (rather than a volumetric flow rate) is the desired end product.**

ASTM D5092: Standard Practice for Design and Installation of Groundwater Monitoring Wells
This practice describes a methodology for designing and installing conventional (screened and filter-packed) groundwater monitoring wells suitable for formations ranging from unconsolidated aquifers (i.e., sands and gravels) to granular materials having grain-size distributions with up to 50 % passing a #200 sieve and as much as 20 % clay-sized material (i.e., silty fine sands with some clay). Formations finer than this (i.e., silts, clays, silty clays, clayey silts) should not be monitored using conventional monitoring wells, as representative groundwater samples, free of artifactual turbidity, cannot be assured using currently available technology. Alternative monitoring technologies (not described in this practice) should be used in these formations.

ASTM D5093: Standard Test Method for Field Measurement of Infiltration Rate Using Double-Ring Infiltrometer with Sealed-Inner Ring

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This test method describes a procedure for measuring the infiltration rate of water through in-place soils using a double-ring infiltrometer with a sealed inner ring. This test method is useful for soils with infiltration rates in the range of 1×10^{-7} m/s to 1×10^{-10} m/s. When infiltration rates $\geq 1 \times 10^{-7}$ m/s are to be measured Test Method D 3385 shall be used.

ASTM D5102: Standard Test Method for Unconfined Compressive Strength of Compacted Soil-Lime Mixtures

This test method covers procedures for preparing, curing, and testing laboratory-compacted specimens of soil-lime and other lime-treated materials (Note 0) for determining unconfined compressive strength. This test method can be used for specimens prepared at the maximum unit weight and optimum water content, or for specimens prepared at other target unit weight and water content levels. Other applications are given in Section on Significance and Use. Note— Lime-based products other than commercial quicklime and hydrated lime are also used in the lime treatment of fine-grained cohesive soils. Lime kiln dust (LKD) is collected from the kiln exhaust gases by cyclone, electrostatic, or baghouse-type collection systems. Some lime producers hydrate various blends of LKD plus quicklime to produce a lime-based product. Cored specimens of soil-lime should be tested in accordance with Test Methods D 2166.

ASTM D5121: Standard Practice for Preparation of Rock Slabs for Durability Testing

This practice covers the preparation of rock slabs for various tests used to evaluate any visual internal defects, or the durability of rock by means of laboratory tests. These tests include, but are not limited to, Test Methods D 5240, D 5312, and D 5313.

ASTM D5126: Standard Guide for Comparison of Field Methods for Determining Hydraulic Conductivity in the Vadose Zone

This guide covers a review of the test methods for determining hydraulic conductivity in unsaturated soils and sediments. Test methods for determining both field-saturated and unsaturated hydraulic conductivity are described.

ASTM D5129: Standard Test Method for Open Channel Flow Measurement of Water Indirectly by Using Width Contractions

This test method is particularly useful to determine the discharge when it cannot be measured directly by some type of current meter to obtain velocities and with sounding weights to determine the cross section. Even under the best conditions, the personnel available cannot cover all points of interest during a major flood. The engineer or technician cannot always obtain reliable results by direct methods if the stage is rising or falling very rapidly, if flowing ice or debris interferes with depth or velocity measurements, or if the cross section of an alluvial channel is scouring or filling significantly. Under the worst conditions, access roads are blocked, cableways and bridges may be washed out, and knowledge of the flood frequently comes too late. Therefore, some type of indirect measurement is necessary. The contracted-opening method is commonly used on valley-floor streams.

ASTM D5130: Standard Test Method for Open-Channel Flow Measurement of Water Indirectly by Slope-Area Method

This test method is particularly useful for determining the discharge when it cannot be measured directly by some type of current meter to obtain velocities and with sounding weights to

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determine the cross section. Even under optimum conditions, the personnel available cannot cover all points of interest during a major flood. Field personnel cannot always obtain reliable results by direct methods if the stage is rising or falling very rapidly, if flowing ice or debris interferes with depth or velocity measurements. Under the worst conditions, access roads are blocked, cableways and bridges may be washed out, and knowledge of the flood frequently comes too late to obtain direct measurements of flow. Therefore, some type of indirect measurement is necessary. The slope-area method is a commonly used method.

ASTM D5143: Standard Test Method for Analysis of Nitroaromatic and Nitramine Explosive in Soil by High Performance Liquid Chromatography
This test method describes a procedure for the laboratory determination of the concentration of nitroaromatic and nitramine explosives in soil. The explosives involved in this test method are as follows: HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine), RDX (hexahydro-1,3,5-trinitrol-1,3,5-triazine), TNT (2,4,6-trinitrotoluene), TNB (1,3,5 trinitrobenzene), DNB (1,3 dinitrobenzene), tetryl (methyl-2,4,6-trinitrophenylnitramine), and 2,4-DNT (2,4-dinitrotoluene).

ASTM D5173: Standard Test Method for On-Line Monitoring of Carbon Compounds in Water by Chemical Oxidation, by UV Light Oxidation, by Both, or by High Temperature Combustion Followed by Gas Phase NDIR or by Electrolytic Conductivity
This test method covers the selection, establishment, and application of monitoring systems for carbon and carbon compounds by continual sampling or continuous flow-through, automatic analysis, and recording or otherwise signaling of output data. The system chosen will depend on the purpose for which it is intended (for example, regulatory compliance, process monitoring, or to alert the user to adverse trends) and on the type of water to be monitored (low purity or high purity, with or without suspended particulates, purgeable organics, or inorganic carbon). If it is to be used for regulatory compliance, the test method published or referenced in the regulations should be used in conjunction with this test method and other ASTM test methods. The test method covers carbon concentrations of 10 g/L to 5000 mg/L.

ASTM D5195: Standard Test Method for Density of Soil and Rock In-Place at Depths below Surface by Nuclear Methods
This test method covers the calculation of the density of soil and rock by the attenuation of gamma radiation, where the gamma source and the gamma detector are placed at the desired depth in a bored hole lined by an access tube.

ASTM D5202: Standard Test Method for Determining Triaxial Compression Creep Strength of Chemical Grouted Soils
This test method covers the determination of long-term strength and deformation of a cylindrical specimen of either a (undisturbed) field sample or laboratory-fabricated chemical grouted soil when it is sheared undrained in compression under a constant sustained load. Note —The voids of chemical grouted soils are most often substantially filled with grout. Thus, pore pressures are unlikely to develop. This test method is not applicable to partially grouted soils in which substantial pore pressures may develop. If pore pressures must be measured, reference is made to Test Method D 4767 for equipment and procedures.

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ASTM D5220: Standard Test Method for Water Mass per Unit Volume of Soil and Rock In-Place by the Neutron Depth Probe Method
This test method covers the calculation of the water mass per unit volume of soil and rock by thermalization or slowing of fast neutrons where the neutron source and the thermal neutron detector are placed at the desired depth in the bored hole lined by an access tube.

ASTM D5239: Standard Practice for Characterizing Fly Ash for Use in Soil Stabilization
This practice covers procedures for characterizing fly ash to be used in soil stabilization. This practice lists representative test methods for determining the chemical, physical, and cementitious properties of fly ash. A broad guideline is provided in that explains the significance of these properties in soil stabilization.

ASTM D5240: Standard Test Method for Testing Rock Slabs to Evaluate Soundness of Riprap by Use of Sodium Sulfate or Magnesium Sulfate
This test method covers test procedures for evaluating the soundness of riprap by the effects of a sodium or magnesium sulfate solution on slabs of rock.

ASTM D5242: Standard Test Method for Open-Channel Flow Measurement of Water with Thin-Plate Weirs
This test method covers measurement of the volumetric flow rate of water and wastewater in channels with thin-plate weirs. Information related to this test method can be found in Rantz and Ackers. The values stated in inch-pound units are to be regarded as the standard. The SI units given in parentheses are for information only.

ASTM D5243: Standard Test Method for Open-Channel Flow Measurement of Water Indirectly at Culverts
This test method covers the computation of discharge (the volume rate of flow) of water in open channels or streams using culverts as metering devices. In general, this test method does not apply to culverts with drop inlets, and applies only to a limited degree to culverts with tapered inlets. Information related to this test method can be found in ISO 748 and ISO 1070. This test method produces the discharge for a flood event if high-water marks are used. However, a complete stage-discharge relation may be obtained, either manually or by using a computer program, for a gauge located at the approach section to a culvert. The values stated in inch-pound units are to be regarded as the standard. The SI units given in parentheses are for information only.

ASTM D5254: Standard Practice for Minimum Set of Data Elements to Identify a Groundwater Site
This practice covers what information should be obtained for any individual groundwater site, also known as monitoring location or sampling station. As used in this practice, a site is meant to be a single point, not a geographic area or property. A groundwater site is defined as any source, location, or sampling station capable of producing water or hydrologic data from a natural stratum from below the surface of the earth. A source or facility can include a well, spring or seep, and drain or tunnel (nearly horizontal in orientation). Other sources, such as excavations, driven devices, bore holes, ponds, lakes, and sinkholes, that can be shown to be hydraulically connected to the groundwater, are appropriate for the use intended (see). Note 1There are many

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additional data elements that may be necessary to identify a site, but are not included in the minimum set of data elements. An agency or company may require additional data elements as a part of their minimum set.

ASTM D5255: Standard Practice for Certification of Personnel Engaged in the Testing of Soil and Rock

This practice provides a guide for evaluation and a certification procedure for personnel engaged in testing soil and rock in accordance with ASTM test methods and is intended for use by independent organizations providing certification services. Qualifications for certification candidates, qualifications of those administering the certification examinations, methods of administering the certification tests, and certain certification organization operating requirements are given.

ASTM D5268: Standard Specification for Topsoil Used for Landscaping Purposes

This specification covers a physical evaluation of an inorganic soil containing a limited amount of organic material, relative to its use as a topsoil for horticultural purposes in construction. For classification, a full agricultural textural classification may be used. The presence in the soil of the correct nutrients and pH status is necessary for healthy plant growth. This specification does not, however, cover a determination of the nutrients, nor their availability. Note-The nutrient content of topsoil is important and the nutrients usually evaluated are nitrogen, phosphate, and potassium. Nutrient deficiencies may be corrected using organic or inorganic fertilizers. Excess soluble salts should be examined as to their desirability. The acidity or alkalinity of the soil is also important. Excess acidity may be corrected by the application of lime. Excess alkalinity may be corrected by the application of sulfur or other suitable acidifying compounds. The latter item, in addition to lowering pH, also could be considered as an aggregate when considering the particle size distribution.

ASTM D5269: Standard Test Method for Determining Transmissivity of Nonleaky Confined Aquifers by the Theis Recovery Method

This test method covers an analytical procedure for determining the transmissivity of a confined aquifer. This test method is used to analyze data from the recovery of water levels following pumping or injection of water to or from a control well at a constant rate.

The analytical procedure given in this test method is used in conjunction with the field procedure in Test Method D 4050.

ASTM D5270: Standard Test Method for Determining Transmissivity and Storage Coefficient of Bounded, Nonleaky, Confined Aquifers

This test method covers an analytical procedure for determining the transmissivity, storage coefficient, and possible location of boundaries for a confined aquifer with a linear boundary. This test method is used to analyze water-level or head data from one or more observation wells or piezometers during the pumping of water from a control well at a constant rate. This test method also applies to flowing artesian wells discharging at a constant rate. With appropriate changes in sign, this test method also can be used to analyze the effects of injecting water into a control well at a constant rate. 1.2 The analytical procedure in this test method is used in conjunction with the field procedure in Test Method D 4050.

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ASTM D5298: Standard Test Method for Measurement of Soil Potential (Suction) Using Filter Paper

This test method covers laboratory filter papers as passive sensors to evaluate the soil matric (matrix) and total potential (suction), a measure of the free energy of the pore-water or tension stress exerted on the pore-water by the soil matrix **(1, 2)**[2]. The term potential or suction is descriptive of the energy status of soil water. This test method controls the variables for measurement of the water content of filter paper that is in direct contact with soil or in equilibrium with the partial pressure of water vapor in the air of an airtight container enclosing a soil specimen. The filter paper is enclosed with a soil specimen in the airtight container until moisture equilibrium is established; that is, the partial pressure of water vapor in the air is in equilibrium with the vapor pressure of pore-water in the soil specimen.

ASTM D5299: Standard Guide for Decommissioning of Groundwater Wells, Vadose Zone Monitoring Devices, Boreholes, and Other Devices for Environmental Activities

This guide covers procedures that are specifically related to permanent decommissioning (closure) of the following as applied to environmental activities. It is intended for use where solid or hazardous materials or wastes are found, or where conditions occur requiring the need for decommissioning. The following devices are considered in this guide:

1. A borehole used for geoenvironmental purposes
2. Monitoring wells
3. Observation wells
4. Injection wells
5. Piezometers
6. Wells used for the extraction of contaminated groundwater, the removal of floating or submerged materials other than water such as gasoline or tetrachloroethylene, or other devices used for the extraction of soil gas
7. A borehole used to construct a monitoring well
8. Any other vadose zone monitoring device.

ASTM D5311: Standard Test Method for Load Controlled Cyclic Triaxial Strength of Soil

This test method covers the determination of the cyclic strength (sometimes called the liquefaction potential) of saturated soils in either undisturbed or reconstituted states by the load-controlled cyclic triaxial technique. The cyclic strength of a soil is evaluated relative to a number of factors, including: the development of axial strain, magnitude of applied cyclic stress, number of cycles of stress application, development of excess pore-water pressure, and state of effective stress. A comprehensive review of factors affecting cyclic triaxial test results is contained in the literature .

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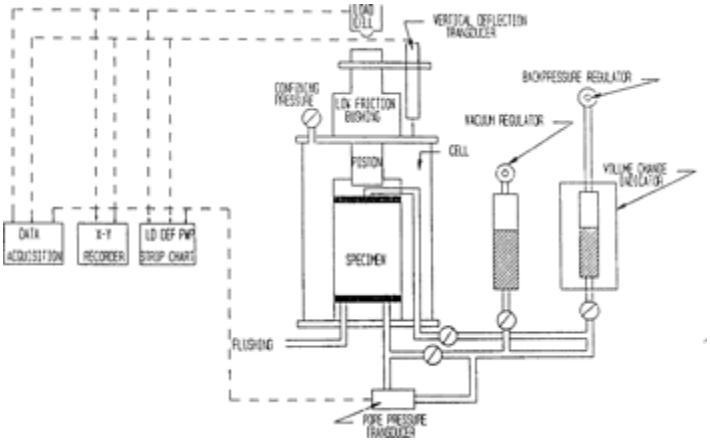


FIGURE 1 Schematic Representation of Load-Controlled Cyclic Triaxial Strength Test Equipment

ASTM D5312: Standard Test Method for Evaluation of Durability of Rock for Erosion Control under Freezing and Thawing Conditions

This test method covers the procedures for evaluating the durability of rock for erosion control when exposed to freezing and thawing conditions.

ASTM D5313: Standard Test Method for Evaluation of Durability of Rock for Erosion Control under Wetting and Drying Conditions

This test method covers procedures for evaluating the durability of rock for erosion control when exposed to wetting and drying conditions.

ASTM D5314: Standard Guide for Soil Gas Monitoring in the Vadose Zone

This guide covers information pertaining to a broad spectrum of practices and applications of soil atmosphere sampling, including sample recovery and handling, sample analysis, data interpretation, and data reporting. This guide can increase the awareness of soil gas monitoring practitioners concerning important aspects of the behavior of the soil-water-gas-contaminant system in which this monitoring is performed, as well as inform them of the variety of available techniques of each aspect of the practice. Appropriate applications of soil gas monitoring are identified, as are the purposes of the various applications. Emphasis is placed on soil gas contaminant determinations in certain application examples. This guide suggests a variety of approaches useful to successfully monitor vadose zone contaminants with instructions that offer direction to those who generate and use soil gas data.

ASTM D5333: Standard Test Method for Measurement of Collapse Potential of Soils

This test method covers the determination of the magnitude of one-dimensional collapse that occurs when unsaturated soils are inundated with fluid.

This test method may be used to determine the magnitude of potential collapse that may occur for a given vertical (axial) stress and an index for rating the potential for collapse.

ASTM D5334: Standard Test Method for Determination of Thermal Conductivity of Soil and Soft Rock by Thermal Needle Probe Procedure

This test method presents a procedure for determining the thermal conductivity of soil and soft rock using a transient heat method. This test method is applicable for both undisturbed and

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remolded soil specimens and soft rock specimens. This test method is suitable only for isotropic materials. This test method is applicable to dry materials over a wide temperature range from <0 to >100°C, depending on the suitability of the thermal needle probe construction to temperature extremes. This method may also be used for specimens containing moisture. However, care must be taken to prevent significant error from: 1) redistribution of water due to thermal gradients resulting from heating of the needle probe, and 2) phase change (melting) of ice in specimens with temperatures <0°C. Both of these errors can be minimized by adding less total heat to the specimen either through minimizing power applied to the needle probe and/or minimizing the heating duration of the measurement. For satisfactory results in conformance with this test method, the principles governing the size, construction, and use of the apparatus described in this test method should be followed. If the results are to be reported as having been obtained by this test method, then all pertinent requirements prescribed in this test method shall be met.
It is not practicable in a test method of this type to aim to establish details of construction and procedure to cover all contingencies that might offer difficulties to a person without technical knowledge concerning the theory of heat flow, temperature measurement, and general testing practices. Standardization of this test method does not reduce the need for such technical knowledge. It is recognized also that it would be unwise, because of the standardization of this test method, to resist in any way the further development of improved or new methods or procedures by research workers. The values stated in SI units are to be regarded as the standard. The inch-pound units given in parentheses are for information only.
All measured and calculated values shall conform to the guidelines for significant digits and rounding established In Practice D 6026. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

ASTM D5335: Standard Test Method for Linear Coefficient of Thermal Expansion of Rock Using Bonded Electric Resistance Strain Gauges
This test method covers the laboratory determination of the linear (one-dimensional) coefficient of thermal expansion of rock using bonded electric resistance strain gauges. This test method is applicable for unconfined pressure conditions over the temperature range from 20 to 260°C (68 to 500°F). Note 1—Unconfined tests performed at elevated temperatures may alter the mineralogy or grain structure of the test specimen. This alteration may change the physical and thermal properties of the test specimen. Note 2—The strain gauges are mounted with epoxy. Most commercially available high temperature epoxies require elevated temperature curing. The elevated temperature required for this curing may alter the physical and thermal properties of the test specimen. Epoxy should be selected based upon the maximum expected test temperature. Room temperature curing epoxy should be used whenever possible.

ASTM D5389: Standard Test Method for Open-Channel Flow Measurement by Acoustic Velocity Meter Systems
This test method is used where high accuracy of velocity or continuous discharge measurement over a long period of time is required and other test methods of measurement are not feasible due to low velocities in the channel, variable stage-discharge relations, complex stage-discharge relations, or the presence of marine traffic. It has the additional advantages of requiring no moving parts, introducing no head loss, and providing virtually instantaneous readings (1 to 100

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readings per second). The test method may require a relatively large amount of site work and survey effort and is therefore most suitable for permanent or semi-permanent installations.
This test method covers the measurement of flow rate of water in open channels, streams, and closed conduits with a free water surface. The test method covers the use of acoustic transmissions to measure the average water velocity along a line between one or more opposing sets of transducers-by the time difference or frequency difference techniques.

ASTM D5390: Standard Test Method for Open-Channel Flow Measurement of Water with Palmer-Bowlus Flumes
This test method covers measurement of the volumetric flow rate of water and wastewater in sewers and other open channels with Palmer-Bowlus flumes. The values stated in inch-pound units are to be regarded as the standard. The SI units given in parentheses are for information only. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Withdrawn Standard: ASTM D5407: Standard Test Method for Elastic Moduli of Undrained Intact Rock Core Specimens in Triaxial Compression Without Pore Pressure Measurement (Withdrawn 2005)
This test method covers the determination of elastic moduli of intact rock core specimens in undrained triaxial compression. It specifies the apparatus, instrumentation, and procedures for determining the stress-axial strain and the stress-lateral strain curves, as well as Young's modulus, E, and Poisson's ratio, v. Note 1-This test method does not include the procedures necessary to obtain a stress-strain curve beyond the ultimate strength. For an isotropic material, the relation between the shear and bulk moduli and Young's modulus and Poisson's ratio are:
Equation 1 - $G = E/2(1 + v)$ Equation 2 - $K = E/3(1 - 2v)$
where:
G = shear modulus,
K = bulk modulus,
E = Young's modulus, and
v = Poisson's ratio.
The engineering applicability of these equations is decreased if the rock is anisotropic. When possible, it is desirable to conduct tests in the plane of foliation, bedding, etc., and at right angles to it to determine the degree of anisotropy. It is noted that equations developed for isotropic materials may give only approximate calculated results if the difference in elastic moduli in any two directions is greater than 10 % for a given stress level. Note 2- Elastic moduli measured by sonic methods may often be employed as preliminary measures of anisotropy.

ASTM D5408: Standard Guide for Set of Data Elements to Describe a Groundwater Site; Part One-Additional Identification Descriptors
This guide covers Part One of three guides to be used in conjunction with Practice D 5254 that delineates the data desirable to describe a groundwater data collection or sampling site. This guide describes additional information beyond the minimum set of data elements that may be needed to identify a groundwater site. Part Two identifies physical descriptors, such as construction, for a site, while Part Three identifies usage descriptors, such as monitoring, for an individual groundwater site.

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ASTM D5409: Standard Guide for Set of Data Elements to Describe a Groundwater Site; Part Two-Physical Descriptors
This guide covers Part Two of three guides to be used in conjunction with Practice D 5254 that delineates the data desirable to describe a groundwater data collection or sampling site. This guide identifies physical descriptors, such as construction and geologic elements, for a site. Part One (Guide D 5408) describes additional information beyond the minimum set of data elements that may be specified to identify any individual groundwater site, while Part Three identifies usage descriptors, such as monitoring, for an individual groundwater site.

ASTM D5410: Standard Guide for Set of Data Elements to Describe a Groundwater Site; Part Three-Usage Descriptors
This guide is Part Three of three guides to be used in conjunction with Practice D 5254 that delineates the data desirable to describe a groundwater data collection or sampling site. This guide identifies usage descriptors, such as monitoring, for an individual groundwater site. Guide D 5408 describes additional information beyond the minimum set of data elements that may be specified to identify a groundwater site, while Guide D 5409 identifies physical descriptors, such as construction, for a site.

ASTM D5434: Standard Guide for Field Logging of Subsurface Explorations of Soil and Rock
This guide describes the type of information that should be recorded during field subsurface explorations in soil and rock. This guide is not intended to specify all of the information required for preparing field logs. Such requirements will vary depending on the purpose of the investigation, the intended use of the field log, and particular needs of the client or user. This guide is applicable to boreholes, auger holes, excavated pits, or other subsurface exposures such as road side cuts or stream banks. This guide may serve as a supplement to Guide D 420.

ASTM D5435: Standard Test Method for Diagnostic Soil Test for Plant Growth and Food Chain Protection
This test method covers the determination of quantity (Q) and intensity (I) results for several elements in soils, spoils, fly-ash, and other soil substitutes to ascertain their suitability for the growth of vegetation and possible adverse effects of metals on the food chain.

ASTM D5447: Standard Guide for Application of a Groundwater Flow Model to a Site-Specific Problem
This guide covers the application and subsequent documentation of a groundwater flow model to a particular site or problem. In this context, "groundwater flow model" refers to the application of a mathematical model to the solution of a site-specific groundwater flow problem. This guide illustrates the major steps to take in developing a groundwater flow model that reproduces or simulates an aquifer system that has been studied in the field. This guide does not identify particular computer codes, software, or algorithms used in the modeling investigation.
This guide is specifically written for saturated, isothermal, groundwater flow models. The concepts are applicable to a wide range of models designed to simulate subsurface processes, such as variably saturated flow, flow in fractured media, density-dependent flow, solute

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transport, and multiphase transport phenomena; however, the details of these other processes are not described in this guide.

ASTM D5472: Standard Test Method for Determining Specific Capacity and Estimating Transmissivity at the Control Well
This test describes a procedure for conducting a specific capacity test, computing the specific capacity of a control well, and estimating the transmissivity in the vicinity of the control well. Specific capacity is the well yield per unit drawdown at an identified time after pumping started.

ASTM D5473: Standard Test Method for (Analytical Procedure for) Analyzing the Effects of Partial Penetration of Control Well and Determining the Horizontal and Vertical Hydraulic Conductivity in a Nonleaky Confined Aquifer
This test method covers an analytical solution for determining the horizontal and vertical hydraulic conductivity of an aquifer by analysis of the response of water levels in the aquifer to the discharge from a well that partially penetrates the aquifer.

ASTM D5474: Standard Guide for Selection of Data Elements for Groundwater Investigations
This guide covers the selection of data elements for the documentation of groundwater sites. The data elements are described in four ASTM standards outlining information that may be collected at groundwater sites. Examples of specific investigations are given with the logic of why to select individual and combinations of data elements to meet the requirements of the studies. Note 1A groundwater site is any source, location, or sampling station capable of producing water or hydrologic data from a natural stratum from below the surface of the earth. A source or facility can include a well, spring or seep, and drain or tunnel (nearly horizontal in orientation). Other sources, such as excavations, driven devices, bore holes, ponds, lakes, and sinkholes, that can be shown to be hydraulically connected to the groundwater, are appropriate for the use intended. The four ASTM standards that describe the data elements for groundwater are Practice D 5254 and Guides D 5408, D 5409, and D 5410.

ASTM D5490: Standard Guide for Comparing Groundwater Flow Model Simulations to Site-Specific Information
This guide covers techniques that should be used to compare the results of groundwater flow model simulations to measured field data as a part of the process of calibrating a groundwater model. This comparison produces quantitative and qualitative measures of the degree of correspondence between the simulation and site-specific information related to the physical hydrogeologic system.

ASTM D5518: Standard Guide for Acquisition of File Aerial Photography and Imagery for Establishing Historic Site-Use and Surficial Conditions (Withdrawn 2003)
This guide is intended to assist potential users in the search for, evaluation of, and acquisition of remotely sensed aerial photography or imagery, or both, to be used for the purpose of establishing the historic site-use and other interpretable surface or near-surface conditions regionally, locally, or at a specified project location.

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ASTM D5519: Standard Test Methods for Particle Size Analysis of Natural and Man-Made Riprap Materials
These test methods cover the particle size and mass analysis of natural and man-made riprap and related materials, including filter stone or coarse bedding materials. These test methods are generally intended for riprap and related materials. They are applicable for mixtures of stones screened from natural deposits, blast rock, processed materials from quarried rock, or recycled concrete. They are applicable for sizes 3 in. (75 mm) and above, with the upper size limited only by equipment available for handling and weighing the individual particles. Four alternate procedures are provided. There is a wide range in the level of effort and the precision of the test procedures. It is important for specifiers to indicate the test procedure.

ASTM D5520: Standard Test Method for Laboratory Determination of Creep Properties of Frozen Soil Samples by Uniaxial Compression
This test method covers the determination of the creep behavior of cylindrical specimens of frozen soil, subjected to uniaxial compression. It specifies the apparatus, instrumentation, and procedures for determining the stress-strain-time, or strength versus strain rate relationships for frozen soils under deviatoric creep conditions. Although this test method is one that is most commonly used, it is recognized that creep properties of frozen soil related to certain specific applications, can also be obtained by some alternative procedures, such as stress-relaxation tests, simple shear tests, and beam flexure tests. Creep testing under triaxial test conditions will be covered in another standard.

ASTM D5521: Standard Guide for Development of Groundwater Monitoring Wells in Granular Aquifers
This guide covers the development of screened wells installed for the purpose of obtaining representative groundwater information and water quality samples from granular aquifers, though the methods described herein could also be applied to wells used for other purposes. Other well-development methods that are used exclusively in open-borehole bedrock wells are not described in this guide. The applications and limitations of the methods described in this guide are based on the assumption that the primary objective of the monitoring wells to which the methods are applied is to obtain representative water quality samples from aquifers. Screened monitoring wells developed using the methods described in this guide should yield relatively sediment-free samples from granular aquifer materials, ranging from gravels to silty sands. While many monitoring wells are considered "small-diameter" wells (that is, less than four inches in inside diameter), some of the techniques described in this guide will be more easily applied to large-diameter wells (that is, four-inches or greater in inside diameter).

ASTM D5522: Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid (Withdrawn 2004)
This specification covers specific criteria for evaluating the technical capabilities of laboratories involved in testing, measuring, inspecting, and calibrating activities related to chemical analysis of earth materials. In this specification, earth materials shall mean soil, rock, and contained fluids. For the sake of brevity, the term "laboratory" is used in this practice to represent all the above.
This specification addresses the minimum requirements for laboratories that analyze earth materials for metals, volatile organic compounds, semivolatile organic compounds, pesticides,

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herbicides, PCBs, radionuclides, and various other parameters by miscellaneous wet chemistry techniques.

ASTM D5539: Standard Specification for Seed Starter Mix
This specification covers the material characteristics, physical requirements, and sampling appropriate to the designation of a soil or soil-like material for seed starting purposes. This would consist of the duration of germination ending after the cotyledon stage (at the opening of the first true leaves). Seed starter mix may include natural soils and artificial mixtures of natural or artificial materials used for the initial germination of seeds to the cotyledon stage.

ASTM D5543: Standard Test Methods for Low-Level Dissolved Oxygen in Water
This test method covers the determination of low-level (<100µ g/L) dissolved oxygen in thermal-cycle steam condensate, deaerated boiler feedwater, boiler water, and deaerated deionized water. The following test method is included:

	Range, µg/L	Sections
Color Comparator Test Method Using Self-Filling Glass Ampoules	0 to 100	8 to 17

This test method may be applicable to electronic-grade, pharmaceutical-grade, and other high-purity waters, although these were not addressed in the collaborative study. This test method is a colorimetric procedure applicable to dissolved oxygen in water in the range from 0 to 100 µg/L. It is the user's responsibility to ensure the validity of these test methods for waters of untested matrices.

ASTM D5550: Standard Test Method for Specific Gravity of Soil Solids by Gas Pycnometer
This test method covers the determination of the specific gravity of soil solids by means of a gas pycnometer. Particle size is limited by the dimensions of the specimen container of the particular pycnometer being used.

ASTM D5607: Standard Test Method for Performing Laboratory Direct Shear Strength Tests of Rock Specimens Under Constant Normal Force
This test method establishes requirements and laboratory procedures for performing direct shear strength tests on rock specimens. It includes procedures for both intact rock strength and sliding friction tests which can be performed on specimens that are homogeneous, or have planes of weakness, including natural or artificial discontinuities. Examples of an artificial discontinuity include a rock-concrete interface or a lift line from a concrete pour. Discontinuities may be open, partially or completely healed or filled (that is, clay fillings and gouge). Only one discontinuity per specimen can be tested. The test is usually conducted in the undrained state with an applied constant normal load. However, a clean, open discontinuity may be free draining, and, therefore, a test on a clean, open discontinuity could be considered a drained test. During the test, shear strength is determined at various applied stresses normal to the sheared plane and at various shear displacements. Relationships derived from the test data include shear strength versus normal stress and shear stress versus shear displacement (shear stiffness).

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ASTM D5608: Standard Practices for Decontamination of Field Equipment Used at Low Level Radioactive Waste Sites

This practice covers the decontamination of field equipment used in the sampling of soils, soil gas, sludges, surface water, and groundwater at waste sites known or suspected of containing low level radioactive wastes. This practice is applicable at sites where low level radioactive wastes are known or suspected to exist. This practice may also be applicable for the decontamination of equipment used in known or suspected transuranic, or mixed wastes when used by itself or in conjunction with Practice D 5088. Procedures are contained in this practice for the decontamination of equipment that comes into contact with the sample matrix (sample contacting equipment), and for ancillary equipment that has not contacted the sample, but may have become contaminated during use (non-contacting equipment). This practice is applicable to most conventional sampling equipment constructed of metallic and hard, smooth synthetic materials. Materials with rough or porous surfaces, or having a high sorption rate should not be used in radioactive waste sampling due to the difficulties with decontamination.

ASTM D5609: Standard Guide for Defining Boundary Conditions in Groundwater Flow Modeling

This guide covers the specification of appropriate boundary conditions that are an essential part of conceptualizing and modeling groundwater systems. This guide describes techniques that can be used in defining boundary conditions and their appropriate application for modeling saturated groundwater flow model simulations. This guide is one of a series of standards on groundwater flow model applications. Defining boundary conditions is a step in the design and construction of a model that is treated generally in Guide D 5447.

ASTM D5610: Standard Guide for Defining Initial Conditions in Groundwater Flow Modeling

This guide covers techniques and procedures used in defining initial conditions for modeling saturated groundwater flow. The specification of initial conditions is an essential part of conceptualizing and modeling groundwater systems.

ASTM D5611: Standard Guide for Conducting a Sensitivity Analysis for a Groundwater Flow Model Application

This guide covers techniques that should be used to conduct a sensitivity analysis for a groundwater flow model. The sensitivity analysis results in quantitative relationships between model results and the input hydraulic properties or boundary conditions of the aquifers. After a groundwater flow model has been calibrated, a sensitivity analysis may be performed. Examination of the sensitivity of calibration residuals and model conclusions to model inputs is a method for assessing the adequacy of the model with respect to its intended function. This guide presents the simplest acceptable techniques for conducting a sensitivity analysis. Other techniques have been developed by researchers and could be used in lieu of the techniques in this guide. This guide is written for performing sensitivity analyses for groundwater flow models. However, these techniques could be applied to other types of groundwater related models, such as analytical models, multi-phase flow models, non-continuum (karst or fracture flow) models, or mass transport models.

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ASTM D5612: Standard Guide for Quality Planning and Field Implementation of a Water Quality Measurement Program

This guide covers planning and implementation of the sampling aspects of environmental data generation activities. Environmental data generation efforts are comprised of four parts: 1) establishment of data quality objectives (DQOs); 2) design of field sampling and measurement strategies and specification of laboratory analyses and data acceptance criteria; 3) implementation of sampling and analysis strategies; and (4) data quality assessment. This guide defines the criteria that must be considered to ensure the quality of the field aspects of environmental data and sample generation activities. DQOs should be adopted prior to the application of this guide. The data generated in accordance with this guide are subject to a final assessment to determine whether the DQOs were met. For example, many screening activities do not require all of the quality assurance (QA) and quality control (QC) steps found in this guide to generate data adequate to meet the project needs. The extent to which all of the requirements must be met remains a matter of technical judgment as it relates to the established DQOs. This guide presents extensive management requirements designed to ensure high-quality samples and data. The words “must,”“ shall,” “may,” and “should” have been selected carefully to reflect the importance placed on many of the statements made in this guide. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

ASTM D5714: Standard Specification for Content of Digital Geospatial Metadata

This specification covers the information content of metadata for a set of digital geospatial data. This specification provides a common set of terminology and definitions for concepts related to these metadata. The use of the term "geographic information system" and its definition in this specification is not intended to introduce a standard definition. This specification covers minimum content and processing requirements for geospatial metadata. There are at least three categories of use for geospatial metadata: 1) to accompany data transfers as documentation, 2) internal, on-line documentation of processing steps and data lineage, and 3) as stand-alone data set synopses for use by spatial data catalogs, indexes, and referral services.

ASTM D5715: Standard Test Method for Estimating the Degree Of Humidification of Peat And Other Organic Soils (Visual/Manual Method)

This test method covers a system for visually discriminating peat and other highly organic soils on the basis of degree of humification.

ASTM D5716: Standard Test Method for Measuring the Rate Of Well Discharge by Circular Orifice Weir

This test method covers construction and operation of a circular orifice weir for measuring the discharge from a well. This test method is a part of a series of standards prepared on in situ determination of hydraulic properties of aquifer systems by single- or multiple- well tests. Selection of a well discharge measurement test is described in guide D5737.

ASTM D5717: Standard Guide for Design of Groundwater Monitoring Systems in Karst and Fractured-Rock Aquifers

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This guide considers the characterization of karst and fractured-rock aquifers as an integral component of monitoring-system design. Hence, the development of a conceptual hydrogeologic model that identifies and defines the various components of the flow system is recommended prior to the design and implementation of a monitoring system.

ASTM D5718: Standard Guide for Documenting a Groundwater Flow Model Application
This guide covers suggested components to be included in documenting and archival of numerical groundwater flow model applications. Model documentation includes a written and graphical presentation of model assumptions and objectives, the conceptual model, code description, model construction, model calibration, predictive simulations, and conclusions. Model archival refers to a file or a set of files (in both written and digital format) that contains logs of significant model simulations (that is, calibration, sensitivity and prediction simulations), supplemental calculations, model documentations, a copy of the model source code(s) or executable files(s) used, or both, and input and output data sets for significant model simulations.

ASTM D5719: Standard Guide for Simulation of Subsurface Airflow Using Groundwater Flow Modeling Codes
This guide covers the use of a groundwater flow modeling code to simulate the movement of air in the subsurface. This approximation is possible because the form of the groundwater flow equations is similar in form to airflow equations. Approximate methods are presented that allow the variables in the airflow equations to be replaced with equivalent terms in the groundwater flow equations. The model output is then transformed back to airflow terms.

ASTM D5720: Standard Practice for Static Calibration of Electronic Transducer-Based Pressure Measurement Systems for Geotechnical Purposes
This practice covers the procedure for static calibration of electronic transducer-based systems used to measure fluid pressures in laboratory or in field applications associated with geotechnical testing.

ASTM D5730: Standard Guide for Site Characterization for Environmental Purposes with Emphasis on Soil, Rock, the Vadose Zone and Groundwater
This guide covers a general approach to planning field investigations that is useful for any type of environmental investigations with a primary focus on the subsurface and major factors affecting the surface and subsurface environment. Generally, such investigations should identify and locate, both horizontally and vertically, significant soil and rock masses and groundwater conditions present within a given site area and establish the characteristics of the subsurface materials by sampling or in situ testing, or both. The extent of characterization and specific methods used will be determined by the environmental objectives and data quality requirements of the investigation. This guide focuses on field methods for determining site characteristics and collection of samples for further physical and chemical characterization. This guide does not address special considerations required for characterization of karst and fractured rock terrain. In such hydrogeologic settings refer to Quinlan and Guide D 5717.

ASTM D5731: Standard Test Method for Determination of the Point Load Strength Index of Rock

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This test method covers the guidelines, requirements, and procedures for determining the point load strength index of rock. Specimens in the form of rock cores, blocks, or irregular lumps can be tested by this method. This test method can be preformed in the field or laboratory because the testing machine is portable. This is an index test and is intended to be used to classify and characterize rock.

ASTM D5737: Standard Guide for Methods for Measuring Well Discharge
This guide covers an overview of methods to measure well discharge. This guide is an integral part of a series of standards prepared on the in-situ determination of hydraulic properties of aquifer systems by single- or multiple-well tests. Measurement of well discharge is a common requirement to the determination of aquifer and well hydraulic properties.

ASTM D5738: Standard Guide for Displaying The Results of Chemical Analyses of Groundwater Major Ions and Trace Elements- Diagrams for Single Analyses
This guide covers the category of water-analysis diagrams that use pictorial or pattern methods (for examples, bar, radiating vectors, pattern and circular) as a basis for displaying each individual chemical components that were determined from the analysis of a single sample of natural groundwater.

ASTM D5753: Standard Guide for Planning and Conducting Borehole Geophysical Logging
This guide covers the documentation and general procedures necessary to plan and conduct a geophysical log program as commonly applied to geologic, engineering, groundwater, and environmental (hereafter referred to as geotechnical)investigations. It is not intended to describe the specific or standard procedures for running each type of geophysical log and is limited to measurements in a single borehole. It is anticipated that standard guides will be developed for specific methods subsequent to this guide.

ASTM D5754: Standard Guide for Displaying the Results of Chemical Analyses of Groundwater Major Ions and Trace Elements- Trilinear Diagrams for two or more Analyses
This guide describes the category of water analysis diagrams that use two-dimensional trilinear graphs as a technique for displaying the common chemical components from two or more complete analyses of natural groundwater on a single diagram. This category includes not only trilinear-shaped diagrams but also the diamond- or parallelogram-, rectangular-, or square-shaped graphs that have trilinear subdivisions.

ASTM D5777: Standard Guide for Using the Seismic Refraction Method for Subsurface Investigation
This guide summarizes the equipment, field procedures, and interpretation methods for the assessment of subsurface conditions using the seismic refractions method. Seismic refraction measurements as described in this guide are applicable in mapping subsurface conditions for various uses including geologic, geotechnical, hydrologic, environmental, mineral exploration, petroleum exploration, and archaeological investigations. The seismic refraction method is used to map geologic conditions including depth to bedrock, or water table, stratigraphy, lithology, structure and fractures or all of these. The calculated seismic wave velocity is related to

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mechanical material properties. Therefore characterization of the material (type of rock, degree of weathering, and rippability) is made on the basis of seismic velocity and other geologic information.

ASTM D5778: Standard Test Method for Performing Electronic Friction Cone and Piezocone Penetration Testing of Soils
This test method covers the procedure for determining the resistance to penetration of a conical pointed penetrometer as it is advanced into subsurface soils at a slow, steady rate.

ASTM D5779: Standard Test Method for Field Determination of Apparent Specific Gravity of Rock and Manmade For Erosion Control
This test method covers the determination of specific gravity of rocks for erosion control. This test method can be used for all types of materials, both naturally occurring or manmade.

ASTM D5780: Standard Test Method for Individual Piles in Permafrost under Static Axial Compressive Load
This test method covers procedures for testing individual vertical piles to determine response of the pile to static compressive load applied axially to the pile. This test method is applicable to all deep foundation units in permafrost that function in a manner similar to piles regardless of their method of installation.

ASTM D5781: Standard Guide for use of Dual-Wall Reverse-Circulation Drilling for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices
This guide covers how dual-wall reverse-circulation drilling may be used for geoenvironmental exploration and installation of subsurface water-quality monitoring devices.

ASTM D5782: Standard Guide for use of Direct Air-Rotary Drilling for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices
This guide covers how direct (straight) air-rotary drilling procedures may be used for geoenvironmental exploration and installation of subsurface water-quality monitoring devices.

ASTM D5783: Standard Guide for use of Direct Rotary Drilling with Water-Based Drilling Fluid for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices
This guide covers how direct (straight) rotary-drilling procedures with water-based drilling fluids may be used for geoenvironmental exploration and installation of subsurface water-quality monitoring devices.

ASTM D5784: Standard Guide for use of Hollow-Stem Augers for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices
This guide covers how hollow-stem auger-drilling system may be used for geoenvironmental exploration and installation of subsurface water-quality monitoring devices.

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ASTM D5785: Standard Test Method for (Analytical Procedure) Determining Transmissvity of Confined Nonleaky Aquifers by Underdamped Well Response to Instantaneous Change in Head (Slug Test)
This test method covers determination of transmissivity from the measurement of the damped oscillation about the equilibrium water level of a well-aquifer system to a sudden change of water level in a well. Underdamped response of water level in a well to a sudden change in water level is characterized by oscillatory fluctuation about the static water level with a decrease in the magnitude of fluctuation and recovery to initial water level. Underdamped response may occur in wells tapping highly transmissive confined aquifers and in deep wells having long water columns.

ASTM D5786: Standard Practice for (Field Procedure) for Constant Drawdown Tests in Flowing Wells for Determining Hydraulic Properties of Aquifer Systems
This practice covers the methods for controlling draw-down and measuring discharge rates and head to analyze the hydraulic of an aquifer or aquifers.

ASTM D5787: Standard Practice for Monitoring Well Protection
This practice identifies design and construction considerations to be applied to monitoring wells for protection from natural and man caused damage or impacts.

ASTM D5850: Standard Test Method for (Analytical Procedure) Determining Transmissivity, Storage Coefficient, and Anisotropy Ratio from a Network of Partially Penetrating Wells
This test method covers an analytical procedure for determining the transmissivity, storage coefficient, and ratio of vertical to horizontal hydraulic conductivity of a confined aquifer using observation well drawdown measurements from a constant-rate pumping test. This test method uses data from a minimum of four partially penetrating, properly positioned observation wells around a partially penetrating control well.

ASTM D5851: Standard Guide for Planning and Implementing a Water Monitoring Program
This guide is generic in its application to surface or groundwater, rivers, lakes, or estuaries (quantity and quality). It proposes a series of options that offer direction without recommending a definite course of action and discusses the major elements that are common to all purposes of water monitoring. The elements described are applicable whether the monitoring is only for one location or integrates multiple measurement sites for the purpose of assessing a whole watershed, estuary, or aquifer system. This guide is intended to outline for planners and administrators the components, process, and procedures which should be considered when proposing, planning, or implementing a monitoring program. The guide is not a substitute for obtaining specific technical advice. The reader is not assumed to be a technical practitioner in the water field; however, practitioners will find it a good summary of practice and a handy checklist. Other standard guides have or will be prepared that address the necessary detail.

ASTM D5852: Standard Test Method for Erodibility Determination of Soil in the Field or in the Laboratory by the Jet Index Method

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This test method covers the estimation of erodibility of a soil by a jet index method. This test method involves either preparing a field site or obtaining a relatively undisturbed soil sample and the subsequent activities for the determination of the erodibility of soil. This test method also may be run on compacted samples in the laboratory.

ASTM D5855: Standard Test Method for (Analytical Procedure) For Determining Transmissivity, Storage Coefficient, of a Confined Nonleaky or Leaky Aquifer by Constant Drawdown Method in a Flowing Well
This test method covers an analytical solution for determining transmissivity and storage coefficient of a leaky or non leaky confined aquifer. It is used to analyze data on the flow rate from a control well while a constant head is maintained in the well.

ASTM D5856: Standard Test Method for Measurement of Hydraulic Conductivity of Porous Material using a Rigid-Wall, Compaction-Mold Permeameter
This test method covers laboratory measurement of the hydraulic conductivity (also referred to as coefficient of permeability) of laboratory-compacted materials with a rigid-wall, compaction-mold permeameter.

ASTM D5872: Standard Guide for use of Casing Advancement Drilling Methods for Geoenvironmental Exploration and Installation of Subsurface Water-Quality Monitoring Devices
This guide covers how casing-advancement drilling and sampling procedures may be used for geoenvironmental exploration and installation of subsurface water-quality monitoring devices.

ASTM D5873: Standard Test Method for Determination of Rock Hardness by Rebound Hammer Method
This test method covers the testing apparatus, sampling, test specimen preparation, and testing procedures for determining the rebound hardness number of rock material using a spring-driven steel hammer, referred to variously as a rebound hammer, impact test hammer, or concrete test hammer.

ASTM D5874: Standard Test Method for Determination of the Impact Value (IV) of a Soil
This test method covers the determination of the impact value (iv) of a soil either in the field or a test mold.

ASTM D5875: Standard Guide for use of Cable-Tool Drilling and Sampling Methods for Geoenvironmental Exploration and Installation of Subsurface Water-Quality Monitoring Devices
This guide covers cable-tool drilling and sampling procedures used for geoenvironmental exploration and installation of subsurface water-quality monitoring devices.

ASTM D5876: Standard Guide for use of Direct Rotary Wireline Casing Advancement Drilling Methods for Geoenvironmental Exploration and Installation of Subsurface Water-Quality Monitoring Devices

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This guide covers how direct (straight) wireline rotary casing advancement drilling and sampling procedures may be used for geoenvironmental exploration and installation of subsurface water-quality monitoring devices.

ASTM D5877: Standard Guide for Displaying Results of Chemical Analyses of Groundwater for Major Irons and Trace Elements-Diagrams Based On Data Analytical Calculations

This guide covers methods that graphically display chemical analyses of multiple groundwater samples, discrete values and also those reduced to comprehensive summaries or parameters. Details required by the investigator to fully use the methods that are found in the listed references. The methods included in this guide are many of the graphical procedures that were not discussed in two previous guides, guides D 5738 and D 5754.

ASTM D5878: Standard Guide for using Rock-Mass Classification Systems for Engineering Purposes

This guide covers the selection of a suitable system of classification of rock mass for specific engineering purposes, such as tunneling and shaft-sinking, excavation of rock chambers, ground support, modification and stabilization of rock slopes, and preparation of foundations and abutments. These classification systems may also be of use in work on rippability of rock, quality of construction materials, and erosion resistance. Although widely used classification systems are treated in this guide, systems not included here may be more appropriate in some situations, and may be added to subsequent editions of this standard.

ASTM D5879: Standard Practice for Surface Site Characterization for On-Site Septic Systems

This practice covers procedures for the characterization of surface conditions at a site for evaluating suitability for an onsite septic system for disposal and treatment of wastewater. This practice provides a method for identifying potentially suitable areas for soil absorption of septic tank wastewater.

ASTM D5880: Standard Guide for Subsurface Flow and Transport Modeling

This guide covers an overview of subsurface fluid-flow (groundwater) modeling. The term subsurface fluid flow is used to reduce misunderstanding regarding groundwater, soil aqueous phase liquids. Increased understanding of fluid-flow phenomena is the combined result of field investigations and theoretical development of mathematical methods for modeling viscous fluids and air flow, in addition to water that are practical and appropriate.

ASTM D5881: Standard Test Method for (Analytical Procedure) Determining Transmissivity, Confined Nonleaky Aquifers by Critically Damped Well Response to Instantaneous Change in Head (Slug)

This test method covers determination of transmissivity from the measurement of water-level response to a sudden change of water level in a well-aquifer system characterized as being critically damped or in the transition range from under-damped to overdamped. Underdamped response is characterized by oscillatory changes in water level; overdamped response is characterized by return of water level to initial static level in an approximately exponential

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manner. Overdamped response is covered in guide D 4043; underdamped response is covered in D 5785.

ASTM D5882: Standard Test Method for Low Strain Integrity Testing of Piles
This test method covers the procedure for determining the integrity of individual vertical or inclined piles by measuring and analyzing the velocity (required) and force (optional) response of the pile induced by an (hand held hammer or other similar type) impact device applied axially to the pile normally at the pile head. This test method is applicable to long structural elements that function in a manner similar to foundation piles, regardless of their method of installation provided that they are receptive to low strain impact testing.

ASTM D5883: Standard Guide for use of Rotary Kiln Produced Expanded Shale, Clay or Slate (ESCS) As a Mineral Amendment in Topsoil Used For Landscaping and Related Purposes
This guide covers the material characteristics, physical requirements, and sapling appropriate for the designation of material as a mineral amendment.

ASTM D5903: Standard Guide for Planning and Preparing for a Groundwater Event.
This guide covers planning and preparing for a groundwater sampling event. It includes the technical and administrative considerations and procedures. Example checklists are also provided as appendices.

ASTM D5911: Standard Practice for Minimum Set of Data Elements to Identify a Soil Sampling Site
This practice covers what in formations should be obtained to uniquely identify any soil sampling or examination sire where an absolute and recoverable location is necessary for quality control of the study, such as a waste disposal project. The minimum set of data elements for sampling site identification (DEFFSI) was developed considering the needs for informational data bases, such as geographic information systems (GIS). Other distinguishing details, such as individual site characteristics help in singularly cataloging the site. For studies that are not environmentally regulated, such as for an agricultural or preconstruction survey, the data specifications established an agency or company may be different from that of the minimum set (See guide D420 and practice D5254)

ASTM D5912: Standard Method for (Analytical Procedure) Determining Hydraulic Conductivity of an Unconfined Aquifer by Overdamped Well Response to Instantaneous Change in Head (Slug)
This test method covers the determination of hydraulic conductivity from the measurement of inertial force free (overdamped) response of a well-aquifer system to a sudden change in water level in a well. Inertial force free response of the water level in a well to a sudden change in water level is characterized by recovery to initial water level in an approximate exponential manner with negligible inertial effects.

ASTM D5918: Standard Test Methods for Frost Heave and Thaw Weakening Susceptibility of Soils

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These laboratory test methods cover the frost heave and thaw weakening susceptibilities of soil that is tested in the laboratory by accompanying the heave rate and thawed bearing ratio with values in an established classification system. This test was developed to classify the frost susceptibility of soils used in pavements. It should be used for soils where frost susceptibility considerations, based on particle size such as the lilt of 3% finer than 20mm in specification D2940, are uncertain. This is most important for frost-susceptibility is determined from the heave rate during freezing. The thaw weakening susceptibility is determined with the bearing ratio test (see test method D1883).

ASTM D5920: Standard Method (Analytical Procedure) for Tests of Anisotropic Unconfined Aquifers by Neuman Method
This test method covers an analytical procedure for determining the transmissivity, storage coefficient, specific yield, and horizontal-to-vertical hydraulic conductivity ratio of unconfined aquifer. It is used to analyze the drawdown of water levels in piezometers and partially or fully penetrating observation wells during pumping from a control well at a constant rate.

ASTM D5921: Standard Practice for Subsurface Site Characterization of Test Pits for On-Site Septic Systems
This practice covers procedures for the characterization of subsurface soil conditions at a site as part of the process for evaluating suitability for an on-site septic system. This practice provides a method for determining the usable unsaturated soil depth for septic tank effluent to infiltrate for treatment and disposal.

ASTM D5922: Standard Guide for Analysis of Spatial Variation in Geospatial Site Investigations
This guide covers recommendations for analyzing, interpreting, and modeling spatial variation of regionalized variables in geotechnical and environmental site investigations.

ASTM D5923: Standard Guide for Selection of Kriging Methods in Geostatistical Site Investigations
This guide covers recommendations for selecting appropriate kriging methods based on study objectives, exploratory data analysis, and analysis of spatial variation.

ASTM D5924: Standard Guide for Selection of Simulation Approaches in Geostatistical Site Investigations
This guide covers the conditions that determine the selection of a suitable simulation approach for a site investigation problem. Alternative simulation approaches considered here are conditional and nonconditional, indicator and Gaussian, single and multiple realization, point, and block.

ASTM D5925: Standard Practice for Preliminary Sizing and Delineation of Soil Absorption Field Areas for On-Site Septic Systems
This practice covers procedures for estimating the dimensions and marking the boundaries of a soil adsorption area for an on-site septic system involving residential-strength wastewater. It can also be used to estimate the dimensions of commercial on-site septic systems where wastewater strengths are similar to residential wastewater.

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ASTM D5971: Standard Practice for Sampling Freshly Mixed Control Low-Strength Material
This practice explains the procedure for obtaining a representative sample to test of freshly mixed controlled low strength material (CLSM) as delivered to the project site. This practice included sampling from revolving-drum truck mixers and from agitating equipment used to transport central-mixed CLSM.

ASTM D5978: Standard Guide for Maintenance and Rehabilitation for Groundwater Monitoring Wells
This guide covers an approach to selecting and implementing a well maintenance and rehabilitation program for groundwater monitoring wells. It provides information on symptoms of problems or deficiencies that indicate the need for maintenance and rehabilitation. It is limited to monitoring wells, that are designed and operated to provide access to, representative water samples from, and information about the hydraulic properties of the saturated subsurface while minimizing impact on the monitored zone. Some methods described herein may apply to other types of wells although the range of maintenance and rehabilitation treatment methods suitable for monitoring wells is more restricted than for other types of wells. Monitoring well include their associated pumps and surface equipment.

ASTM D5979: Standard Guide for Conceptualization and Characterization of Groundwater Systems
This guide covers an integrated, stepwise method for qualitative conceptualization of groundwater flow systems, including the unsaturated zone, for natural or human-induced behavior or changes.

ASTM D5980: Standard Guide for Selection and Documentation of Existing Wells for use in Environmental Site Characterization and Monitoring
This guide covers the use of existing wells for environmental site characterization and monitoring. It covers the following major topics: criteria for determining the suitability of existing wells for hydrogeologic characterization and groundwater quality monitoring, types of data required to document the suitability of an existing well, and the relative advantages and disadvantages of existing large- and small- capacity wells.

ASTM D5981: Standard Guide for Calibrating a Groundwater Flow Model Application
This guide covers techniques that can be used to calibrate a groundwater flow model. The calibration of a model is the process of matching historical data, and is usually a prerequisite for making predictions with the model.

ASTM D5982: Standard Test Method for Determining Cement Content of Fresh Soil-Cement (Heat of Neutralization Method)
This test method covers the procedure for determining the cement content of fresh soil-cement. This test method can be used to determining the cement content of specimens that contain 3 to 16% cement. This test method is appropriate for soil-cement containing up to 55% plus 4.75mm (No. 4) sieve-size particles with a maximum particle size of 75mm (3in.). It should not be used for determining the class F pozzolan content of these mixtures.

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ASTM D5922: Standard Guide for Analysis of Spatial Variation in Geostatistical Site Investigations
This guide covers recommendations for analyzing, interpreting, and modeling spatial variation of regionalized variables in geotechnical and environmental site investigations. The measures of spatial variation discussed in this guide include variograms and correlograms. This guide is intended to assist those who are already familiar with the geostatistical tools discussed herein and does not provide introductory information on the analysis, interpretation, and modeling of spatial variation.

ASTM D5995: Standard Guide for Environmental Site Characterization in Cold Regions
Use this guide in conjunction with guide D5730. This guide describes special problems to be considered when planning field investigations in cold regions. The primary focus of this guide is representing the special problems and concerns of site characterization in the cold regions of the world.

ASTM D6000: Standard Guide for Presentation of Water-Level Information from Groundwater Sites
This guide covers and summarizes methods for the presentation of water-level data from groundwater sites. This guide describes the basic tabular and graphic methods of presenting groundwater levels for a single groundwater site and several sites over the area of a project. These methods were developed by hydrologists to assist in the interpretation of hydraulic-head data. This guide covers a series of options, but does not specify a course of action. It should not be used as the sole criterion or basis of comparison, and does not replace or relieve professional judgment.

ASTM D6001: Standard Guide for Direct-Push Water Sampling for Geoenvironmental Investigations
This guide covers a review of methods for sampling groundwater at discrete points or in increments by insertion of sampling devices by static force or impact without drilling and removal of cuttings. By directly pushing the sampler, the soil is displaced and helps to form an annular seal above the sampling zone. Direct-push water sampling can be one time, or multiple sampling events. Methods for obtaining water samples for water quality analysis and detection of contaminants are presented.

ASTM D6023: Standard Test Method for Unit Weight, Yield, Cement Content, and Air Content (Gravimetric) of Controlled Low Strength Material (CLSM)
This test method explains determination of the mass per cubic foot (cubic meter) of freshly mixed Controlled Low Strength Material (CLSM) and gives formulas for calculating the yield, cement content, and the air content of the CLSM. This test method is based on test method C 138 for concrete.

ASTM D6024: Standard Test Method for Ball Drop on Controlled Low Strength Material (CLSM) To Determine Suitability for Load Application
This specification explains the determination of the ability of Controlled Low Strength Material (CLSM) to withstand loading by repeatedly dropping a metal weight onto the in-place material.

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ASTM D6025: Standard Guide for Developing and Evaluating Groundwater Modeling Codes

This guide covers a systematic approach to the development, testing, evaluating, and documentation of groundwater modeling codes. The procedures presented constitute the quality assurance framework for a groundwater modeling code. They include code review, testing, and evaluation using quantitative and qualitative measures. This guide applies to both the initial development and the subsequent maintenance and updating of groundwater modeling codes.

ASTM D6026: Standard Practice for using Significant Digits in Geotechnical Data

This practice is intended to promote uniformity in recording significant digits for measured and calculated values involving geotechnical date. The guidelines presented are industry standard, and are representative of the significant digits that should generally be retained. The guidelines do not consider material variation, purpose for obtaining the date, special purpose studies, or any considerations for the user's special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits, of reported data to commensurate with these considerations. It is beyond the scope of this practice to consider significant digits used in analysis methods for engineering design.

ASTM D6027: Standard Practice for Calibrating Linear Displacement Transducers for Geotechnical Purposes

This practice outlines the procedure for calibrating linear displacement transducers (LDTs) and their readout systems for geotechnical purposes. It covers any transducer used to measure displacement which gives an electrical output that is linearly proportional to displacement. This includes linear variable displacement transducers (LVDTs), linear displacement transducers (LDTs) and linear strain transducers (LSTs).

ASTM D6028: Standard Test Method (Analytical Procedure) for Determining Hydraulic Properties of a Confined Aquifer Taking into Consideration Storage of Water in Leaky Confining Beds by Modified Hantush Method.

This test method covers an analytical procedure for determining the transmissivity and storage coefficient of a confined aquifer taking into consideration the change in storage of water in overlying or underlying confining beds, or both. This test method is used to analyze water-level or head data collected from one or more observation wells or piezometers during the pumping of water from a control well at a constant rate. With appropriate changes in sign, this test method also can be used to analyze the effects of injecting water into a control well at a constant rate.

ASTM D6029: Standard Test Method (Analytical Procedure) for Determining Hydraulic Properties of a Confined Aquifer and a Leaky Confining Bed with Negligible Storage by the Hantush-Jacob Method

This test method covers an analytical procedure for determining the transmissivity and storage coefficient of a confined aquifer and the leakance value of an overlying or underlying confining bed for the case where there is negligible change of water in storage in a confining bed. This test method is used to analyze water-level or head data collected from one or more observation wells or piezometers during the pumping of water from a control well at a constant rate. With

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appropriate changes in sign, this test method also can be used to analyze the effects of injecting water into a control well at a constant rate.

ASTM D6030: Standard Guide for Selection of Methods for Assessing Groundwater or Aquifer Sensitivity and Vulnerability
This guide covers information needed to select one or more methods for assessing the sensitivity of groundwater or aquifers and the vulnerability of groundwater or aquifers to water-quality degradation by specific contaminants.

ASTM D6031: Standard Test Method for Logging In Situ Moisture Content and Density of Soil and Rock by the Nuclear Method In Horizontal, Slanted, and Vertical Access Tubes
This test method covers collection and comparison of logs of thermalized-neutron counts and back-scattered gamma counts along horizontal or vertical air-filled access tubes.

ASTM D6032: Standard Test Method for Determining Rock Quality Designation (RQD) of Rock Core
This test method covers the determination of the rock quality designation (RQD) as a standard parameter in drill core logging.

ASTM D6033: Standard Guide for Describing the Functionality of a Groundwater Modeling Code
This guide presents a systematic approach to the classification and description of computer codes used in groundwater modeling. Due to the complex nature of fluid flow and biotic and chemical transport in the subsurface, many different types of groundwater modeling codes exist, each having specific capabilities and limitations. Determining the most appropriate code for a particular application requires a thorough analysis of the problem at hand and the required and available resources, as well as a detailed description of the functionality of potentially applicable codes.

ASTM D6034: Standard Test Method (Analytical Procedure) for Determining the Efficiency of a Production Well in a Confined Aquifer from a Constant Rate Pumping Test.
This test method describes an analytical procedure for determining the hydraulic efficiency of a production well in a confined aquifer. It involves comparing the actual drawdown in the well to the theoretical minimum drawdown achievable and is based upon data and aquifer coefficients obtained from a constant rate pumping test.

ASTM D6035: Standard Test Determining the Effect Of Freeze-Thaw on Hydraulic Conductivity of Compacted or Undisturbed Soil Specimens Using a Flexible Wall Permeameter
This test method covers laboratory measurement of the effect of freeze-thaw on the hydraulic conductivity of compacted or undisturbed soil specimens (using test method D5084 and a flexible wall permeameter to determine hydraulic conductivity). This test method does not provide steps to perform sampling of, or testing on, in situ soils that have already been subjected to freeze-thaw conditions.

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ASTM D6036: Standard Guide for Displaying the Results of Chemical Analyses of Groundwater Ions and Trace Elements – Use of Maps
This guide offers a series of options but does not specify a course of action. It should not be used as the sole criterion or basis of comparison and does not replace or relieve professional judgment. This guide covers methods that display, as mapped information, the chemical constituents of groundwater samples. Details required by the investigator to use fully the methods are found in the listed references

ASTM D6066: Standard Practice for Determining the Normalized Penetration Resistance of Sands for Evaluation of Liquefaction Potential
This practice outlines a procedure to obtain a record of normalized resistance of sands to the penetration of a standard sampler driven by a standard energy for estimating soil liquefaction potential during earthquakes. The normalized penetration resistance determined in this practice may be useful for determination of other engineering properties of sands.

ASTM D6067: Standard Guide for Using Electronic Cone Penetrometer for Environmental Site Characterization
The electronic cone penetrometer test often is used to determine subsurface stratigraphy for geotechnical and environmental site characterization purposes. The geotechnical application of the electronic cone penetrometer test it discussed in detail in test method D5778, however, the use of electronic cone penetrometer test in environmental site characterization applications involves further considerations that are not discussed.

ASTM D6089: Standard Guide for Documenting a Groundwater Sampling Event
This guide covers what and how information should be recorded in the field when sampling a groundwater monitoring well. Following these recommendations will provide adequate documentation in most monitoring programs. In some situations, it may be necessary to record additional or different information, or both, to thoroughly document the sampling event. In other cases, it may not be necessary to record all of the information recommended in this guide. The level of documentation will be based on site-specific conditions and regulatory requirements.

ASTM D6092: Standard Practice for Specifying Standard Sizes of Stone for Erosion Control
This practice covers size designations and maximum ranges in mass or gradation for standard sizes for riprap, spalls, or bedding, or both, used for slope protection of dam embankments, stream bank erosion control, bridge piers and abutments. Sizes used for outer harbor structures such as break walls, revetments, confined diked disposal structures (heretofore described as armor stone, cover stone, or dimension stone) for which stone sizes range between 5 and 25 tons, or that require cut dimensions for layered-up structures are beyond the scope of this practice.

ASTM D6103: Standard Test Method for Flow Consistency of Controlled Low Strength Material (CLSM)
This test method covers the procedure for determination of the flow consistency of fresh Controlled Low Strength Material (CLSM). This test method applies to flowable CLSM with maximum particle size of 19.0mm (3/4 in.) or less, or to the portion of CLSM that passes a 19.0m (3/4 in.) sieve.

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ASTM D6106: Standard Guide for Establishing the Nomenclature of Groundwater Aquifers

This guide offers a series of options but does not specify a course of action. It should not be used as sole criterion or basis of comparison and does not replace or relieve professional judgment.

This guide contains instructions and suggestions for authors of groundwater (hydrogeologic) reports in assigning appropriately derived and formatted aquifer nomenclature. Discussed are the water-bearing units that may require name identification, which are, ranked from largest to smallest, aquifer system, aquifer, and zone. Guidance is given on choosing the source of aquifer names, those are from lithologic terms, rock-stratigraphic units, and geographic names.

ASTM D6128: Standard Test Method for Shear Testing of Bulk Solids Using the Jenike Shear Cell

This method covers the apparatus and procedures for measuring the cohesive strength of bulk solids both continuous flow and after storage at rest. In addition, measurements of internal friction, bulk density, and wall friction on various wall surfaces are included.

ASTM D6145: Standard Guide for Monitoring Sediment in Watersheds

This guide is intended to provide general guidance on a watershed monitoring program directed toward sediment. The guide offers a series of general steps without setting forth a specific course of action. It gives advice for establishing a monitoring program, not an implementation program. Sedimentation as referred to in this guide is the detachment, entrainment, transportation, and deposition of eroded soil and rock particles. Specific types or parameters of sediment may include: suspended sediment, bedload, bed material, turbidity, wash load, sediment concentration, total load, sediment deposits, particle size distribution, sediment volumes and particle chemistry. Monitoring may include not only sediments suspended in water but sediments deposited in fields, floodplains, and channel bottoms. This guide applies to surface waters as found in streams and rivers; lakes, ponds, reservoirs, estuaries, and wetlands.

ASTM D6146: Standard Guide for Monitoring Aqueous Nutrients in Watersheds

This guide is intended to provide general guidance on a watershed monitoring program directed toward the plant nutrients nitrogen and phosphorus. The guide offers a series of general steps without setting forth a specific course of action. It gives assistance for developing a monitoring program but not a program for implementing measures to improve water quality. This guide applies to waters found in streams and rivers; lakes, ponds, and reservoirs; estuaries; wetlands; the atmosphere; and the vadose and subsurface saturated zones (including aquifers). This guide does not apply to nutrients found in soils, plants, or animals.

ASTM D6151: Standard Practice for Using Hollow-Stem Augers for Geotechnical Exploration and Soil Sampling

This practice covers how to obtain soil samples using hollow-stem sampling systems and use of hollow-stem auger drilling methods for geotechnical exploration. This practice addresses how to obtain soil samples suitable for engineering properties testing.

ASTM D6167: Standard Guide for Conducting Borehole Geophysical Logging: Mechanical Caliper

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This guide covers the general procedures necessary to conduct caliper logging of boreholes, wells, access tubes, caissons, or shafts (hereafter referred as boreholes) as commonly applied to geologic. Engineering, groundwater and environmental (hereafter referred as geotechnical) investigations. Caliper logging for mineral or petroleum exploration and development are excluded.

ASTM D6168: Standard Guide for Selection of the Minimum Set of Data Elements Required to Identify Locations Chosen for the Field Collection of Information to Describe Soil, Rock, and their Contained Fluids
This guide covers factors to consider for the selection of the minimum set of data elements required for the accurate location and cataloging of information collected for geological sciences (geoscience) investigations, which includes geoecology.

ASTM D6169: Standard Guide for Selection of Soil and Rock Sampling Devices Used With Drill Rigs for Environmental Investigations
This guide covers guidance for the selection of soil and rock sampling devices used with drill rigs for the purpose of characterizing in situ physical and hydraulic properties, chemical characteristics, subsurface lithology, stratigraphy and structure, and hydrogeologic units in environmental investigations.

ASTM D6170: Standard Guide for Selecting a Groundwater Modeling Code
This guide covers a systematic approach to the determination of the requirements for and the selection of computer codes used in a groundwater modeling project. Due to the complex nature of fluid flow and biotic and chemical transport in the subsurface many different groundwater modeling codes exist, each having specific capabilities and limitations. Furthermore, a wide variety of situations may be encountered in projects where groundwater models are used. Determining the most appropriate code for a particular application requires a thorough analysis of the problem at hand and the required and available resources, as well as detailed description of the functionality of candidate codes.

ASTM D6171: Standard Guide for Documenting a Groundwater Modeling Code
This guide covers suggested components of the documentation of a groundwater modeling code. Documentation of groundwater modeling code consists of textural and graphical information recorded during its design, development, and maintenance regarding its capabilities, development history, theoretical foundation, operation, and verification. It is the principal instrument for those involved in its development and use, such as code development and maintenance staff, network mangers, code users and project managers, to communicate regarding all aspects of the software.

ASTM D6187: Standard Practice for Cone Penetrometer Technology Characterization of Petroleum Contaminated Sites with Nitrogen Laser-Induced Fluorescence
This practice covers the method for delineating the subsurface presence of petroleum hydrocarbons and other hydrocarbons using fiber optic based nitrogen laser-induced fluorescence sensor system.

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ASTM D6230: Standard Test Method for Monitoring Ground Movement Using Probe-Type Inclinometers
This test method covers the use of inclinometers to monitor the internal movement of ground. The test method covers types of instruments, installation procedures, operating procedures and maintenance requirements. It also provides formulae for data reduction.

ASTM D6235: Standard Practice for Expedited Site Characterization of Vadose Zone and Groundwater Contamination at Hazardous Waste Contaminated Sites
This practice covers a process for expedited site characterization (ESC) of hazardous waste contaminated sites to identify vadose zone, groundwater and other relevant contaminant migration pathways and determine the distribution, concentration, and fate of contaminants for the purpose of providing and ESC client, regulatory authority and stakeholders with the necessary information to choose a course of action. Generally, the process is applicable to larger-scale projects, such as CERCLA (Superfund) remedial investigations and RCRA facility investigations. The ESC process is also applicable to other contaminated sites where the ESC process can be reasonably expected to reduce the time and cost of site characterization compared to alternative approaches. The ESC process has been applied successfully at a variety of sites in different states and EPA regions. It typically achieves significant cost and schedule savings compared to traditional site characterization.

ASTM D6236: Standard Guide for Cement and Logging Cement – or Lime-Stabilized Soil
This guide covers guidance for obtaining cores of cement or lime stabilized soil used in determining compressive strength, lift thickness and bond strength, and other physical properties. This guide is primarily for use in coring through shallow (0.3 to 3 meter (1 to 10ft) thick) layers of cement or lime stabilized soils containing particles <50mm (2in.) in diameter to the underlying foundation.

ASTM D6274: Standard Guide for Conducting Borehole Geophysical Logging-Gamma
This guide covers the general procedures necessary to conduct gamma, natural gamma, total count gamma, or gamma ray (hereafter referred to as gamma) logging of boreholes, wells, access tubes, caissons, or shafts (hereafter referred to as boreholes) as commonly applied to geologic, engineering, groundwater and environmental (hereafter referred to as geotechnical) investigations. Spectral gamma and logging where measurements are made in conjunction with a nuclear source are excluded (for examples, neutron activation and gamma-gamma density logs). Gamma logging for minerals or petroleum applications are excluded.

ASTM D6276: Standard Test Method for Using Ph to Estimate the Soil-Lime Proportion Requirement for Soil Stabilization
This test method provides a means for estimating the soil-lime proportion requirement for stabilization of a soil. This test method is performed on soil passing the -425-µm (No. 40) sieve. The optimum soil-lime proportion for soil stabilization is determined by tests of specific characteristics of stabilized soil such as unconfined compressive strength or plasticity index.

ASTM D6282: Standard Guide for Direct Push Soil Sampling for Environmental Site Characterizations

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This guide addresses direct push soil samplers, which also may be driven into the ground from the surface or through prebored holes. The samplers can be continuous or discrete interval units. Samplers are advanced by a combination of static push, or impacts from hammers, or vibratory methods, or a combination thereof, to the depth of interest. The guide does not cover open chambered samplers operated by hand such as augers, agricultural samplers operated at shallow depths, or side wall samplers. This guide does not address single sampling events in the immediate base of the drill hole using rotary drilling equipment with incremental drill hole excavation. Other sampling standards, such as test methods D1586 and D1587 and practice D3550 apply to rotary drilling activities. This guide does not address advancement of sampler barrel systems with methods that employ cuttings removal as the sampler is advanced. Other drilling and sampling methods may apply for samples needed for engineering and construction applications.

ASTM D6285: Standard Guide for Locating Abandoned Wells
This guide provides an approach to selecting and implementing a program to identify the locations of abandoned wells. This guide provides descriptions of methods to be used as starting points in the search for these locations. It is not intended to be a step-by-step procedure to conduct the search program. This guide also provides listings of government agencies that may have well location information. It is understood that addresses and phone numbers change and that the included information may not be accurate in the future.

ASTM D6286: Standard Guide for Selection Drilling Methods for Environmental Site Characterization
This guide provides descriptions of various drilling methods for environmental site characterization along with advantages and disadvantages associated with each method discussed. A comprehensive description of these drilling methods can be found in individual ASTM standards, see Section 2. This guide is intended to aid in the selection of drilling method(s) for environmental soil and rock borings and the installation of monitoring wells and other water-quality monitoring devices.

ASTM D6301: Standard Practice for Collection of On-Line Composite Samples of Suspended Solids and Ionic Solids in Process Water
This practice is applicable for sampling condensed steam or water, such as boiler feedwater, for the collection of suspended solids and (optional) ionic solids using a 0.45-µm membrane filter (suspended solids) and ion exchange media (ionic solids). As the major suspended component found in most boiler feedwaters is some form of corrosion product from the preboiler system, the device used for this practice is commonly called a corrosion product sampler. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

ASTM D6312: Standard Guide for Developing Appropriate Statistical Approaches for Groundwater Detection Monitoring Programs
This guide covers the context of groundwater monitoring at waste disposal facilities, regulations have required statistical methods as the basis for investigating potential environmental impact due to waste disposal facility operation. Owner/operators must perform a statistical analysis on a

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quarterly or semiannual basis. A statistical test is performed on each of many constituents (for example, 10 to 50 or more) for each of many wells (5 to 100 or more). The result is potentially hundreds, and in some cases, a thousand or more statistical comparisons performed on each monitoring event. Even if the false positive rate for a single test is small (for example, 1%), the possibility of failing at least one test on any monitoring event is virtually guaranteed. This assumes you have done the correct statistic in the first place.

ASTM D6391: Standard Test Method for Field Measurement of Hydraulic Conductivity Limits of Porous Materials Using Two Stages of Infiltration from a Borehole
This test method covers field measurement of limiting values for vertical and horizontal hydraulic conductivities (also referred to as coefficients of permeability) of porous materials using the two-stage, cased borehole technique. These limiting hydraulic conductivity values are the maximum possible for the vertical direction and minimum possible for the horizontal direction. Determination of actual hydraulic conductivity values requires further analysis by qualified personnel.

ASTM D6393: Standard Test Method for Bulk Solids Characterization by Carr Indices
This test method covers the apparatus and procedures for measuring properties of bulk solids, henceforth referred to as Carr Indices.

ASTM D6429: Standard Guide for Selecting Geophysical Methods
This guide covers the selection of surface geophysical methods, as commonly applied to geologic, geotechnical, hydrologic, and environmental investigations (hereafter referred to as site characterization), as well as forensic and archaeological applications. This guide does not describe the specific procedures for conducting geophysical surveys. Individual guides are being developed for each surface geophysical method.

ASTM D6430: Standard Guide for Using The Gravity Method for Subsurface Investigation
This guide summarizes the equipment, field procedures, and interpretation methods for the assessment of subsurface conditions using the gravity method. The gravity method described in this guide is applicable to investigation of a wide range of subsurface conditions. Gravity measurements indicate variations in the earth's gravitational field caused by lateral differences in the density of the subsurface soil or rock or the presence of natural voids or man-made structures. By measuring spatial changes in the gravitational field, variations in subsurface conditions can be determined. Detailed gravity surveys (commonly called microgravity surveys) are used for near-surface geologic investigations and geotechnical, environmental, and archaeological studies. Geologic and geotechnical applications include location of buried channels, bedrock structural features, voids, and caves, and low-density zones in foundations. Environmental applications include site characterization, groundwater studies, landfill characterization, and location of underground storage tanks.

ASTM D6431: Standard Guide for Using Direct Current Resistivity Method for Subsurface Investigation
This guide summarizes the equipment, field procedures, and interpretation methods for the assessment of the electrical properties of subsurface materials and their pore fluids, using the

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direct current (DC) resistivity method. Measurements of the electrical properties of subsurface materials are made from the land surface and yield an apparent resistivity. These data can then be interpreted to yield an estimate of the depth, thickness, and resistivity of subsurface layer(s). Resistivity measurements as described in this guide are applied in geological, geotechnical, environmental, and hydrologic investigations. The resistivity method is used to map geologic features such as lithology, structure, fractures, and stratigraphy; hydrologic features such as depth to water table, depth to aquitard, and groundwater salinity; and to delineate groundwater contaminants.

ASTM D6432: Standard Guide for Using the Surface Penetrating Radar Method for Subsurface Investigation

This guide summarizes the equipment, field procedures, and interpretation methods for the assessment of sub surface materials using the impulse Ground Penetrating Radar (GPR) Method. GPR is most often employed as a technique that uses high-frequency electromagnetic (EM) waves (from 10 to 3000 MHz) to acquire subsurface information. GPR detects changes in EM properties (dielectric permittivity, conductivity, and magnetic permeability), that in a geologic setting, are a function of soil and rock material, water content, and bulk density. Data are normally acquired using antennas placed on the ground surface or in boreholes. The transmitting antenna radiates EM waves that propagate in the subsurface and reflect from boundaries at which there are EM property contrasts. The receiving GPR antenna records the reflected waves over a selectable time range. The depths to the reflecting interfaces are calculated from the arrival times in the GPR data if the EM propagation velocity in the subsurface can be estimated or measured.

ASTM D6449: Standard Test Method for Flow of Fine Aggregate Concrete for Fabric Formed Concrete (Flow Cone Method)

This test method covers a procedure, used both in the laboratory and in the field, for determining the time of efflux of a specified volume of the fine aggregate concrete through a standardized flow cone and used for fabric formed concrete (FFC); however, the test method may also be used for other fluid concrete.

ASTM D6452: Standard Guide for Purging Methods for Wells Used for Groundwater Quality Investigations

This guide covers methods for purging wells used for groundwater quality investigations and monitoring programs. These methods could be used for other types of programs but are not addressed in this guide. This guide applies only to wells sampled at the well-head.

ASTM D6453: Standard Guide for Format of Computerized Exchange of Soil and Rock Test Data (Withdrawn 2008)

This guide covers recommended data formats for the exchange of mechanical test data for soils and rocks. From this guide, a standardized file of data can be prepared that can be read by others who use this guide. The format specified in this guide is used for the exchange of data between computer programs, users, agencies, etc. It is not necessary that test data for internal use be stored in this format, only that a program adhering to this guide has the capability to read, or write test data in this format, or both.

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ASTM D6459: Standard Test Method for Determination of Erosion Control Blanket (ECB) Performance in Protecting Hillslopes from Rainfall-Induced Erosion
This test method covers the guidelines, requirements and procedures for evaluating the ability of Erosion Control Blankets (ECBs) to protect hillslopes from rainfall-induced erosion. Critical elements of this protection are the ability of the ECB to: Absorb the impact force raindrops, thereby reducing soil particle loosening through "splash" mechanisms; low runoff and encourage infiltration, thereby reducing soil particle displacement and transport through "overland flow" mechanisms; Absorb shear forces of overland flow; and, Trap soil particles beneath.

ASTM D6460: Standard Test Method for Determination of Erosion Control Blanket (ECB) Performance in Protecting Earthen Channels from Stormwater-Induced Erosion
This test method covers the guidelines, requirements and procedures for evaluating the ability of Erosion Control Blankets (ECBs) to protect earthen channels from stormwater-induced erosion. Critical elements of this protection are the ability of the ECB to: Neutralize and absorb the hydraulic force of stormwater, thereby reducing soil particle loosening through "scour" mechanisms, and; Slow runoff and encourage sedimentation, thereby reducing soil particle transport downstream; Absorb shear forces of overland flow; and, Trap soil particles beneath.

ASTM D6461: Standard Specification for Silt Fence Materials
This specification covers requirements and test methods for geotextile fabrics and associated components used in temporary silt fence applications. This is a material purchasing specification based on AASHTO M288.

ASTM D6462: Standard Practice for Silt Fence Installation
This practice covers common installation requirements for temporary silt fence applications. This practice is based on AASHTO M288.

ASTM D6467: Standard Test Method for Torsional Ring Shear Test to Determine Drained Residual Shear Strength of Cohesive Soils
This test method provides a procedure for performing a torsional ring shear test under a drained condition to determine the residual shear strength of cohesive soils. An undisturbed specimen can be used for testing. However, obtaining a natural slip surface specimen, determining the direction of field shearing, and trimming and properly aligning the usually non-horizontal shear surface in the ring shear apparatus is difficult. As a result, this test method focuses on the use of a remolded specimen. This test method is performed by deforming a presheared, remolded specimen at a controlled displacement rate until the constant minimum drained shear resistance is offered on a single shear plane determined by the configuration of the apparatus. An unlimited amount of continuous shear displacement can be achieved to obtain a residual strength condition. Generally, three or more normal stresses are applied to a test specimen to determine the drained residual failure envelope. A separate test specimen may be used for each normal stress.

ASTM D6473: Standard Test Method for Specific Gravity and Absorption of Rock Erosion Control
This test method covers the determination of specific gravity and absorption of rock for erosion control, commonly referred to as riprap or armor stone. The specific gravity may be expressed as

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bulk specific gravity or apparent specific gravity. Bulk specific gravity and absorption are based on a 24-h soaking time for the rock specimens tested.

ASTM D6475: Standard Test Method for Measuring Mass Per Unit Area Erosion Control Blankets
This test method can be used as an index test to determine the mass per unit area of all erosion control blankets (ECBs). This measurement allows for a simple control of the delivered material by a comparison of the mass per unit area of the delivered material and the specified mass per unit area.

ASTM D6517: Standard Guide for Field Preservation of Groundwater Samples
This guide covers methods for field preservation of groundwater samples. Laboratory preservation methods are not described in this guide. This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

ASTM D6519: Standard Practice for Sampling Soil Using the Hydraulically Operated Stationary Piston Sampler
This practice covers a procedure for sampling of cohesive, organic, or fine-grained soils, or combination thereof, using a thin-walled metal tube that is inserted into the soil formation by means of a hydraulically operated piston. It is used to collect relatively undisturbed soil samples suitable for laboratory tests to determine structural and chemical properties for geotechnical and environmental site characterizations.

ASTM D6527: Standard Test Method for Determining Unsaturated and Saturated Hydraulic Conductivity in Porous Media by Steady-State Centrifugation
This test method covers the determination of the hydraulic conductivity, or the permeability relative to water, of any porous medium in the laboratory, in particular, the hydraulic conductivity for water in subsurface materials, for example, soil, sediment, rock, concrete, and ceramic, either natural or artificial, especially in relatively impermeable materials or materials under highly unsaturated conditions. This test method covers determination of these properties using any form of steady-state centrifugation (SSC) in which fluid can be applied to a specimen with a constant flux or steady flow during centrifugation of the specimen. This test method only measures advective flow on core specimens in the laboratory.

ASTM D6528: Standard Test Method for Consolidated Undrained Direct Simple Shear Testing of Cohesive Soils
This test method defines equipment specifications and testing procedures for the measurement of constant volume strength and stress-strain characteristics of cohesive soils after one-dimensional consolidation using a constant rate of simple shear deformation mode of loading. The constant volume condition is equivalent to the undrained condition for saturated specimens.

ASTM D6539: Standard Test Method for Measurement of Pneumatic Permeability of Partially Saturated Porous Materials by Flowing Air

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This test method covers laboratory determination of the coefficient of permeability for the flow of air (pneumatic permeability) through partially saturated porous materials. This test method applies to the one-dimensional laminar (viscous) flow of air in porous materials such as soil, and is intended to support soil remediation operations such as: soil vapor extraction, air sparging, backfilling of soils in utility trenches, and similar engineering activities.

ASTM D6564: Standard Guide for Field Filtration of Groundwater Samples
This guide covers methods for field filtration of groundwater samples collected from groundwater monitoring wells, excluding samples that contain non-aqueous phase liquids (either Dense Non-Aqueous Phase Liquids (DNAPLs) or Light Non-Aqueous Phase Liquids (LNAPLs)). Methods of field filtration described herein could also be applied to samples collected from wells used for other purposes. Laboratory filtration methods are not described in this guide.

ASTM D6565: Standard Test Method for Determination of Water (Moisture) Content of Soil by the Time-Domain Reflectometry (TDR) Method
This test method covers the determination of water content (or moisture content) in soil by the use of the electromagnetic technique called Time-Domain Reflectometry (TDR). The TDR application covered in this test method is that used for point measurements of moisture content in soil. The application is either through manual insertion into the soil or by burying a probe in the subsurface to acquire moisture content data at a specific location. In addition, core samples may be tested with TDR at a drill site to acquire real-time soil moisture data.

ASTM D6568: Standard Guide for Planning, Carrying Out, and Reporting Traceable Chemical Analyses of Water Samples
This guide sets a protocol for generating and reporting chemical analyses that are traceable to SI Units or to Certified Reference Materials in laboratories that serve the water and environmental industry. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

ASTM D6569: Standard Test Method for On-Line Measurement of pH
This test method covers the continuous determination of pH of water by electrometric measurement using the glass, the antimony or the ion-selective field-effect transistor (ISFET) electrode as the sensor. This test method does not cover measurement of samples with less than 100 μS/cm conductivity. Refer to Test Method D 5128. This test method does not cover laboratory or grab sample measurement of pH. Refer to Test Method D 1293. The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

ASTM D6572: Standard Test Methods for Determining Dispersive Characteristics of Clayey Soils by the Crumb Test
These test methods provide a qualitative indication of the natural dispersive characteristics of clayey soils.
The crumb test method provides a simple, quick method for field or laboratory identification of a dispersive clay soil. The internal erosion failures of a number of homogeneous earth dams,

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erosion along channel or canal banks, and rainfall erosion of earthen structures have been attributed to colloidal erosion along cracks or other flow channels formed in masses of dispersive clay

ASTM D6598: Standard Guide for Installing and Operating Settlement Platforms for Monitoring Vertical Deformation

This guide provides recommended designs and procedures for the fabrication, installation, operation, and reading of settlement platform to determine the magnitude and rate of foundation, fill settlements, or both, generally under a fill or embankment load. Two types of settlement platforms are described - those be monitored by elevation surveys from an external bench mark and those that include an internal reference system supported on unyielding soil or rock beneath the compressible layer(s) of interest.

ASTM D6599: Standard Practice for Construction of Live Fascines on Slopes

This practice covers the material, fabrication and installation work to construct live fascines.
Live fascines are used to provide erosion and sedimentation control by increasing infiltration, slowing or redirecting runoff, and trapping seed and sediments. The method provides shallow mechanical surface slope stabilization, and provides enhanced values through vegetative growth and additional shallow soil reinforcement through the development of the roots. The ability of live fascines to function properly depends on the quality and choice of the materials used to construct the live fascine, the means and methods of fabrication and installation, and proper consideration of site characteristics and time of year. For the live fascine to function completely, it is important that the live fascine develops suitable growth.

ASTM D6629: Standard Guide for Selection Methods for Estimating Soils Loss by Erosion

This guide describes methods used for estimating soil losses due to rill and interrill erosion. All known methods for water erosion with a sediment yield component are listed. This guide provides complete reference citations for the various methods currently used to estimate soil loss by rill and interrill erosion. The referenced methods contain the rationale and detailed procedures for determining rill and interrill soil loss. For each method listed, specific applicability, limitations, and level of complexity are briefly outlined in terms of the relative spatial scale, land use, hydrology, erosion, time scale, input requirements, and output.

ASTM D6634: Standard Guide for Selection of Purging and Sampling Devices for Groundwater Monitoring Wells

This guide describes the characteristics and operating principles of purging and sampling devices available for use in groundwater monitoring wells and provides criteria for selecting appropriate devices for specific applications. The selected device(s) should be capable of purging the well and providing valid representative samples of groundwater and any included dissolved constituents. The scope does not include procedures for purging or collecting samples from monitoring wells, sampling devices for non-aqueous phase liquids, diffusion-type sampling devices or sampling from devices other than monitoring wells.

ASTM D6635: Standard Test Method for Performing the Flat Plate Dilatometer

This test method describes an in-situ penetration plus expansion test. The test is initiated by forcing the steel, flat plate, dilatometer blade, with its sharp cutting edge, into a soil. Each test

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consists of an increment of penetration, generally vertical, followed by the expansion of a flat, circular, metallic membrane into the surrounding soil. The test provides information about the soil's in-situ stratigraphy, stress, strength, compressibility, and pore-water pressure for use in the design of earthworks and foundations.

ASTM D6639: Standard Guide for Using the Frequency Domain Electromagnetic Method for Subsurface Investigations
This standard guide summarizes the equipment, field procedures, and interpretation methods for the assessment of subsurface conditions using the frequency domain electromagnetic (FDEM) method. FDEM measurements as described in this standard guide are applicable to mapping subsurface conditions for geologic, geotechnical, hydrologic, environmental, agricultural, archaeological and forensic investigations as well as mineral exploration. The FDEM method is sometimes used to map such diverse geologic conditions as depth to bedrock, fractures and fault zones, voids and sinkholes, soil and rock properties, and saline intrusion as well as man-induced environmental conditions including buried drums, underground storage tanks (USTs), landfill boundaries and conductive groundwater contamination.

ASTM D6642: Standard Guide for Comparison of Techniques to Quantify the Soil-Water (Moisture) Flux
This guide describes techniques that may be used to quantify the soil-water (or soil-moisture) flux, the soil-water movement rate, and/or the recharge rate within the vadose zone. This guide is not intended to be all-inclusive with regard to available methods. However, the techniques described do represent the most widely used methods currently available.

ASTM D6682: Standard Test Method for Measuring the Shear Stresses of Powders Using the Peschl Rotational Split Level Shear Tester
This test method is applied to the measurement of the mechanical properties of powders as a function of normal stress.

ASTM D6683: Standard Test Method for Measuring Bulk Density Values of Powders and Other Bulk Solids
This test method covers an apparatus and procedure for determining a range of bulk densities of powders and other bulk solids as a function of compaction pressure.

ASTM D6684: Standard Specification for Materials and Manufacture of Articulating Concrete Block (ACB) Revetment Systems
The purpose of this Standard is to provide specifications for articulating concrete block (ACB) revetment system structural components, material composition and physical properties, manufacturing methods and testing requirements.

ASTM D6685: Standard Guide for the Selection of Test Methods for Fabrics Used for Fabric Formed Concrete (FFCC)
This guide lists ASTM standards available for the testing evaluation of fabrics used to form fine aggregate concrete.

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ASTM D6711: Standard Practice for Specifying Rock To Fill Gabions, Revet Mattresses, and Gabion Mattresses
This practice covers the sizes and quality of rock to fill gabions and mattresses. The term mattress as used in this standard shall include the terminology of gabion mattresses and revet mattresses used in Specifications A974 and A975.

ASTM D6724: Standard Guide for Installation of Direct Push Groundwater Monitoring Wells
This guide describes various direct push groundwater monitoring wells and provides guidance on their selection and installation for obtaining representative groundwater samples and monitoring water table elevations. Direct push wells are used extensively for monitoring groundwater quality in unconsolidated formations. This guide also includes discussion of some groundwater sampling devices which can be permanently emplaced as monitoring wells.

ASTM D6725: Standard Practice for Direct Push Installation of Prepacked Screen Monitoring Wells in Unconsolidated Aquifers
This practice is based on recognized methods by which direct push monitoring wells may be designed and installed for the purpose of detecting the presence or absence of a contaminant, and collecting representative groundwater-quality data. The design standards and installation procedures herein are applicable to both detection and assessment monitoring programs for facilities.

ASTM D6726: Standard Guide for Conducting Borehole Geophysical Logging- Electromagnetic Induction
This guide is focused on the general procedures necessary to conduct electromagnetic-induction, induction, electromagnetic-conductivity, or electromagnetic-resistivity logging (hereafter referred as induction logging) of boreholes, wells, access tubes, caissons, or shafts (hereafter referred as boreholes) as commonly applied to geologic, engineering, groundwater and environmental (hereafter referred as geotechnical) investigations. Induction logging for minerals or petroleum applications is excluded.

ASTM D6727: Standard Guide for Conducting Borehole Geophysical Logging- Neutron
This guide is focused on the general procedures necessary to conduct neutron or neutron porosity (hereafter referred to as neutron) logging of boreholes, wells, access tubes, caissons, or shafts (hereafter referred to as boreholes) as commonly applied to geologic, engineering, groundwater and environmental (hereafter referred to as geotechnical) investigations. Neutron soil moisture measurements made using neutron moisture gauges, are excluded. Neutron logging for minerals or petroleum applications is excluded, along with neutron activation logs where gamma spectral detectors are used to characterize the induced gamma activity of minerals exposed to neutron radiation.

ASTM D6758: Standard Test Method for Measuring Stiffness and Apparent Modulus of Soil and Soil Aggregate in-Place by an Electro-Mechanical Method
This method covers the measurement by electro-mechanical means of the in-place stiffness of soil or soil-aggregate mixtures so as to determine a Young's modulus based on certain assumptions. The apparatus and procedure provide a fairly rapid means of testing so as to

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minimize interference and delay of construction. The test procedure is intended for evaluating the stiffness or modulus of materials used in earthworks and road works. Rapid in-place stiffness testing supports U.S. federal and state efforts to specify the in-place performance of construction materials based on modulus. Results obtained from this method are applicable to the evaluation of granular cohesionless materials. They are also applicable to the evaluation of silty and clayey materials with more than 20 % fines that are not subject to a change in moisture content. If the silty and clayey material experiences a change in moisture content, then moisture content shall be taken into account if the results of this method are to be applicable. The stiffness measured with this method is influenced by boundary conditions, specifically the support offered by underlying layers as well as the thickness and modulus of the layer being tested. Since this method approximates the layer(s) being evaluated as a half-space, then the modulus measured is also approximate.

ASTM D6760: Standard Test Method for Integrity Testing of Concrete Deep Foundations by Ultrasonic Crosshole Testing

This test method covers procedures for checking the homogeneity and integrity of concrete in a deep foundation such as bored piles, drilled shafts, concrete piles or augercast piles. This method can also be extended to diaphragm walls, barrettes, dams etc. The test measures the propagation time and relative energy of an ultrasonic pulse between parallel access ducts installed in the pile or structure. This method is most applicable when performed between tubes that are installed during pile construction.

ASTM D6764: Standard Guide for Collection of Water Temperature, Dissolved-Oxygen Concentrations, Specific Electrical Conductance, and pH Data from Open Channels

This guide describes procedures to collect cross-sectional means of temperature, dissolved oxygen, specific electrical conductance, and pH of water flowing in open channels. This guide provides guidelines for preparation and calibration of the equipment to collect cross-sectional means of temperature, dissolved oxygen, specific electrical conductance, and pH of water flowing in open channels. This guide describes what equipment should be used to collect cross-sectional means of temperature, dissolved oxygen, specific electrical conductance, and pH of water flowing in open channels. This guide covers the cross-sectional means of temperature, dissolved oxygen, specific electrical conductance, and pH of fresh water flowing in open channels.

ASTM D6765: Standard Practice for Live Staking

This practice covers the material, preparation and installation work required for live stake construction.

ASTM D6771: Standard Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Groundwater Quality Investigations

This practice covers the method for purging and sampling wells and devices used for groundwater quality investigations and monitoring programs known as low-flow purging and sampling. This method is also known by the terms minimal drawdown purging or low-stress purging. This method could be used for other types of groundwater sampling programs but these uses are not specifically addressed in this practice.

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ASTM D6773: Standard Shear Test Method for Bulk Solids Using the Schulze Ring Shear Tester
This method covers the apparatus and procedures for measuring the unconfined yield strength of bulk solids during both continuous flow and after storage at rest. In addition, measurements of internal friction, bulk density, and wall friction on various wall surfaces are included. The SI system of units has been used throughout.

ASTM D6780: Standard Shear Test Method for Water Content and Density of Soil in Place by Time Domain Reflectometry (TDR)
This test method may be used to determine the water content of soils and the in-place density of soils using a TDR apparatus.

ASTM D6820: Standard Guide for Use of the Time Domain Electromagnetic Method for Subsurface Investigation
This guide summarizes the equipment, field procedures, and interpretation methods for the assessment of subsurface materials and their pore fluids using the Time Domain Electromagnetic (TDEM) method. This method is also known as the Transient Electromagnetic Method (TEM), and in this guide is referred to as the TDEM/TEM method. Time Domain and Transient refer to the measurement of a time-varying induced electromagnetic field. The TDEM/TEM method is applicable to investigation of a wide range of subsurface conditions. TDEM/TEM methods measure variations in the electrical resistivity (or the reciprocal, the electrical conductivity) of the subsurface soil or rock caused by both lateral and vertical variations in various physical properties of the soil or rock. By measuring both lateral and vertical changes in resistivity, variations in subsurface conditions can be determined. Electromagnetic measurements of resistivity as described in this guide are applied in geologic studies, geotechnical studies, hydrologic investigations, and for mapping subsurface conditions at waste disposal sites. Resistivity measurements can be used to map geologic changes such as lithology, geological structure, fractures, stratigraphy, and depth to bedrock. In addition, measurement of resistivity can be applied to hydrologic investigations such as the depth to water table, depth to aquitard, presence of coastal or inland groundwater salinity, and for the direct exploration for groundwater.

ASTM D6825: Standard Guide for Placement of Riprap Revetments
This guide covers methods to place riprap with associated filters for erosion control purposes. This guide does not recommend a specific course of action because of the diverse methods and procedures that are capable of producing a functional product. This guide identifies favorable riprap qualities and recommends practices best suited to obtain those qualities. The production of rock, use of recycled materials, rock with cut dimensions, and engineering and design is beyond the scope of this guide. Special forms of riprap, including hand placed riprap, grouted riprap, or keyed (plated) riprap that is tamped into place to smooth the surface, are also beyond the scope of this guide.

ASTM D6836: Standard Test Methods for Determination of the Soil Water Characteristic Curve for Desorption Using a Hanging Column, Pressure Extractor, Chilled Mirror Hygrometer, and/or Centrifuged

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This test method covers the determination of soil water characteristic curves (SWCCs) for desorption (drying). SWCCs describe the relationship between suction and volumetric water content, gravimetric water content, or degree of water saturation. SWCCs are also referred to as soil water retention curves, soil water release curves, or capillary pressure curves.

ASTM D6911: Standard Guide for Packaging and Shipping Environmental Samples for Laboratory Analysis

This standard provides guidance in determining the most appropriate procedures for packaging and shipping environmental samples. Use of this guide by personnel involved in packaging and shipping environmental samples will facilitate safe, effective and compliant procedures. This standard provides guidance on the selection of procedures for proper packaging and shipment of environmental samples to the laboratory for analysis to ensure compliance with appropriate regulatory programs and protection of sample integrity during shipment.

ASTM D6913: Standard Test Methods for Particle-Size Distribution (Gradation) of Soils Using Sieve Analysis

Soils consist of particles with various shapes and sizes. This test method is used to separate particles into size ranges and to determine quantitatively the mass of particles in each range. These data are combined to determine the particle-size distribution (gradation). This test method uses a square opening sieve criterion in determining the gradation of soil between the 3-in. (75-mm) and No. 200 (75-m) sieves.

ASTM D6914: Standard Test Methods for Particle-Size Distribution (Gradation) of Soils Using Sieve Analysis

The gradation of the soil is used for classification in accordance with Practice D 2487.

The gradation (particle-size distribution) curve is used to calculate the coefficient of uniformity and the coefficient of curvature. Selection and acceptance of fill materials are often based on gradation. For example, highway embankments, backfills, and earthen dams may have gradation requirements. The gradation of the soil often controls the design and quality control of drainage filters, and groundwater drainage. Selection of options for dynamic compaction and grouting is related to gradation of the soil. The gradation of a soil is an indicator of engineering properties. Hydraulic conductivity, compressibility, and shear strength are related to the gradation of the soil. However, engineering behavior is dependent upon many factors (such as effective stress, stress history, mineral type, structure, plasticity, and geologic origins) and cannot be based solely upon gradation. Soils consist of particles with various shapes and sizes. This test method is used to separate particles into size ranges and to determine quantitatively the mass of particles in each range. These data are combined to determine the particle-size distribution (gradation). This test method uses a square opening sieve criterion in determining the gradation of soil between the 3-in. (75-mm) and No. 200 (75-μm) sieves.

ASTM D6938: Standard Test Method for in-Place Density and Water Content of Soil and Soil-Aggregate by Nuclear Methods (Shallow Depth)

This test method describes the procedures for measuring in-place density and moisture of soil and soil-aggregate by use of nuclear equipment. The density of the material may be measured by direct transmission, backscatter, or backscatter/air-gap ratio methods. Measurements for water

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(moisture) content are taken at the surface in backscatter mode regardless of the mode being used for density. It is the intent of this subcommittee that this standard replace D 2922 and D 3017.

ASTM D6939: Standard Practice for Brushmattressing
This practice covers the material, preparation and installation work required for brush- mattress construction used in soil bioengineering.

ASTM D6940: Standard Practice for Measuring Sifting Segregation Tendencies of Bulk Solids
This practice covers an apparatus and procedure for simulating the segregation tendencies of bulk solids by means of the sifting mechanism.

ASTM D7012: Standard Test Method for Compressive Strength and Elastic Moduli of Intact Rock Core Specimens under Varying States of Stress and Temperatures
Unconfined compressive strength of rock is used in many design formulas and is sometimes used as an index property to select the appropriate excavation technique. Deformation and strength of rock are known to be functions of confining pressure. The confined compression test is commonly used to simulate the stress conditions under which most underground rock masses exist. The elastic constants are used to calculate the stress and deformation in rock structures. The deformation and strength properties of rock cores measured in the laboratory usually do not accurately reflect large-scale in situ properties because the latter are strongly influenced by joints, faults, inhomogeneities, weakness planes, and other factors. Therefore, laboratory values for intact specimens must be employed with proper judgment in engineering applications. This test method covers the determination of the strength of intact rock core specimens in uniaxial compression and confined compression. The tests provide data in determining the strength of rock, namely: the uniaxial strength, shear strengths at varying pressures and varying temperatures, angle of internal friction, (angle of shearing resistance), and cohesion intercept. The test method specifies the apparatus, instrumentation, and procedures for determining the stress-axial strain and the stress-lateral strain curves, as well as Young's modulus, *E*, and Poisson's ratio, . It should be observed that this method makes no provision for pore pressure measurements and specimens are undrained (platens are not vented). Thus the strength values determined are in terms of total stress, that is, are not corrected for pore pressures. This test method does not include the procedures necessary to obtain a stress-strain curve beyond the ultimate strength.

ASTM D7013: Standard Guide for Nuclear Surface Moisture and Density Gauge Calibration Facility Setup
This guide outlines procedures for setup of nuclear moisture density gauge in shielded, Method A, and unshielded, Method B, configurations.

ASTM D7015: Standard Practices for Obtaining Intact Block (Cubical and Cylindrical) Samples of Soils
These practices outline the procedures for obtaining intact block (cubical and cylindrical) soil samples. Intact block samples are obtained for laboratory tests to determine the strength, consolidation, permeability, and other geotechnical engineering or physical properties of the intact soil.

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ASTM D7045: Standard Guide for Optimization of Groundwater Monitoring Constituents for Detection Monitoring Programs for RCRA Waste Disposal Facilities
This standard provides a general method of selecting effective constituents for detection monitoring programs at RCRA Waste Disposal Facilities. The process described in this standard presents a methodology that takes into consideration physical and chemical characteristics of the source material(s), the surrounding hydrogeologic regime, and site-specific geochemistry to identify and select those parameters that provide most effective detection of a potential release from a waste management unit (WMU).

ASTM D7046: Standard Guide for Use of the Metal Detection Method for Subsurface Investigation
This guide summarizes the equipment, field procedures, and interpretation methods for the assessment of subsurface materials using the metal detection method. Metal detectors respond to the presence of both ferrous and nonferrous metals by inducing eddy currents in conductive objects. Metal detectors are either frequency domain (continuous frequency or wave) or time domain (pulsed) systems. A wide range of metal detectors is commonly available. Metal detectors can detect any kind of metallic material, including both ferrous metals such as iron and steel, and non-ferrous metals such as aluminum and copper. In contrast, magnetometers only detect ferrous metals. Metal detector measurements can be used to detect the presence of buried metal trash, drums (Tyagi et al, 1983) **(1)** and tanks, abandoned wells (Guide D 6285); to trace buried utilities; and to delineate the boundaries of landfill metal and trench metal. They are also used to detect metal based unexploded ordnance (UXO). Benson (1982) **(2)** and U.S. EPA (1993) **(3)** provide an overview of metal detectors.

ASTM D7048: Standard Guide for Applying Statistical Methods for Assessment and Corrective Action Environmental Monitoring Programs
The scope and purpose of this guidance is to present a variety of statistical approaches for assessment, compliance and corrective action environmental monitoring programs. Although the methods provided here are appropriate and often optimal for many environmental monitoring problems, they do not preclude use of other statistical approaches that may be equally or even more useful for certain site-specific applications.

ASTM D7069: Standard Guide for Field Quality Assurance in a Groundwater Sampling Event
Field QA demonstrates the effectiveness of field quality control procedures. Effective QA facilitates the collection of statistically significant data that is defendable scientifically and in a court of law. QA also involves the use of consistent procedures, increasing the validity of data comparison among sampling locations and events. This guide should be used by a professional or technician who has training or experience in groundwater sampling. This guide covers the quality assurance (QA) methods that may be used to assure the validity of data obtained during the sampling of a groundwater monitoring well. QA is any action taken to ensure that performance requirements are met by following standards and procedures. Following QA practices becomes even more critical if the data must be validated in a court of law. Under certain conditions, it may be necessary to follow additional or different QA practices from those

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listed in this guide. QA practices should be based upon data quality objectives, site-specific conditions, and regulatory requirements.

ASTM D7070: Standard Test Methods for Creep of Rock Core Under Constant Stress and Temperature

There are many underground structures that are created for permanent or long-term use. Often, these structures are subjected to an approximately constant load. Creep tests provide quantitative parameters for stability analysis of these structures. The deformation and strength properties of rock cores measured in the laboratory usually do not accurately reflect large-scale in situ properties, because the latter are strongly influenced by joints, faults, inhomogeneities, weakness planes, and other factors. Therefore, laboratory values for intact specimens must be employed with proper judgment in engineering applications.

These test methods cover the creep behavior of intact soft and hard rock core in fixed states of stress and temperature. They specify the apparatus, instrumentation, and procedures for determining the strain as a function of time under sustained load. Hard rocks are those with a maximum axial strain at failure of less than 2%. Soft rocks include such materials as salt and potash, which often exhibit very large strain at failure.

ASTM D7099: Standard Terminology Relating to Frozen Soil and Rock

This terminology includes all of those terms which relate to frozen soils and rocks. It is based on: a list of definitions drawn up by ASTM Sub-Committee D18.19 (ASTM Z6948 Z, WK 3613); ASTM Standards; a list of definitions drawn up by the Canadian Geomorphology Research Group (CGRG); the Glossary of Permafrost and Related Ground-Ice Terms developed by the National Snow and Ice Data Center (NSIDC), at the University of Colorado, at Boulder; the Keys to Soil Taxonomy of the United States Department of Agriculture (USDA); and contributions by a number of individuals.

ASTM D7100: Standard Test Method for Hydraulic Conductivity Compatibility Testing of Soils With Aqueous Solutions

This test method covers hydraulic conductivity compatibility testing of saturated soils in the laboratory with aqueous solutions that may alter hydraulic conductivity (for example, waste related liquids) using a flexible-wall permeameter. A hydraulic conductivity test is conducted until both hydraulic and chemical equilibrium are achieved such that potential interactions between the soil specimen being permeated and the aqueous solution are taken into consideration with respect to the measured hydraulic conductivity.

ASTM D7101: Standard Index Test Method for Determination of Unvegetated Rolled Erosion Control Product (RECP) Ability to Protect Soil from Rain Splash and Associated Runoff Under Bench-Scale Conditions

This index test method establishes the guidelines, requirements and procedures for evaluating the ability of unvegetated rolled erosion control products (RECPs) to protect soils from simulated rainfall (rain splash) and minimal runoff induced erosion. The critical element of this protection is the ability of the unvegetated RECP to absorb the impact force of raindrops, thereby reducing soil particle loosening through "splash" mechanisms, and limiting the ability of runoff to carry the loosened soil particles.

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ASTM D7128: Standard Guide for Using the Seismic-Reflection Method for Shallow Subsurface Investigation

This guide summarizes the technique, equipment, field procedures, data processing, and interpretation methods for the assessment of shallow subsurface conditions using the seismic-reflection method. Seismic reflection measurements as described in this guide are applicable in mapping shallow subsurface conditions for various uses including geologic, geotechnical, hydrogeologic, and environmental uses. The seismic-reflection method is used to map, detect, and delineate geologic conditions including the bedrock surface, confining layers (aquitards), faults, lithologic stratigraphy, voids, water table, fracture systems, and layer geometry (folds). The primary application of the seismic-reflection method is the mapping of lateral continuity of lithologic units and, in general, detection of change in acoustic properties in the subsurface. This guide will focus on the seismic-reflection method as it is applied to the near surface. Near-surface seismic reflection applications are based on the same principles as those used for deeper seismic reflection surveying, but accepted practices can differ in several respects. Near-surface seismic-reflection data are generally high-resolution (dominant frequency above 80 Hz) and image depths from around 6 m to as much as several hundred meters. Investigations shallower than 6 m have occasionally been undertaken, but these should be considered experimental.

ASTM D7207: Standard Test Method for Determination of Unvegetated Rolled Erosion Control Product (RECP) Ability to Protect Sand from Hydraulically-Induced Shear Stresses under Bench-Scale Conditions

This index test method indicates an unvegetated RECP's ability to reduce soil erosion caused by shear stress induced by moving water under bench-scale conditions. Only tangential shear is measured in this method. Radial and uplift forces generated by the circular motion of the water are not measured. This test method is bench-scale and therefore, appropriate as an index test for general soil/product composite behavior under hydraulic shear conditions, and for product quality assurance/conformance testing. The results of this test shall not be interpreted as indicative of field performance. This index test method establishes the guidelines, requirements and procedures for evaluating the ability of unvegetated Rolled Erosion Control Products (RECPs) to protect soil (sand) from hydraulically induced shear stress in a bench-scale apparatus.

ASTM D7208: Standard Test Method for Determination of Temporary Ditch Check Performance in Protecting Earthen Channels from Stormwater-Induced Erosion

This test method covers the guidelines, requirements, and procedures for evaluating the ability of temporary ditch checks to protect earthen channels from stormwater-induced erosion. Critical elements of this protection are the ability of the temporary ditch check to: Slow and/or pond runoff to encourage sedimentation, thereby reducing soil particle transport downstream; Trap soil particles up stream of structure; and decrease soil erosion.

ASTM D7242: Standard Practice for Field Pneumatic Slug (Instantaneous Change in Head) Tests to Determine Hydraulic Properties of Aquifers with Direct Push Groundwater Samplers

This standard practice covers the field methods used to conduct an instantaneous change in head (slug) test when pneumatic pressure is used to initiate the change in head pressure within the well or piezometer. While this practice specifically addresses use of pneumatic initiation of slug tests

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with direct push tools these procedures may be applied to wells or piezometers installed with rotary drilling methods when appropriate.

ASTM D7243: Standard Guide for Measuring the Saturated Hydraulic Conductivity of Paper Industry Sludges

This guide is intended to supplement the methods and procedures described in Test Methods D 5084. When following the recommendations in this guide to test paper sludges, all assumptions and limitations described in Test Methods D 5084 apply. This guide only applies to hydraulic conductivity tests on paper industry sludges where one-dimensional laminar flow of water is imposed using a flexible-wall permeameter. The hydraulic conductivity of sludges, and other porous materials, generally decreases as the degree of water saturation decreases. This guide applies only to water-saturated sludge containing negligible amounts of gas. This guide applies only to permeation of paper industry sludges with water. Information on testing porous materials with liquids other than water can be found in Test Method D 7100. The hydraulic conductivity of paper sludge measured in the laboratory following Test Methods D 5084 and the recommendations in this guide may or may not be comparable to the hydraulic conductivity of in-place sludge. The issue has not been fully investigated. Therefore, the results should be applied to field situations with caution and by qualified personnel. Paper industry sludges are industrial by-products derived from wastewater treatment operations at plants associated with the manufacturing of paper. These sludges typically consist of clay and organic matter. They may also contain low levels of inorganic and organic contaminants and can be rich in microbes. Traditionally, paper industry sludges have been disposed in municipal solid waste landfills or solid waste monofills. However, in the interest of sustainability, applications are being developed where sludges can be used beneficially. One application is using sludge to construct hydraulic barriers (for example, for use in a landfill cap). Such applications generally require that the hydraulic conductivity of the sludge be measured.

ASTM D7299: Standard Practice for Verifying Performance of a Vertical Inclinometer Probe

Inclinometer monitoring programs often run several years or more. During this time, hundreds of surveys can be collected. Each new survey is processed by comparing it to a baseline survey. Over a period of years, normal wear and tear can gradually degrade the probe's ability to produce new surveys that are directly comparable to the baseline survey. This may go unnoticed for some time, because the quality of readings may degrade in very small increments. When function tests are incorporated into an inclinometer monitoring program, the degradation of reading quality can be avoided. Probes that pass the tests can be used with confidence. Probes that fail the tests should be returned to the probe manufacturer for servicing. It should be noted that manufacturers calibrate inclinometer probes using high-precision, electronically-controlled equipment in temperature-controlled environments. Ordinary users do not have access to such equipment, so the pass/fail criteria suggested for these tests accommodate typical results produced by less precise equipment in a less controlled environment. The quality of the result produced by this standard is dependent on the competence of the personnel performing it and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D 3740 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of this standard are cautioned that compliance with Practice D 3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D 3740 provides a

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means of evaluating some of those factors. This practice describes three function tests that together can be used to verify that a vertical inclinometer probe is working properly.

ASTM D7300: Standard Test Method for Laboratory Determination of Strength Properties of Frozen Soil at a Constant Rate of Strain

Understanding the mechanical properties of frozen soils is of primary importance to frozen ground engineering. Data from strain rate controlled compression tests are necessary for the design of most foundation elements embedded in, or bearing on frozen ground. They make it possible to predict the time-dependent settlements of piles and shallow foundations under service loads, and to estimate their short and long-term bearing capacity. Such tests also provide quantitative parameters for the stability analysis of underground structures that are created for permanent or semi-permanent use. It must be recognized that the structure of frozen soil in situ and its behavior under load may differ significantly from that of an artificially prepared specimen in the laboratory. This is mainly due to the fact that natural permafrost ground may contain ice in many different forms and sizes, in addition to the pore ice contained in a small laboratory specimen. These large ground-ice inclusions (such as ice lenses) will considerably affect the time-dependent behavior of full-scale engineering structures. In order to obtain reliable results, high-quality undisturbed representative permafrost samples are required for compression strength tests. The quality of the sample depends on the type of frozen soil sampled, the in situ thermal condition at the time of sampling, the sampling method, and the transportation and storage procedures prior to testing. The best testing program can be ruined by poor-quality samples. In addition, one must always keep in mind that the application of laboratory results to practical problems requires much caution and engineering judgment. This test method covers the determination of the strength behavior of cylindrical specimens of frozen soil, subjected to uniaxial compression under controlled rates of strain. It specifies the apparatus, instrumentation, and procedures for determining the stress-strain-time, or strength versus strain rate relationships for frozen soils under deviatoric creep conditions.

ASTM D7315: Standard Test Method for Determination of Turbidity Above 1 Turbidity Unit (TU) in Static Mode

This test method covers the static determination of turbidity in water. Static refers to a sample that is removed from its source and tested in an isolated instrument. This test method is applicable to the measurement of turbidities greater than 1.0 turbidity unit (TU). The upper end of the measurement range was left undefined because different technologies described in this test method can cover very different ranges. The round robin study covered the range of 0-4000 turbidity units because instrument verification in this range can typically be covered by standards that can be consistently reproduced.

ASTM D7322: Standard Test Method for Determination of Rolled Erosion Control Product (RECP) Ability to Encourage Seed Germination and Plant Growth under Bench-Scale Conditions

This test method covers guidelines, requirements, and procedures for evaluating the effect of Rolled Erosion Control Products (RECPs) on seed germination and vegetation enhancement.

ASTM D7351: Standard Test Method for Determination of Sediment Retention Device Effectiveness in Sheet Flow Applications

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This test method quantifies the ability of a sediment retention device (SRD) to retain eroded sediments caused by sheet flowing water under full-scale conditions. This test method may also assist in identifying physical attributes of SRDs that contribute to their erosion control performance. The effectiveness of SRDs is installation dependent. Thus, replicating field installation techniques is an important aspect of this test method. This test method is full-scale and therefore, appropriate as an indication of product performance, for general comparison of product capabilities, and for assessment of product installation techniques. This test method establishes the guidelines, requirements and procedures for evaluating the ability of Sediment Retention Devices (SRDs) to retain sediment when exposed to sediment-laden water "sheet" flows.

ASTM D7367: Standard Test Method for Determining Water Holding Capacity of Fiber Mulches for Hydraulic Planting

The meaning of the test is related to the manufacturing and end use of the material, to determine characteristics of products. The water holding capacity of hydraulically applied mulches for hydraulic planting correlates directly with enhanced slurry and spray patterns by providing better soil/slurry binding ability and rate of seed germination. This quantitative test method determines water holding capacity of fiber mulches, including wood, paper, and agriculturally derived and blended fiber mulches used for hydraulic planting.

ASTM D7382: Standard Test Methods for Determination of Maximum Dry Unit Weight and Water Content Range for Effective Compaction of Granular Soils Using a Vibrating Hammer

For many cohesionless, free-draining soils, the maximum dry unit weight is one of the key components in evaluating the state of compactness of a given soil mass that is either naturally occurring or is constructed (fill). Soil placed as an engineered fill is compacted to a dense state to obtain satisfactory engineering properties such as shear strength, compressibility, permeability, or combinations thereof. Also, foundation soils are often compacted to improve their engineering properties. Laboratory compaction tests provide the basis for determining the percent compaction and water content needed at the time of compaction to achieve the required engineering properties, and for controlling construction to assure that the required unit weights and water contents are achieved. It is generally recognized that percent compaction is a good indicator of the state of compactness of a given soil mass. However, the engineering properties, such as strength, compressibility, and permeability of a given soil, compacted by various methods to a given state of compactness can vary considerably. Therefore, considerable engineering judgment must be used in relating the engineering properties of soil to the state of compactness. Degradation—Soils containing particles that degrade during compaction are a problem, especially when more degradation occurs during laboratory compaction than field compaction, as is typical. Degradation typically occurs during the compaction of a granular-residual soil or aggregate. When degradation occurs, the maximum dry unit weight increases so that the laboratory maximum value is not representative of field conditions. Often, in these cases, the maximum dry unit weight is impossible to achieve in the field. One method to design and control the compaction of such soils is to use a test fill to determine the required degree of compaction and the method to obtain that compaction, followed by the use of a method specification to control the compaction. Components of a method specification typically contain the type and size of compaction equipment to be used, the lift thickness, and the number of passes. These test

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methods cover the determination of the maximum dry unit weight and water content range for effective compaction of granular soils. A vibrating hammer is used to impart a surcharge and compactive effort to the soil specimen.

ASTM D7384: Standard Practice for Minimum Geospatial Data for a Coal Surface Mining Permit Boundary

This practice covers the minimum elements for the accurate location and description of data for defining a coal surface mining permit boundary. This practice addresses coal mining geospatial data relative to the Surface Mining Control and Reclamation Act of 1977 (SMCRA). This geospatial data shall be obtained from each state or federal, or both, coal mining Regulatory Authority (RA) authorized under SMCRA to regulate coal surface mining operations (CSMO). Each RA shall be the authoritative data source (ADS) for coal mining geospatial data. As used in this practice, a coal surface mining boundary represents an area where coal removal and reclamation has occurred or is occurring within a defined CSMO.

ASTM D7400: Standard Test Methods for Downhole Seismic Testing

The seismic downhole method provides a designer with information pertinent to the seismic wave velocities of the materials in question. The P-wave and S-wave velocities are directly related to the important geotechnical elastic constants of Poisson's ratio, shear modulus, bulk modulus, and Young's modulus. Accurate in-situ P-wave and S-wave velocity profiles are essential in geotechnical foundation designs. These parameters are used in both analyses of soil behavior under both static and dynamic loads where the elastic constants are input variables into the models defining the different states of deformations such as elastic, elasto-plastic, and failure. Another important use of estimated shear wave velocities in geotechnical design is in the liquefaction assessment of soils. A fundamental assumption inherent in the test methods is that a laterally homogeneous medium is being characterized. In a laterally homogeneous medium the source wave train trajectories adhere to Snell's law of refraction. Another assumption inherent in the test methods is that the stratigraphic medium to be characterized can have transverse isotropy. Transverse isotropy is a particularly simple form of anisotropy because velocities only vary with vertical incidence angle and not with azimuth. By placing and actuating the seismic source at offsets rotated 90° in plan view; it may be possible to evaluate the transverse anisotropy of the medium. These test methods are limited to the determination of the interval velocities from arrival times and relative arrival times of compression (P) and vertically (SV) and horizontally (SH) polarized shear (S) seismic waves which are generated near surface and travel down to an array of vertically installed seismic sensors. A preferred method intended to obtain data for use on critical projects where the highest quality data is required is included. Also included is an optional method intended for use on projects which do not require measurements of a high degree of precision.

ASTM D7512: Standard Guide for Monitoring of Suspended-Sediment Concentration in Open Channel Flow Using Optical Instrumentation

This guide covers the equipment and basic procedures for installation, operation, and calibration of optical equipment as a surrogate for the continuous determination of suspended-sediment concentration (SSC) in open channel flow. This guide emphasizes general principles for the application of optical measurements to be used to estimate suspended-sediment concentration

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(SSC) in water. Only in a few instances are step-by-step instructions given. Continuous monitoring is a field-based operation, methods and equipment are usually modified to suit local conditions. The modification process depends upon the operator skill and judgment. This guide covers the use of the output from an optical instrument, such as turbidity and suspended-solids meters, to record data that can be correlated with suspended-sediment concentration. It does not cover the process of collecting data for continuous turbidity record, which would require additional calibration of the turbidity readings to the mean turbidity of the measurement cross section. For the purposes of this method it is assumed that the dependent variable will be mean cross-sectional suspended-sediment concentration data.

ASTM E617: Standard Specification for Laboratory Weights and Precision Mass Standards
This specification covers weights and mass standards used in laboratories, specifically classes 0, 1, 2, 3, 4, 5, 6 and 7. This specification replaces National Bureau of Standards Circular 547, Section, which is out of print.

ASTM METHODS FOR WATER SAMPLES

ASTM D511: Standard Test Methods for Calcium and Magnesium in Water
These test methods cover the determination of calcium and magnesium in water by complexometric titration and atomic absorption spectrophotometer procedures.

ASTM D512: Standard Test methods for Chloride Ion in Water
These test methods cover the determination of chloride ion in water, wastewater and brines. The following three methods are included (a) Mercurimetric Titration, (b) Silver Nitrate Titration, and (c) Ion Selective Electrode.

ASTM D513: Standard Test Methods for Total and Dissolved Carbon Dioxide in Water
These test methods provide for the measurement of total or dissolved carbon present as carbon dioxide (CO_2), carbonic acid, bicarbonate ion, and carbonate ion in water. Two methods are included: (a) Gas Sensing Electrode, and (b) CO_2 Evolution Colorimetric Titration.

ASTM D516: Standard Test Method for Sulfate Ion in Water
This turbidimetric test method covers the determination of sulfate in water in the range from 1 to 40 mg/L of sulfate ion (SO_4).

ASTM D596: Standard Guide for Reporting Results of Analysis of Water
This guide provides guidelines for reporting inorganic and organic results of analyses of drinking water, wastewater, process water, groundwater, and surface water to laboratory clients in a complete and systematic fashion.

ASTM D807: Standard Practice for Assessing the Tendency of Industrial Boiler Waters to Cause Embrittlement (USBM[1]) Embrittlement Detector Method
This practice, known as the embrittlement-detector method, covers the apparatus and procedure for determining the embrittling or nonembrittling characteristics of the water in an operating boiler.

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ASTM D857: Standard Test Method for Aluminum in Water
This test method covers the direct flame atomic absorption determination of aluminum in the nitrous oxide-acetylene flame.

ASTM D858: Standard Test Methods for Manganese in Water
These test methods cover the atomic absorption determination of dissolved and total recoverable manganese in water and certain wastewaters. Three test methods are given.

ASTM D859: Standard Test Method for Silica in Water
This test method covers the determination of silica in water and wastewater.
This test method is a colorimetric method that determines molybdate-reactive silica. It is applicable to most waters, but some waters may require filtration and dilution to remove interferences from color and turbidity. This test method is useful for concentrations as low as 20 μg/L. This test method covers the photometric determination of molybdate-reactive silica in water. Due to the complexity of silica chemistry, the form of silica measured is defined by the analytical method as molybdate-reactive silica. Those forms of silica that are molybdate-reactive include dissolved simple silicates, monomeric silica and silicic acid, and an undetermined fraction of polymeric silica.

ASTM D887: Standard Practices for Sampling Water-Formed Deposits
These practices cover the sampling, physical, biological, or radiological analysis. The practices cover both field and laboratory sampling. It also defines the various type of deposits.

ASTM D888: Standard Test Method for Dissolved Oxygen in Water
These test methods cover the determination of dissolved oxygen in water. Three procedures are included: (a) Titrimetric – High Level, (b) Electrochemical probe, and (c) Luminescence-based sensor

ASTM D932: Standard Test Method for Iron Bacteria in Water and Water Deposits
This test method covers the determination of iron bacteria by examination under the microscope. The method provides for the identification of the following genera of bacteria found in water and water-formed deposits: *Sidero-capsa*, *Gallionella* (*Dioymohelix*), *Sphaerotilus, Crenothrix*, *Leptothrix*, and *Clonothrix*.

ASTM D1066: Standard Practice for Sampling Steam
This practice covers the sampling of saturated and superheated steam. It is applicable to steam produced in fossil fired and nuclear boilers or by any other process means that is at a pressure sufficiently above atmospheric to establish representative sample flow. It is also applicable to steam at a lower and sub atmospheric pressures for which means must be provided to establish representative flow.

ASTM D1067: Standard Test Methods for Acidity or Alkalinity of Water
These test methods cover the determination of acidity or alkalinity of all types of water. Three test methods are given as follows: (a) Electrometric Titration, (b) Electrometric or Color Change Titration, and (c) Color-Change Titration after Hydrogen Peroxide Oxidation and Boiling.

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ASTM D1068: Standard Test Methods for Iron in Water
These test methods cover the determination of iron in water. Procedures are given for determining total iron, dissolved iron, and ferrous iron. Undissolved iron may be calculated from the total iron and dissolved iron determinations. The test methods are given as follows: (a) Atomic Absorption, (b) Atomic Absorption–Graphite Furnace, and (c) Photometric-Bathophenanthroline

ASTM D1125: Standard Test Methods for Electrical Conductivity and Resistivity of Water
These test methods cover the determination of the electrical conductivity and resistivity of water. The following test methods are included: (a) Field and Laboratory Measurement of Static (non-flowing) Samples, and (b) Continuous In-Line Measurement.

ASTM D1126: Standard Test Method for Hardness in Water
This test method covers the determination of hardness in water by titration. This test method is applicable to waters that are clear in appearance and free of chemicals that will complex calcium or magnesium. The lower detection limit of this test method is approximately 2 to 5 mg/L as $CaCO_3$, the upper limit can be extended to all concentrations by sample dilution. It is possible to differentiate between hardness due to calcium ions and that due to magnesium ions by this test method.

ASTM D1129: Standard Terminology Relating to Water[1,2]
A list of terms and abbreviations related to water analysis.

ASTM D1179: Standard Test Method for Fluoride Ion in Water
These test methods cover the determination of fluoride ion in water. The following two test methods are given: (a) Distillation and subsequent measurement by Ion Selective Electrode, and (b) Ion Selective Electrode Analysis of simple fluoride without a preliminary distillation.

ASTM D1192: Standard Guide for Equipment for Sampling Water and Steam in Closed Conduits
This guide covers only that equipment commonly used for the sampling of water and steam in closed conduits. It does not cover specialized equipment required for and unique to a specific test method of analysis. Items such as valves, fittings, piping/tubing, cooling coils and condensers, filters, pumps, sample containers, and packaging materials are included but items such as sampling nozzles that are used for obtaining steam or water samples from their source and apparatus used in subsequent methods of test and analysis are excluded.

ASTM D1246: Standard Test Method for Bromide Ion in Water
This test method is applicable to the measurement of bromide ion in water, groundwater, and drinking water. Bromide is measured potentiometrically using a Bromide Ion Selective Electrode.

ASTM D1252: Standard Test Method for Chemical Oxygen Demand (Dichromate Oxygen Demand) of Water.

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These test methods cover the determination of the quantity of oxygen that certain impurities in water will consume, based on the reduction of a dichromate solution under specified conditions. The following test methods are included: (a) macro COD by reflux digestion and titration, and (b) micro COD by sealed digestion and spectrometry.

ASTM D1253: Standard Test Method for Residual Chlorine in Water
This test method covers the determination of residual chlorine in water by direct amperometric titration with phenylarsine oxide.

ASTM D1291: Standard Practice for Estimation of Chlorine Requirement or Demand of Water, or Both
This practice provides a means of estimating the quantity of chlorine required to be added to a unit volume of water to accomplish a predetermined treatment objective or to completely react with all chlorine reactable substances in the water or both.

ASTM D1292: Standard Test Method for Odor in Water
This test method covers the determination of the odor (that is, the property that affects the sense of smell) of water. A suggested system for classifying odors is given as annex A1. The test method is applicable to the determination of odor intensity in terms of odor intensity index or threshold odor number.

ASTM D1293: Standard Test Methods for pH of Water
These test methods cover the determination of pH by electrometric measurement using the glass electrode as the sensor. Two test methods are given as follows: (a) precise laboratory measurement and, (b) routine or continuous measurement

ASTM D1385: Standard Test Method for Hydrazine in Water
This test method covers the colorimetric determination of hydrazine in boiler feed waters, condensates, natural, and well waters that have been treated with hydrazine(N_2H_4). This test method is usable in the range from 5.0 to 200ug/L(ppb) hydrazine. The range is for photometric measurements made at 458 nm in 50 mm cell. Higher concentrations of hydrazine can also be determined by taking a more diluted sample.

ASTM D1426: Standard Test Methods for Ammonia in Water
These test methods cover the determination of ammonia nitrogen, exclusive of organic nitrogen, in water. Two test methods are included as follows: (a) direct Nessler, and (b) Ion Selective Electrode.

ASTM D1429: Standard Test Methods for Specific Gravity of Water and Brine
These test methods cover the determination of the specific gravity of water and brine free of separable oil, as follows: (a) pycnometer, (b) balance, (c) Erlenmeyer flask, and (d) hydrometer

ASTM D1498: Standard Practice for Oxidation-Reduction Potential of Water
This practice covers the apparatus and procedure for the electrometric measurement of oxidation – reduction potential (ORP) in water. It does not deal with the manner in which the solutions are prepared, the theoretical interpretation of the oxidation-reduction potential, or the establishment

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of a standard oxidation-reduction potential for any given system. The practice described has been designed for the routine and process measurement of oxidation-reduction potential.

ASTM D1687: Standard Test Methods for Chromium in Water
These test methods cover the determination of hexavalent and total chromium in water. Three test methods are included as follows: (a) photometric-diphenylcarbazide, (b) atomic absorption – direct, and (c) atomic absorption – graphite furnace

ASTM D1688: Standard Test Methods for Copper in Water
These test methods cover the determination of copper in water by atomic absorption spectrophotometry. Three test methods are included as follows: (a) atomic absorption direct, (b) atomic absorption – chelation extraction, and (c) atomic absorption – graphite furnace.

ASTM D1691: Standard Test methods for Zinc in water
These test methods cover the determination of zinc in water. Two methods are given as follows: (a) atomic absorption – direct, and (b) atomic absorption – chelation extraction

ASTM D1886: Standard test methods for nickel in water
These test methods cover the atomic absorption determination of nickel in water and wastewaters. Three test methods are given as follows: a) atomic absorption direct, (b) atomic absorption – chelation extraction, and (c) atomic absorption–graphite furnace.

ASTM D1890: Standard Test Method for Beta Particle Radioactivity of Water
This test method covers the measurement of beta particle activity of water, as referenced to the beta energy of ^{137}Cs, not corrected for conversion electrons. It is applicable to beta emitters having maximum energies above 0.1 MeV and at activity levels above 0.02 Bq/mL of radioactive homogeneous water most counting systems. This test method is not applicable to samples containing radionuclides that are volatile under conditions of the analysis.

ASTM D1889: Standard Test method for Turbidity of Water
This test method covers the determination of turbidity in water and wastewater.

ASTM D1941: Standard Test Method for Open Channel Flow Measurement of Water with the Parshall Flume
This test method covers measurement of the volumetric flow rate of water and wastewater in open channels with the Parshall Flume.

ASTM D1943: Standard Test Method for Alpha Particle Radioactivity of Water
This test method covers the measurement of alpha particle activity of water. It is applicable to alpha emitters having maximum energies above 3.9 MeV and at activity levels of 0.02 Bq/mL of radioactive homogeneous water. This test method is not applicable to samples containing alpha-emitting radionuclide that are volatile under conditions of the analysis.

ASTM D1971: Standard Practices for Digestion of Water Samples for Determination of Metals by Flame Atomic Absorption, Graphite Furnace Atomic Absorption, Plasma Emission Spectroscopy, or Plasma Mass Spectrometry

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Most atomic absorption and plasma emission spectroscopy, and plasma-mass spectrometric test methods require that the metals of interest be dissolved in a liquid phase before being introduced into the spectrophotometer. These practices describe digestion or dissolution procedures whereby analyte metals associated with the solid fraction of a sample can be brought into solution for subsequent analysis. The following practices are included:

Practice A-Digestion with Mineral Acids and Elevated Pressure
Practice B-Digestion with Mineral Acids and Heating at Atmospheric Pressure
Practice C-In-bottle digestion with Mineral Acids

These practices have been demonstrated to be applicable to a wide variety of sample types and sample matrices, and in many cases, will give complete dissolution of the analyte metals of interest. They are by no means the only digestion procedures available.

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ASTM D1976: Standard Test Method for Elements in Water by Inductively-Coupled Argon Plasma Atomic Emission Spectroscopy

This test method covers the determination of dissolved, total-recoverable, or total elements in drinking water, surface water, domestic, or industrial wastewaters. Table 1 of the standard lists elements for which this test method applies, with recommended wavelengths and typical estimated instrumental detection limits using conventional pneumatic nebulization. Actual working detection limits are sample dependent and as the sample matrix varies, these detection limits may also vary.

ASTM D2036: Standard Test Methods for Cyanides in Water.

This test method covers the determination of cyanides in water. The following test methods are included: (a) total cyanides after distillation, (b) cyanide amenable to chlorination – by difference, (c) weak acid dissociable cyanides, and (d) cyanides amenable to chlorination without distillation (short cut method).

ASTM D2331: Standard Practices for Preparation and Preliminary Testing of Water-Formed Deposits.

These practices provide directions for the preparation of the sample for analysis, the preliminary examination of the sample, and methods for dissolving the analytical sample or selectively separating constituents of concern.

ASTM D2332: Standard Practice for Analysis of Water-Formed Deposits by Wavelength-Dispersive X-ray Fluorescence.

This practice covers X-ray spectrochemical analysis of water-formed deposits. Certain elements present in water-formed deposits are identified. Concentration levels of the elements are estimated. Deposit analysis assists in providing proper water conditioning. Deposits formed from or by water in all its phases may be further classified as scale, sludge, corrosion products, or biological deposits. The overall composition of a deposit or some part of a deposit may be determined by chemical or spectrographic analysis; the constituents actually present as chemical substances may be identified by microscope or X-ray diffraction studies. Organisms may be identified by microscopical or biological methods.

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ASTM D2460: Standard Test Method for Alpha-Particle-Emitting Isotopes of Radium in Water.
This test method covers the separation of dissolved radium from water for the purpose of measuring its radioactivity. Although all radium isotopes are separated, the test method is limited to alpha-particle-emitting isotopes by choice of radiation detector. This most important of these radioisotopes are radium-223, radium-224 and radium-226. The lower limit of concentration to which this test method is applicable is $3.7 * 10^{-2}$ Bq/L (pCi/L)

ASTM D2688: Standard Test Methods for Corrosivity of Water in the Absence of Heat Transfer (Weight Loss Methods)
This test method covers the determination of the corrosivity of water by evaluating pitting and by measuring the weight loss of metal specimens. Pitting is a form of localized corrosion: weight loss is a measure of the average corrosion rate. The rate of corrosion of a metal immersed in water is a function of the tendency for the metal to corrode and is also a function of the tendency for water and the materials it contains to promote (or inhibit) corrosion. The test method employs flat, rectangular-shaped metal coupons which are mounted on pipe plugs and exposed to the water flowing in metal piping in municipal, building, and industrial water systems using a side stream corrosion specimen rack.

ASTM D2791: Standard Test Method for On-line Determination of Sodium in Water
This test method covers the on-line determination of trace amounts of sodium in water using an ion-selective electrode.

ASTM D2907: Standard Test Methods for Microquantities of Uranium in Water by Fluorometry.
These test methods cover the determination of microquantities of uranium in water in the concentration range from 0.005 to 50 mg/l. The following test methods are included: (a) direct fluorometric, and (b) extraction fluorometric.

ASTM D2908: Standard Practice for Measuring Volatile Organic Matter in Water by Aqueous-Injection Gas Chromatography.
This practice covers general guidelines applicable to certain test methods for the qualitative and quantitative determination of specific organic compounds, or classes of compounds, in water by direct aqueous injection gas chromatography.

ASTM D2972: Standard Test Methods for Arsenic in Water
These test methods cover the photometric and atomic absorption determination of arsenic in most waters and wastewaters. Three test methods are given as follows: (a) silver diethyldithiocarbamate colorimetric, (b) atomic absorption – hydride generation, and (c) atomic absorption graphite furnace.

ASTM D3082: Standard Test Method for Boron In Water
This test method covers the determination of boron in water and wastewaters by the curcumin colorimetric-extraction method in concentrations between 0.1 and 1.0 mg/L. The range can be

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extended by dilution of the sample. Only dissolved boron is determined. This test method requires that the water sample be filtered through a 0.45-μm membrane filter before analysis.

ASTM D3223: Standard Test Method for Total Mercury in Water
This test method covers the determination of total mercury in water in the range from 0.5 to 10.0 μg Hg/L (1). The test method is applicable to fresh waters, saline waters, and some industrial and sewage effluents. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices. Both organic and inorganic mercury compounds may be analyzed by this procedure if they are first converted to mercuric ions. Using potassium persulfate and potassium permanganate as oxidants, and a digestion temperature of 95°C, approximately 100 % recovery of organomercury compounds can be obtained.

ASTM D3352: Standard Test Method for Strontium Ion in Brackish Water, Seawater, and Brines
This test method covers the determination of soluble strontium ion in brackish water, seawater, and brines by atomic absorption spectrophotometry. Samples containing from 5 to 2100 mg/L of strontium may be analyzed by this test method.

ASTM D3372: Standard Test Method for Molybdenum in Water
This test method covers the determination of dissolved and total recoverable molybdenum in most waters, wastewaters, and brines by atomic absorption spectroscopy. This test method is applicable in the range from 1 to 25 g/L of molybdenum. The range may be extended by dilution of the sample.

ASTM D3373: Standard Test Method for Vanadium in Water
This test method covers the determination of dissolved and total recoverable vanadium in most waters and wastewater by graphite furnace atomic absorption spectrophotometry. The optimum range of this test method is 10 to 200 μg/L of vanadium based on a 20-L sample size. Concentrations higher than 200 g/L may be determined by proper dilution of sample. A detection level as low as 4 μg/L of vanadium has been reported.

ASTM D3454: Standard Test Method for Radium-226 in Water
This test method covers the measurement of soluble, suspended, and total radium-226 in water in concentrations above $3.7 * 10^{-3}$ Bq/L. This test method is not applicable to the measurement of other radium isotopes.

ASTM D3557: Standard Test Methods for Cadmium in Water
These test methods cover the determination of dissolved and total recoverable cadmium in water and wastewater. Four test methods are included as follows: (a) atomic absorption–direct, (b) atomic absorption–chelation extraction, (c) differential pulse anodic stripping voltammetry, and (d) atomic absorption–graphite furnace.

ASTM D3559: Standard Test Methods for Lead in Water
These test methods cover the determination of dissolved and total recoverable lead in water and wastewater by atomic-absorption spectrophotometry and differential pulse anodic stripping voltammetry. Four test methods are included as follows: a) atomic absorption–direct, b) atomic

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absorption–chelation extraction, c) differential pulse anodic stripping voltammetry, and d) atomic absorption– raphite furnace.

ASTM D3561: Standard Test Methods for Lithium, Potassium, and Sodium Ions of Brackish Water, Seawater, and Brines by Atomic Absorption Spectrophotometry.
This test method covers the determination of soluble lithium, potassium, and sodium ions in brackish water, seawater and brines by atomic absorption spectrophotometry. Samples containing from 0.1 to 70 000 mg/l of lithium, potassium, and sodium may be analyzed by this test method.

ASTM D3590: Standard Test Methods for Total Kjeldahl Nitrogen in Water
These test methods cover the determination of total Kjeldahl nitrogen. The following test methods are included: (a) manual digestion and distillation followed by titration, direct Nesslerization, or direct potentiometry, and (b) manual digestion followed by the semi-automated Berthelot colorimetric method.

ASTM D3645: Standard Test Methods for Beryllium in Water
These test methods cover the determination of dissolved and total recoverable beryllium in most waters and wastewaters: (a) atomic absorption–direct and (b) atomic absorption–graphite furnace.

ASTM D3648: Standard Practices for the Measurement of Radioactivity
These practices cover a review of the accepted counting practices currently used in radiochemical analyses. The general information sections contain information applicable to all types of radioactive measurements, while each of the other sections id specific for a particular type of radiation.

ASTM D3649: Standard Practice for High-Resolution Gamma-Ray Spectrometry of Water
This practice covers the measurement of gamma-ray emitting radionuclides in water by means of gamma-ray spectrometry. It is applicable to nuclides emitting gamma-rays with energies greater than 20 keV. For typical counting system and samples types, activity levels of about 40 Bq are easily measured and sensitivities as low as 0.4 Bq are found for many nuclides. Count rates in excess of 2000 counts per second should be avoided because of electronic limitations. High count rate samples can be accommodated by dilution or by increasing the sample to detector distance.

ASTM D3651: Standard Test Method for Barium in Brackish Water, Seawater, and Brines
This test method covers the determination of soluble barium ion in brackish water, sea-water, and brines by atomic absorption spectrophotometry. The actual working range of this test method is 1 to 5 mg/L barium.

ASTM D3697: Standard Test Method for Antimony in Water
This test method covers the determination of dissolved and total recoverable antimony in water by atomic absorption spectroscopy. This test method is applicable in the range from 1 to 15 g/L of antimony. The range may be extended by less scale expansion or by dilution of the sample.

ASTM D3859: Standard Test Methods for Selenium in Water

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These test methods cover the determination of dissolved and total recoverable selenium in most waters and wastewaters. Both test methods utilize atomic absorption procedures, as follows: (a) atomic absorption – gaseous hydride, and (b) atomic absorption – graphite furnace. These test methods are applicable to both inorganic and organic forms of dissolved selenium. They are applicable also to particulate forms of the element, provided that they are solubilized in the appropriate acid digestion step. However, certain selenium-containing heavy metallic sediments may not undergo digestion.

ASTM D3865: Standard Test Method for Plutonium in Water
This test method covers the determination of alpha-particle-emitting isotopes of plutonium concentrations over 0.01 Bq/L (0.3 pCi/L) in water by means of chemical separations and alpha pulse-height analysis (alpha-particle spectrometry). The isotopes, ^{239}Pu, ^{240}Pu, and ^{238}Pu, are chemically separated from 1-L water sample by coprecipitation with ferric hydroxide, anion exchange and elcectrodeposition. The test method applies to soluble plutonium and to suspended particulate matter containing plutonium. In the latter situation, an acid dissolution step is required to assure that all of the plutonium dissolves.

ASTM D3866: Standard Test Methods for Silver in Water
These test methods cover the atomic absorption determination of silver in water. Three test methods are given as follows: (a) atomic absorption – chelation extraction, (b) atomic absorption – direct, and (c) atomic absorption – graphite furnace.

ASTM D3867: Standard Test Methods for Nitrite-Nitrate in Water
These test methods cover the determination of nitrite nitrogen, nitrate nitrogen, and combined nitrite-nitrate nitrogen in water and wastewater in the range from 0.05 to 1.0 mg/L nitrogen. Two test methods are given as follows: (a) automated cadmium reduction, and (b) manual cadmium reduction.

ASTM D3868: Standard Test Method for Fluoride Ions in Brackish Water, Seawater, and Brines
This test method covers the determination of soluble fluoride ion in brackish water, seawater and brines by use of a fluoride selective electrode and standard additions.

ASTM D3869: Standard Test Method for the Determination of Soluble Iodide and Bromide Ions in Brackish Water, Seawater and Brines
These test methods cover the determination of soluble iodide and bromide ions, or both in brackish water, seawater, and brines. The following test methods are included: (a) for both iodide and bromide ions – volumetric, (b) for iodide ion – colorimetric, (c) for iodide ion – selective electrode, and (d) for bromide ion – colorimetric.

ASTM D3875: Standard Test Method for Alkalinity in Brackish Water, Seawater, and Brines
This test method covers the determination of alkalinity in brackish water, seawater, and brines by potentiometric titration.

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ASTM D3919: Standard Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry

This practice covers the general considerations for the quantitative determination of trace elements in water and wastewater by graphite furnace atomic absorption spectrophotometry. Furnace atomizers are a most useful means of extending detection limits; however, the practice should only be used at concentration levels below the optimum range of direct flame aspiration atomic absorption spectrophotometry. Because of differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided for each instrument. Instead, the analyst should follow the instructions provided by the manufacturer of a particular instrument. Wavelengths, estimated detection limits, and optimum concentration ranges are given in the individual methods. Ranges may be increased or decreased by varying the volume of sample injected or the instrumental settings or by the use of a secondary wavelength. Samples containing concentrations higher than those given in the optimum range may be diluted or analyzed by other techniques. This technique is generally not applicable to brines and seawater. Special techniques such as separation of the trace elements from the salt, careful temperature control through ramping techniques, or matrix modification may be useful for these samples.

ASTM D3920: Standard Test Method for Strontium in Water

This test method covers the determination of dissolved and total recoverable strontium in water and wastewater by atomic absorption spectroscopy. The test method is applicable in the range from 0.1 to 1 mg/L of strontium. The range may be extended by dilution of the original sample.

ASTM D3972: Standard Test Method for Isotopic Uranium in Water by Radiochemistry

This test method covers the determination of alpha-particle-emitting isotopes of uranium in water by means of chemical separations and alpha pulse-height analysis (also known as alpha-particle spectrometry). Uranium is chemically separated from a water sample by coprecipitation with ferrous hydroxide, anion exchange, and electrodeposition. The test method applies to soluble uranium as well as to any uranium that might be present in suspended matter in the water sample. This test method is applicable for uranium processing effluents as well as substitute ocean water. When suspended matter is present, an acid dissolution step is added to assure that all of the uranium dissolves. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

ASTM D3974: Standard Practices for Extraction of Trace Elements from Sediments

These practices describe the partial extraction of soils, bottom sediments, suspended sediments, and waterborne materials to determine the extractable concentrations of certain trace elements.

1. Practice A is capable of extracting concentrations of aluminum, boron, barium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, molybdenum, nickel, potassium, sodium, strontium, vanadium, and zinc from the preceding material. Other metals may be determined using this practice. This extraction is the more vigorous and more complicated of the two.
2. Practice B is capable of extracting concentration of aluminum, cadmium, chromium, cobalt, copper, iron, lead, manganese, nickel, and zinc from the preceding materials. Other metals may be determined using this practice. This extraction is less vigorous and less complicated than practice A.

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ASTM D3976: Standard Practice for Preparation of Sediment Samples for Chemical Analysis
This practice describes standard procedures for preparation of test samples (including the removal of occluded water and moisture) of field samples collected from locations such as streams, rivers, ponds, lakes, and oceans.

ASTM D3977: Standard Test Methods for Determining Sediment Concentration in Water Samples
These test methods cover the determination of sediment concentrations in water and wastewater samples collected from lakes and other quiescent-water bodies, concentrations of sediment in samples are nearly equal to concentrations at sampling points; in most instances, sample concentrations are not strongly influenced by collection techniques. In rivers and other flowing bodies, concentrations of sediment in samples depend upon the manner in which the samples are collected. Concentrations in isokinetically-collected samples can be multiplied by water discharges to obtain sediment discharges in the vicinity of the sampling ponts.

ASTM D3986: Standard Test Method for Barium in Brines, Seawater, and Brackish Water by Direct-Current Argon Plasma Atomic Emission Spectroscopy
This test method covers the determination of dissolved and total recoverable barium in brines, seawater, and brackish waters by direct-current argon plasma atomic emission spectroscopy (DCP-AES). This test method has been tested in the range from 10 mg/L to 20 mg/L. Samples shall be diluted to contain concentrations within the calibration range. Higher concentrations can also be determined by changing to a less sensitive emission line. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

ASTM D4013: Standard Practice for Preparation of Test Specimens of Bituminous Mixtures by Means of Gyratory Shear Compactor
This practice covers the preparation of 10-cm or 4-in. diameter test specimens of bituminous mixtures containing 22.4-mm (7/8-in.) aggregate.

ASTM D4025: Standard Practice for Reporting Results of Examination and Analysis of Deposits Formed from Water for Subsurface Injection
This practice covers the manner in which the various results of examination and analysis to determine the composition of deposits formed from water for subsurface injection are to be reported. All analyses shall be made in accordance with the test methods of ASTM, unless otherwise specified. Note 1—While reporting of inorganic constituents in water-formed deposits as specified in Test Methods D 4412 is sufficient for certain industries, this practice provides for the reporting of organic and biological materials as well as inorganic constituents. Note 2—Consistent with practices in industries where subsurface injection of water is practiced, reporting includes specifying of inorganic constituents as probable molecular combinations of the species for which analyses are performed.

ASTM D4107: Standard Test Method for Tritium in Drinking Water

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This test method covers the determination of tritium in drinking water (as T2O or HTO) by liquid scintillation counting of the tritium beta particle activity. This test method was used successfully with drinking water. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices. 1.3 The tritium concentrations which can be measured by this test method utilizing currently available liquid scintillation instruments, range from less than 0.037 Bq/mL (1 pCi/mL) to 555 Bq/mL (15000 pCi/mL) for a 10-mL sample aliquot. Higher tritium concentrations can be measured by diluting or using smaller sample aliquots, or both. The maximum contaminant level for tritium in drinking water as given by the National Interim Primary Drinking Water Regulations (NIPDWR) is 0.740 Bq/mL (20000 pCi/L). The NIPDWR lists a required detection limit for tritium in drinking water of 0.037 Bq/mL (1 pCi/mL), meaning that drinking water supplies, where required, should be monitored for tritium at a sensitivity of 0.037 Bq/mL (1 pCi/mL). In Appendix X1, Eq X1.2 is given for determining the necessary counting time to meet the required sensitivity for drinking water monitoring.

ASTM D4129: Standard Test Method for Total and Organic Carbon in Water by High Temperature Oxidation and by Coulometric Detection

This test method covers the determination of total and organic carbon in water and wastewater, including brackish waters and brines in the range from 2 to 20000 mg/L. This test method has the advantages of a wide range of concentration which may be determined without sample dilution and the provision for boat or capillary introduction of samples containing sediments and particulate matter where syringe injection is inappropriate. This procedure is applicable only to that carbonaceous matter in the sample that can be introduced into the reaction zone. When syringe injection is used to introduce samples into the combustion zone, the syringe needle opening size limits the maximum size of particles that can be present in samples. Sludge and sediment samples must be homogenized prior to sampling with a micropipetor or other appropriate sampler and ladle introduction into the combustion zone is required.

ASTM D4130: Standard Test Method for Sulfate Ion in Brackish Water, Seawater, and Brines

This test method covers the turbidimetric determination of sulfate ion in brackish water, seawater, and brines. It has been used successfully with synthetic brine grade waters; however, it is the user's responsibility to ensure the validity of this test method to other matrices. This test method is applicable to waters having an ionic strength greater than 0.65 mol/L and a sulfate ion concentration greater than 25 mg/L. A concentration less than 25 mg/L sulfate can be determined by using a standard addition method. For brines having an ionic strength of less than 0.65 mol/L, refer to Test Methods D 516.

ASTM D4165: Standard Test Method for Cyanogen Chloride in Water

This test method covers the determination of cyanogen chloride in water. Cyanogen chloride is normally present only at very low concentrations; it is a very labile and sparsely soluble gaseous compound. Water samples may contain cyanogen chloride after the chlorination of wastewaters containing cyanide or thiocyanate compounds.

ASTM D4190: Standard Test Method for Elements in Water by Direct-Current Argon Plasma Atomic Emission Spectroscopy

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This test method covers the determination of dissolved and total recoverable elements in water, which includes drinking water, lake water, river water, seawater, snow, and Type II reagent water by direct current plasma atomic emission spectroscopy (DCP).

ASTM D4191: Standard Test Method for Sodium in Water by Atomic Absorption Spectrophotometry
This test method covers the determination of low amounts of sodium in waters having low solids content. The applicable range of this test method is from 0.20 to 3.0 mg/L when using the 589.6-nm resonance line. This range may be extended upward by dilution of an appropriate aliquot of sample or by use of the less-sensitive 330.2-nm resonance line (see Test Method D 3561). Many workers have found that this test method is reliable for sodium levels to 0.005 mg/L, but use of this test method at this low level is dependent on the configuration of the aspirator and nebulizer system available in the atomic absorption spectrophotometer as well as the experience and skill of the analyst. The precision and bias data presented are insufficient to justify use of this test method in the 0.005 to 0.20-mg/L range.

ASTM D4192: Standard Test Method for Potassium in Water by Atomic Absorption Spectrophotometry
This test method covers the determination of low amounts of potassium in waters having low solids content. The applicable range of this test method is 0.20 to 4.0 mg/L when using the 766.5-nm resonance line. The range may be extended upward by dilution of an appropriate aliquot of sample or by using the less-sensitive 404.4-nm resonance line. Many workers have found that this test method is reliable for potassium levels to 0.02 mg/L, but use of this test method at this low level is dependent on the configuration of the aspirator and nebulizer systems available in the atomic absorption spectrophotometer as well as the skill of the analyst. The precision and bias data presented are insufficient to justify use of this test method in the 0.02-mg/L range.

ASTM D4193: Standard Test Method for Thiocyanate in Water
This test method covers the determination of dissolved thiocyanate in water, wastewater, and saline water in the range from 0.1 to 2.0 mg/L. For higher concentrations, use an aliquot from the diluted sample.

ASTM D4282: Standard Test Method for Determination of Free Cyanide in Water and Wastewater by Microdiffusion
This test method covers the determination of free cyanides in waters and wastewaters. Free cyanide is here defined as the cyanide which diffuses as cyanide (HCN), at room temperature, from a solution at pH 6. This test method does not include complexes that resist dissociation, such as hexacyanoferrates and gold cyanide, nor does it include thiocyanate and cyanohydrin. This test method may be applied to water and wastewater samples containing free cyanide from 10 to 150 μg/L.

ASTM D4309: Standard Practice for Sample Digestion Using Closed Vessel Microwave Heating Technique for the Determination of Total Metals in Water
This practice covers the general considerations for quantitative sample digestion for total metals in water using closed vessel microwave heating technique. This practice is applicable to surface,

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saline, domestic, and industrial wastewater. Because of the differences among various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. This practice can be used with the following ASTM standards, providing the user determines precision and bias based on this digestion practice: Test Methods D 857, Test Methods D 858, Test Methods D 1068, Specification D 1192, Test Methods D 1687, Test Methods D 1688, Test Methods D 1691, Test Methods D 1886, Practices D 3370, Test Methods D 3557, Test Methods D 3559, Practice D 3919, Test Methods D 4190, Practice D 4453, and Practice D 4691.

ASTM D4327: Standard Test Method for Anions in Water by Chemically Suppressed Ion Chromatography

This test method covers the sequential determination of fluoride, chloride, nitrite, ortho-phosphate, bromide, nitrate, and sulfate ions in water by chemically suppressed ion chromatography. Note 1Order of elution is dependent upon the column used. This test method is applicable to drinking and wastewaters. The ranges tested for this test method for each anion were as follows (measured in mg/L):

	Fluoride	0.26 to 8.49
	Chloride	0.78 to 26.0
	Nitrite-N	0.36 to 12.0
	Bromide	0.63 to 21.0
	Nitrate-N	0.42 to 14.0
	o-Phosphate	0.69 to 23.1
	Sulfate	2.85 to 95.0

ASTM D4328: Standard Practice for Calculation of Supersaturation of Barium Sulfate, Strontium Sulfate, and Calcium Sulfate Dihydrate (Gypsum) in Brackish Water, Seawater, and Brines

This practice covers the calculation of supersaturation of barium sulfate, strontium sulfate, and calcium sulfate dihydrate (gypsum) in brackish water, seawater, and brines in which barium, strontium, and calcium ions either coexist or exist individually in solution in the presence of sulfate ions. This practice is not applicable for calculating calcium sulfate dihydrate supersaturation if the temperatures of saline waters under investigation exceed 95°C. At temperatures above 95°C, hemianhydrate and anhydrite would be major insoluble forms.

ASTM D4374: Standard Test Methods for Cyanides in Water-Automated Methods for Total Cyanide, Weak Acid Dissociable Cyanide, and Thiocyanate

These test methods cover the determination of different species of cyanides and thiocyanate in water and wastewater, namely weak acid dissociable cyanide, total cyanide, and thiocyanate.

This test method determines all the weak acid dissociable cyanides and the strong metal-cyano-complexes, such as ferrocyanide $[Fe(CN)6]^{4}$, ferricyanide $[Fe(CN)6]^{3}$, hexacyanocolbaltate $[Co(CN)6]^{3}$, and those of gold and platinum. This test method basically determines free cyanides, as CN and HCN, and weak metal-cyano-complexes such as $[Cd(CN)4]^{2}$ and $[Mn(CN)6]^{3}$. Iron complexes are not included. This test method determines the thiocyanate as

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the difference between another measurement that includes total cyanide plus thiocyanate and the value of total cyanide, that is, thiocyanate = total cyanide plus thiocyanate total cyanide.

ASTM D4382: Standard Test Method for Barium in Water, Atomic Absorption Spectrophotometry, Graphite Furnace
This test method covers the determination of dissolved and total recoverable barium in most waters and wastewaters.
This test method was evaluated in the range from 33.5 to 132 g/L of barium. The range can be increased or decreased by varying the volume of sample injected or the instrumental settings. High concentrations may be diluted but preferably should be analyzed by direct aspiration atomic absorption spectrophotometry.

ASTM D4411: Standard Guide for Sampling Fluvial Sediment in Motion
This guide covers the equipment and basic procedures for sampling to determine discharge of sediment transported by moving liquids. Equipment and procedures were originally developed to sample mineral sediments transported by rivers but they are applicable to sampling a variety of sediments transported in open channels or closed conduits. Procedures do not apply to sediments transported by flotation. This guide does not pertain directly to sampling to determine nondischarge-weighted concentrations, which in special instances are of interest. However, much of the descriptive information on sampler requirements and sediment transport phenomena is applicable in sampling for these concentrations, and 9.2.8 and 13.1.3 briefly specify suitable equipment. Additional information on this subject will be added in the future.

ASTM D4458: Standard Test Method for Chloride Ions in Brackish Water, Seawater, and Brines
This test method is applicable to the measurement of chloride in highly mineralized waters such as oil field brines, seawater, and brackish water. The test method is based upon the titration of chloride with silver nitrate, using a visual indicator. Samples containing from 10 to 150 mg of chloride can be analyzed by this test method. These levels are achieved by dilution as described in the test method.

ASTM D4517: Standard Test Method for Low-Level Total Silica in High-Purity Water by Flameless Atomic Absorption Spectroscopy
This test method covers the determination of total silica in water. This test method is applicable in the range from 25 to 250 [mu]g/L of silica as SiO_2. Higher concentrations may be determined by decreasing the aliquot volume. Concentration range should not be extended by dilution. This test method determines total silica, and does not distinguish between soluble and insoluble forms.

ASTM D4520: Standard Practice for Determining Water Injectivity Through the Use of On-Site Floods
This practice covers a procedure for conducting on-site core flood tests to determine the filtration and chemical treatment requirements for subsurface injection of water. This practice applies to water disposal, secondary recovery, and enhanced oil recovery projects and is applicable to injection waters with all ranges of total dissolved solids contents.

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ASTM D4581: Standard Guide for Measurement of Morphologic Characteristics of Surface Water Bodies

This guide covers the methods used for defining the morphologic characteristics of surface water bodies. This guide references manuals that provide various rationale and procedures necessary to conduct a morphologic survey. The references were written for specific agency use and may not be applicable in all cases (1-6)[2]. The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

ASTM D4638: Standard Guide for Preparation of Biological Samples for Inorganic Chemical Analysis

This guide describes procedures for the preparation of test samples collected from such locations as streams, rivers, ponds, lakes, estuaries, oceans, and toxicity tests and is applicable to such organisms as plankton, mollusks, fish, and plants. The procedures are applicable to the determination of volatile, semivolatile, and nonvolatile inorganic constituents of biological materials. Analyses may be carried out or reported on either a dry or wet basis.

ASTM D4658: Standard Test Method for Sulfide Ion in Water

This test method uses an ion-selective electrode to determine sulfide ion in water. The test method is applicable in the range from 0.04 to 4000 mg/L of sulfide.

ASTM D4691: Standard Practice for Measuring Elements in Water by Flame Atomic Absorption Spectrophotometry

This practice covers general considerations for the quantitative determination of elements in water and wastewater by flame atomic absorption spectrophotometry. Flame atomic absorption spectrophotometry is simple, rapid, and applicable to a large number of elements in drinking water, surface waters, and domestic and industrial wastes. While some waters may be analyzed directly, others will require pretreatment. Detection limits, sensitivity, and optimum ranges of the elements will vary with the various makes and models of satisfactory atomic absorption spectrometers. The actual concentration ranges measurable by direct aspiration are given in the specific test method for each element of interest. In the majority of instances the concentration range may be extended lower by use of electrothermal atomization and conversely extended upwards by using a less sensitive wavelength or rotating the burner head. Detection limits by direct aspiration may also be extended through sample concentration, solvent extraction techniques, or both. Where direct aspiration atomic absorption techniques do not provide adequate sensitivity, the analyst is referred to Practice D 3919 or specialized procedures such as the gaseous hydride method for arsenic (Test Methods D 2972) and selenium (Test Methods D 3859), and the cold vapor technique for mercury (Test Method D 3223). Because of the differences among various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead the analyst should follow the instructions provided by the manufacturer of a particular instrument.

ASTM D4698: Standard Practice for Total Digestion of Sediment Samples for Chemical Analysis of Various Metals

This practice covers two procedures for the total digestion of sediments for subsequent determination of metals by such techniques as flame atomic absorption spectrophotometry,

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graphite-furnace atomic absorption spectrophotometry, atomic emission spectroscopy, etc. This practice is applicable in the subsequent determination of volatile, semivolatile, and nonvolatile metals of sediments. Actual metal quantitation can be accomplished by following the various test methods outlined under other appropriate ASTM standards for the metal(s) of interest. Before selecting either of the digestion techniques outlined in this practice, the user should consult the appropriate quantitation standard(s) for any special analytical considerations, and Practice D 3976 for any special preparatory considerations.

ASTM D4743: Standard Test Method for Efficacy of Solvent Systems for Dissolving Water-Formed Deposits (Withdrawn 2003)
This test method covers the determination of the efficacy of solvent systems for dissolving either water-formed deposits that have been removed from the underlying material or synthetically prepared deposits.

ASTM D4763: Standard Practice for Identification of Chemicals in Water by Fluorescence Spectroscopy
This practice allows for the identification of 90 chemicals that may be found in water or in surface layers on water. This practice is based on the use of room-temperature fluorescence spectra taken from lists developed by the U.S. Environmental Protection Agency and the U.S. Coast Guard is the primary source for these spectra. This practice is also based on the assumption that such chemicals are either present in aqueous solution or are extracted from water into an appropriate solvent.

ASTM D4785: Standard Test Method for Low-Level Analysis of Iodine Radioisotopes in Water
This test method covers the quantification of low levels of radioactive iodine in water by means of chemical separation and counting with a high-resolution gamma ray detector. Iodine is chemically separated from a 4-L water sample using ion exchange and solvent extraction and is then precipitated as cuprous iodide for counting.

ASTM D4822: Standard Guide for Selection of Methods of Particle Size Analysis of Fluvial Sediments (Manual Methods)
This guide covers the selection of methods for determining the size distribution of fluvial sediments particles in the range greater than 0.45 μm using manual methods. Manual methods are defined as those methods that require the operator to do some actual measurements and calculations. An automated method would be one which, after the sample is prepared and inserted into an instrument, the instrument (machine) does the measuring and calculations, not the operator. Not all manual methods are presented in this guide. However, where available, at least two methods for each particle size range are given.

ASTM D4823: Standard Guide for Core Sampling Submerged, Unconsolidated Sediments
This guide covers core-sampling terminology, advantages and disadvantages of different types of core samplers, core-distortions that may occur during sampling, techniques for detecting and minimizing core distortions, and methods for dissecting and preserving sediment cores.

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ASTM D4839: Standard Test Method for Total Carbon and Organic Carbon in Water by Ultraviolet, or Persulfate Oxidation, or Both, and Infrared Detection
This test method covers the determination of total carbon (TC), inorganic carbon (IC), and total organic carbon (TOC) in water, wastewater, and seawater in the range from 0.1 mg/L to 4000 mg/L of carbon. This test method was used successfully with reagent water spiked with sodium carbonate, acetic acid, and pyridine. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices. This test method is applicable only to carbonaceous matter in the sample that can be introduced into the reaction zone. The syringe needle or injector opening size generally limit the maximum size of particles that can be so introduced.

ASTM D4922: Standard Test Method for Determination of Radioactive Iron in Water
This test method covers the determination of ^{55}Fe in the presence of ^{59}Fe by liquid scintillation counting. The a-priori minimum detectable concentration for this test method is E-3 Bq/mL.
This test method was developed principally for the quantitative determination of ^{55}Fe. However, after proper calibration of the liquid scintillation counter with reference standards of each nuclide, ^{59}Fe may also be quantified.

ASTM D4962: Standard Practice for NaI(Tl) Gamma-Ray Spectrometry of Water
This practice covers the measurement of radionuclides in water by means of gamma-ray spectrometry. It is applicable to nuclides emitting gamma-rays with energies greater than 50 keV. For typical counting systems and sample types, activity levels of about 40 Bq (1080 pCi) are easily measured and sensitivities of about 0.4 Bq (11 pCi) are found for many nuclides **(1-10).** Count rates in excess of 2000 counts per second should be avoided because of electronic limitations. High count rate samples can be accommodated by dilution or by increasing the sample to detector distance. This practice can be used for either quantitative or relative determinations. In tracer work, the results may be expressed by comparison with an initial concentration of a given nuclide which is taken as 100 %. For radioassay, the results may be expressed in terms of known nuclidic standards for the radionuclides known to be present. In addition to the quantitative measurement of gamma-ray activity, gamma-ray spectrometry can be used for the identification of specific gamma-ray emitters in a mixture of radionuclides.

ASTM D5072: Standard Test Method for Radon in Drinking Water (Withdrawn 2008)
This test method covers the measurement of radon in drinking water in concentrations above 0.04 Bq/L. This test method may be used for absolute measurements by calibrating with a radium-226 standard or for relative measurements by comparing the measurements made with each other.

ASTM D5073: Standard Practice for Depth Measurement of Surface Water
This is a general practice intended to give direction in the selection of depth measuring procedures and equipment for use under a wide range of conditions encountered in surface water bodies. Physical conditions at the measuring site, the quality of data required, and the availability of appropriate measuring equipment govern the selection process. A step-by-step procedure for actually obtaining a depth measurement is not discussed. This practice is to be used in conjunction with a practice on positioning techniques and another practice on bathymetric survey procedures to obtain horizontal location and bottom elevations of points on a water body.

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ASTM D5174: Standard Test Method for Trace Uranium in Water by Pulsed-Laser Phosphorimetry
This test method covers the determination of total uranium, by concentration, in water within the calibrated range of the instrument, 0.1 g/L or greater. Samples with uranium by mass, levels above the laser phosphorimeter dynamic range are diluted to bring the concentration to a measurable level.

ASTM D5176: Standard Test Method for Total Chemically Bound Nitrogen in Water by Pyrolysis and Chemiluminescence Detection
This test method covers the determination of the total nitrogen content of water in concentrations from 0.5 to 1000 mg/L. Higher nitrogen concentrations may be determined by making the proper dilutions. This test method does not determine molecular nitrogen (N_2).

ASTM D5256: Standard Test Method for Relative Efficacy of Dynamic Solvent Systems for Dissolving Water-Formed Deposits
This test method covers the determination of the relative efficacy of dynamic solvent systems for dissolving water-formed deposits that have been removed from the underlying material or deposits attached to the underlying material.

ASTM D5257: Standard Test Method for Dissolved Hexavalent Chromium in Water by Ion Chromatography
This test method covers procedures for the determination of dissolved hexavalent chromium in wastewater, surface water, and drinking water.

ASTM D5258: Standard Practice for Acid-Extraction of Elements from Sediments Using Closed Vessel Microwave Heating
This practice covers the digestion of soils and sediments for subsequent determination of acid-extractable concentrations of certain elements by such techniques as atomic absorption and atomic emission spectroscopy. Concentrations of arsenic, cadmium, copper, lead, magnesium, manganese, nickel, and zinc can be extracted from the preceding materials. Other elements may be determined using this practice.

ASTM D5413: Standard Test Methods for Measurement of Water Levels in Open-Water Bodies
These test methods cover equipment and procedures used in obtaining water levels of rivers, lakes, and reservoirs or other water bodies. Three types of equipment are available as follows:

Test Method A-Nonrecording water-level measurement devices
Test Method B-Recording water-level measurement devices
Test Method C-Remote-interrogation water-level measurement devices

The procedures detailed in these test methods are widely used by those responsible for investigations of streams, lakes, reservoirs, and estuaries, for example, the U.S. Agricultural Research Service, the U.S. Army Corp of Engineers, and the U.S. Geological Survey. The referenced ISO standard also furnishes useful information.

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ASTM D5462: Standard Test Method for On-Line Measurement of Low-Level Dissolved Oxygen in Water

This test method covers the on-line determination of dissolved oxygen (DO) in water samples primarily in ranges from 0 to 500 μg/L (ppb), although higher ranges may be used for calibration. On-line instrumentation is used for continuous measurements of DO in samples that are brought through sample lines and conditioned from high-temperature and high-pressure sources when necessary.

ASTM D5463: Standard Guide for Use of Test Kits to Measure Inorganic Constituents in Water

This guide covers general considerations for the use of test kits for quantitative determination of analytes in water and wastewater. Test kits are available from various manufacturers for the determination of a wide variety of analytes in drinking water, surface or groundwaters, domestic and industrial feedwaters and wastes, and water used in power generation and steam raising. See Table 1 for a listing of some of the types of kits that are available for various inorganic analytes in water. Ranges, detection limits, sensitivity, accuracy, and susceptibility to interferences vary from kit to kit, depending on the methodology selected by the manufacturer. In some cases, kits are designed to replicate exactly an official test method of a standard-setting organization such as the Association of Official Analytical Chemists (AOAC), American Public Health Association (APHA), ASTM, or the U.S. Environmental Protection Agency (USEPA). In other cases, minor modifications of official test methods are made for various reasons, such as to improve performance, operator convenience, or ease of use. Adjustments may be made to sample size, reagent volumes and concentrations, timing, and details of the analytical finish. In yet other cases, major changes may be made to the official test method, such as the omission of analytical steps, change of the analytical finish, omission of reagents, or substitution of one reagent for another. Reagents in test kits are often combined to obtain a fewer number and make the test easier to use. Additives may also be used to minimize interferences and to make the reagent more stable with time. A kit test method may be based on a completely different technology, not approved by any official or standard-setting organization. Combinations of test kits—multi-parameter test kits—may be packaged to satisfy the requirements of a particular application conveniently. The test kits in such combination products may be used to make dozens of determinations of several parameters.

ASTM D5613: Standard Test Method for Open-Channel Measurement of Time of Travel Using Dye Tracers

This test method covers the use of fluorescent dye tracers in streams to determine the rate that a solute moves along a streamline for a given river reach and the rate at which a solute disperses as it moves downstream. Accurate measurements of a stream's velocity and dispersion coefficient that can be determined by a tracer study are important parameters for water-quality models. Determined in advance to potential spilled or released noxious substances, velocity and dispersion rates are used to predict the time of arrival, passage time, and maximum concentration. Public health officials need this information to decide whether, when, and how long to suspend operations of public water-supply intakes in the reach downstream of a spill.

ASTM D5614: Standard Test Method for Open Channel Flow Measurement of Water with Broad-Crested Weirs

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This test method covers measurement of the volumetric flow rate of water in open channels with two types of horizontal broad-crested weirs: those having a square (sharp) upstream corner and those having a well-rounded upstream corner. The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

ASTM D5640: Standard Guide for Selection of Weirs and Flumes for Open-Channel Flow Measurement of Water

This guide covers recommendations for the selection of weirs and flumes for the measurement of the volumetric flow rate of water and wastewater in open channels under a variety of field conditions. This guide emphasizes the weirs and flumes for which ASTM standards are available, namely, thin-plate weirs, broad-crested weirs, Parshall flumes, and Palmer-Bowlus (and other long-throated) flumes. However, reference is also made to other measurement devices and methods that may be useful in specific situations.

ASTM D5673: Standard Test Method for Elements in Water by Inductively Coupled Plasma-Mass Spectrometry

This test method covers the determination of dissolved elements in groundwater, surface water, and drinking water. It may also be used for the determination of total-recoverable elements in these waters as well as wastewater.

ASTM D5674: Standard Guide for Operation of a Gaging Station

The guide covers procedures used commonly for the systematic collection of stream flow information. Continuous stream flow information is necessary for understanding the amount and variability of water for many uses, including water supply, waste dilution, irrigation, hydropower, and reservoir design. The procedures described in this guide are used widely by those responsible for the collection of stream flow data, for example, the U.S. Geological Survey, Bureau of Reclamation, U.S. Army Corps of Engineers, U.S. Department of Agriculture, Water Survey Canada, and many state and provincial agencies. The procedures are generally from internal documents of the preceding agencies, which have become the defacto standards used in North America.

ASTM D5811: Standard Test Method for Strontium-90 in Water

This test method covers the determination of radioactive ^{90}Sr in environmental water samples (for example, non-process and effluent waters) in the range of 0.037 Bq/L (1.0 pCi/L) or greater. The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only. This test method has been used successfully with tap water. It is the user's responsibility to ensure the validity of this test method for samples larger than 1 L and for waters of untested matrices.

ASTM D5904: Standard Test Method for Total Carbon, Inorganic Carbon, and Organic Carbon in Water by Ultraviolet, Persulfate Oxidation, and Membrane Conductivity Detection

This test method covers the determination of total carbon (TC), inorganic carbon (IC), and total organic carbon (TOC) in water in the range from 0.5 to 30 mg/L of carbon. Higher levels may be determined by sample dilution. The test method utilizes ultraviolet-persulfate oxidation of

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organic carbon, coupled with a CO2 selective membrane to recover the CO_2 into deionized water. The change in conductivity of the deionized water is measured and related to carbon concentration in the oxidized sample. Inorganic carbon is determined in a similar manner without the requirement for oxidation. In both cases, the sample is acidified to facilitate CO_2 recovery through the membrane. The relationship between the conductivity measurement and carbon concentration is described by a set of chemometric equations for the chemical equilibrium of CO_2, HCO_3^-, H^+, and the relationship between the ionic concentrations and the conductivity. The chemometric model includes the temperature dependence of the equilibrium constants and the specific conductances. This test method has the advantage of a very high sensitivity detector that allows very low detection levels on relatively small volumes of sample. Also, use of two measurement channels allows determination of CO_2 in the sample independently of organic carbon. Isolation of the conductivity detector from the sample by the CO_2 selective membrane results in a very stable calibration, with minimal interferences.

ASTM D5907: Standard Test Method for Filterable and Nonfilterable Matter in Water
This test method covers the determination of filterable and nonfilterable matter in drinking, surface, and saline waters, domestic and industrial wastes. The practical range of the determination of nonfilterable particulate matter is 4 to 20 000 mg/L. The practical range of the determination of filterable matter is 10 to 20 000 mg/L. Since the results measured by this test are operationally defined, careful attention must be paid to following the procedure as specified.

ASTM D6238: Standard Test Method for Total Oxygen Demand in Water
This test method covers the determination of total oxygen demand in the range from 100 to 100,000 mg/L, in water and wastewater including brackish waters and brines. Larger concentrations, or samples with high suspended solids, or both, may be determined by suitable dilution of the sample.

ASTM D6239: Standard Test Method for Uranium in Drinking Water by High-Resolution Alpha-Liquid-Scintillation Spectrometry
This test method covers determining the total soluble uranium activity in drinking water in the range of 0.037 Bq/L (1 pCi/L) or greater by selective solvent extraction and high-resolution alpha-liquid-scintillation spectrometry. The energy resolution obtainable with this technique also allows estimation of the ^{238}U to ^{234}U activity ratio.

ASTM D6501: Standard Test Method for Phosphonate in Brines
This test method covers the colorimetric determination of phosphonate (PNA) in brines from gas and oil production operations in the range from 0.1 to 5 mg/L. This phosphonate method is intended for use to analyze low concentration of phosphonate in brine containing interfering elements. This test method is most useful for analyzing phosphonate at 0.1 to 1 mg/L range in brines with interfering elements; however, it requires personnel with good analytical skill.

ASTM D6508: Standard Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte
This test method cover the determination of the inorganic anions fluoride, bromide, chloride, nitrite, nitrate, ortho-phosphate, and sulfate in drinking water, wastewater, and other aqueous matrices using capillary ion electrophoresis (CIE) with indirect UV detection. The test method

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uses a chromate-based electrolyte and indirect UV detection at 254 nm. It is applicable for the determination or inorganic anions in the range of 0.1 to 50 mg/L except for fluoride whose range is 0.1 to 25 mg/L.

ASTM D6581: Standard Test Methods for Bromate, Bromide, Chlorate, and Chlorite in Drinking Water by Suppressed Ion Chromatography
This multi-test method covers the determination of the oxyhalides—chlorite, bromate, and chlorate, and bromide, in raw water, finished drinking water and bottled (non-carbonated) water by chemically and electrolytically suppressed ion chromatography.

ASTM D6696: Standard Guide for Understanding Cyanide Species
This guide defines standard terminology used for the classification of the various chemical forms of cyanide. It is intended to provide a general understanding of the chemical nature of distinct cyanide species as related to chemical analysis and environmental fate and transport.

ASTM D6697: Standard Test Method for Determination for Chemical Oxygen Demand (Manganese III Oxygen Demand) of Water
This test method covers the colorimetric determination of the quantity of oxygen that certain impurities in water will consume, based on the reduction of a manganese III solution under specified conditions. This standard method does not use characteristic heavy metal reagents, thus eliminating environmental and disposal concerns apparent in other methods. This test method determines chemical oxygen demand colorimetrically using manganese III to obtain a visible color intensity inversely proportional to the chemical oxygen demand of the sample. Analytical test kits conforming to these methods are available commercially in ranges from 80 to 1,000 mg/L (ppm) chemical oxygen demand. It is the user's responsibility to ensure the validity of these test methods for their specific samples and matrices.

ASTM D6800: Standard Practice for Preparation of Water Samples Using Reductive Precipitation Preconcentration Technique for ICP-MS Analysis of Trace Metals
Toxic elements may be present in ambient waters and may enter the food chain via uptake by plants and animals; the actual concentrations of toxic metals are usually sub-ng/mL. The U.S. EPA has published its Water Quality Standards in the U.S. Federal Register 40 CFR 131.36, Minimum requirements for water quality standards submission, Ch. I (7-1-00 Edition). The U.S. EPA has also developed Method 1640 to meet these requirements. Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) is a technique with sufficient sensitivity to routinely measure toxic elements in ambient waters, both fresh and saline (Test Method D 5673). However saline and hard water matrices pose analytical challenges for direct multi-element analysis by ICP-MS at the required sub-ng/mL levels.

ASTM D6850: Standard Guide for QC of Screening Methods in Water
This guide covers general considerations for the Quality Control practices for use with screening methods for organic and inorganic constituents in water. Methods are provided by various standard setting bodies, governmental agencies, as well as many domestic and international manufacturers. This guide provides general QC procedures that are applicable to a broad range of screening methodologies. These procedures help to ensure the quality of data that is generated.

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Additional, method-specific or project specific requirements may be necessary. This guide also includes general considerations regarding proper utilization of screening methods.

ASTM D6888: Standard Test Method for Available Cyanide with Ligand Displacement and Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection

This method is used to determine the concentration of available inorganic cyanide in an aqueous wastewater or effluent. The method detects the cyanides that are free (HCN and CN^-) and metal-cyanide complexes that are easily dissociated into free cyanide ions. The method does not detect the less toxic strong metal-cyanide complexes, cyanides that are not "amenable to chlorination." Total cyanide can be determined for samples that have been distilled as described in Test Methods D 2036, Test Method A, Total Cyanides after Distillation. The cyanide complexes are dissociated and absorbed into the sodium hydroxide capture solution, which can be analyzed with this test method. This procedure is applicable over a range of approximately 2 to 400 µg/L (parts per billion) available cyanide. Higher concentrations can be analyzed by dilution or lower injection volume.

ASTM D6919: Standard Test Method for Determination of Dissolved Alkali and Alkaline Earth Cations and Ammonium in Water and Wastewater by Ion Chromatography

This test method is valid for the simultaneous determination of the inorganic alkali and alkaline earth cations, lithium, sodium, potassium, magnesium, and calcium, as well as the ammonium cation in reagent water, drinking water, and wastewaters by suppressed and nonsuppressed ion chromatography. The anticipated range of the method is 0.05–200 mg/L. The specific concentration ranges tested for this method for each cation were as follows (measured in mg/L):

Lithium	0.4–10.0
Sodium	4.0–40.0
Ammonium	0.4–10.0
Potassium	1.2–20.0
Magnesium	2.4–20.0
Calcium	4.0–40.0

The upper limits may be extended by appropriate dilution or by the use of a smaller injection volume. In some cases, using a larger injection loop may extend the lower limits.

ASTM D6994: Standard Test Method for Determination of Metal Cyanide Complexes in Wastewater, Surface Water, Groundwater and Drinking Water Using Anion Exchange Chromatography with UV Detection

This test method covers the determination of the metal cyanide complexes of iron, cobalt, silver, gold, copper and nickel in waters including groundwaters, surface waters, drinking waters and wastewaters by anion exchange chromatography and UV detection. The use of alkaline sample preservation conditions (see 10.3) ensures that all metal cyanide complexes are solubilized and recovered in the analysis (1-3). Metal cyanide complex concentrations between 0.20 to 200 mg/L may be determined by direct injection of the sample. This range will differ depending on the specific metal cyanide complex analyte, with some exhibiting greater or lesser detection sensitivity than others. Approximate concentration ranges are provided in 12.1. Concentrations greater than the specific analyte range can be determined after appropriate dilution. This test

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method is not applicable for matrices with high ionic strength (conductivity greater than 500 meq/L as Cl) and TDS (greater than 30 000 mg/L), such as ocean water. Metal cyanide complex concentrations less than 0.200 mg/L may be determined by on-line sample preconcentration coupled with anion exchange chromatography as described in 11.3. This range will differ depending on the specific metal cyanide complex analyte, with some exhibiting greater or lesser detection sensitivity than others. The preconcentration method is not applicable for silver and copper cyanide complexes in matrices with high TDS (greater than 1000 mg/L).

ASTM D7168: Standard Test Method for ^{99}Tc in Water by Solid Phase Extraction Disk
This test method describes a solid phase extraction (SPE) procedure to separate ^{99}Tc from environmental water samples (that is, non-process and effluent waters). Technetium-99 beta activity is measured by liquid scintillation spectrometry. This test method is designed to measure ^{99}Tc in the range of approximately 0.037 Bq/L (1.0 pCi/L) or greater for a one liter sample.

ASTM D7237: Standard Test Method for Free Cyanide with Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection
This test method is used to establish the concentration of free cyanide in an aqueous wastewater or effluent. The test conditions of this method are used to measure free cyanide (HCN and CN^-) and cyanide bound in the metal-cyanide complexes that are easily dissociated into free cyanide ions at the pH of 6. Free cyanide is determined at pH 6 at room temperature. The aquatic free cyanide can be determined by matching the pH to the water in the receiving environment in the range of pH 6 to 8. The extent of HCN formation is less dependent on temperature than the pH; however, the temperature can be regulated if deemed necessary for aquatic free cyanide to further simulate the actual aquatic environment. The free cyanide method is based on the same instrumentation and technology that is described in standard test method D6888, but employs milder conditions (pH 6-8 buffer versus HCl or H_2SO_4 in the reagent stream), and does not utilize ligand displacement reagents.

ASTM D7282: Standard Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements
This practice covers consensus criteria for the calibration and quality control of nuclear instruments. This practice is provided for establishing appropriate quality control parameters at instrument startup, calibration of nuclear counting instruments and the continuing monitoring of quality control parameters. Calibrations are usually performed to establish the operating parameters of the instrument. This practice addresses the typically used nuclear counting instruments: alpha spectrometer, gamma spectrometer, gas proportional counter and liquid scintillation counter.

ASTM D7283: Standard Test Method for Alpha- And Beta- Activity in Water by Liquid Scintillation Counting
This test method covers the measurement of gross alpha- and beta- activity concentrations in a homogeneous water sample. It is applicable to alpha emitters with activity concentration levels above 0.037 Bq/L (1 pCi/L) and beta emitters with activity concentration levels above 0.15 Bq/L (4 pCi/L). This test method is not applicable to samples containing radionuclides that are volatile under conditions of the analysis. This test method may also be used for the direct measurement of gross alpha- and beta- activity concentrations in homogeneous water samples with alpha

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emitter activity concentration levels above 1.8 Bq/L (50 pCi/L) and beta emitter activity concentration levels above 3.7 Bq/L (100 pCi/L). This test method was tested using a single-operator test. Round-robin testing is currently in progress on this test method.

ASTM D7284: Standard Test Method for Total Cyanide in Water by Micro Distillation followed by Flow Injection Analysis with Gas Diffusion Separation and Amperometric Detection

This test method is used to determine the concentration of total cyanide in an aqueous wastewater or effluent. The method detects the cyanides that are free (HCN and CN^-) and strong-metal-cyanide complexes that dissociate and release free cyanide when refluxed under strongly acidic conditions. This method may not be applicable to process solutions from precious metals mining operations. This procedure is applicable over a range of approximately 2 to 400 µg/L (parts per billion) total cyanide. Higher concentrations can be measured with sample dilution or lower injection volume.

ASTM D7365: Standard Practice for Sampling, Preservation and Mitigating Interferences in Water Samples for Analysis of Cyanide

This practice is applicable for the collection and preservation of water samples for the analysis of cyanide. This practice addresses the mitigation of known interferences prior to the analysis of cyanide. Responsibilities of field sampling personnel and the laboratory are indicated. The sampling, preservation and mitigation of interference procedures described in this practice are recommended for the analysis of total cyanide, available cyanide, weak acid dissociable cyanide, and free cyanide by Test Methods D 2036, D 4282, D 4374, D 6888, D 6994, D 7237, D 7284, and D 7511. The information supplied in this practice can also be applied to other analytical methods for cyanide, for example, EPA Method 335.4.

ASTM D7511: Standard Test Method for Total Cyanide by Segmented Flow Injection Analysis, In-Line Ultraviolet Digestion and Amperometric Detection

This method is used for determining total cyanide in drinking and surface waters, as well as domestic and industrial wastes. Cyanide ion (CN^-), hydrogen cyanide in water (HCN(aq)), and the cyano-complexes of zinc, copper, cadmium, mercury, nickel, silver, and iron may be determined by this method. Cyanide ions from Au(I), Co(III), Pd(II), and Ru(II) complexes are only partially determined. The method detection limit (MDL) is 1.0 µg/L cyanide and the minimum level (ML) is 3 µg/L. The applicable range of the method is 3 to 500 µg/L cyanide using a 200-µL sample loop. Extend the range to analyze higher concentrations by sample dilution or changing the sample loop volume. This method can be used by analysts experienced with equipment using segmented flow analysis (SFA) and flow injection analysis (FIA) or working under the close supervision of such qualified persons.

ASTM D7535: Standard Test Method for Lead-210 in Water

This test method covers the determination of radioactive ^{210}Pb in environmental water samples (for example, drinking, non-process and effluent waters) in the range of 37 mBq/L (1.0 pCi/L) or greater. The values stated in SI units are to be regarded as standard. The values given in parentheses are provided for information purposes only. This method has been used successfully with tap water. It is the user's responsibility to ensure the validity of this test method for samples larger than 500 mL and for waters of untested matrices.

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ASTM D7572: Standard Guide for Recovery of Aqueous Cyanides by Extraction from Mine Rock and Soil after Remediation of Process Releases

This guide is applicable for the collection, extraction and preservation of extracts from mine rock and soil samples after remediation of cyanide process releases for the analysis of cyanide in the extracts. Responsibilities of field sampling personnel and the laboratory are indicated. The sampling, preservation and extraction procedures described in this practice are recommended for the analysis of total cyanide, available cyanide, weak acid dissociable cyanide, and free cyanide by Test Methods D 2036, D 4282, D 4374, D 6888, D 6994, D 7237, and D 7284. The information supplied in this practice can also be applied to other analytical methods for cyanide, for example, US EPA Method 335.4.

ASTM D7573: Standard Test Method for Total Carbon and Organic Carbon in Water by High Temperature Catalytic Combustion and Infrared Detection

This test method covers the determination of total carbon (TC), inorganic carbon (IC), total organic carbon (TOC), dissolved organic carbon (DOC), and non-purgable organic carbon (NPOC) in water, wastewater, and seawater in the range from 0.5 mg/L to 4000 mg/L of carbon. Higher levels may be determined by sample dilution. The sample is injected onto a quartz bed heated at 680ºC. The sample converts into a gaseous phase and forced through a layer of catalyst ensuring conversion of all carbon containing compounds to CO_2. A non-dispersive infrared (NDIR) detector measures the resulting CO_2. For TOC and DOC analysis a portion of the sample is injected to determine TC or dissolved carbon (DC). A portion of the sample is then acidified and purged to remove the IC. The purged inorganic carbon is measured as TIC, or DIC. TOC or DOC is calculated by subtracting the inorganic fraction from the total carbon. TOC = TC – IC.

For NPOC analysis a portion of sample is acidified and purged to remove IC. The purged sample is then injected to determine NPOC. This test method was used successfully with reagent water spiked with potassium hydrogen phthalate, sucrose, nicotinic acid, benzoquinone, sodium dodecyl benzene sulfonate, urea, acetic acid, and humic acid. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

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APPENDIX 3—SUMMARY OF FIELD SAMPLING AND ANALYTICAL METHODS WITH BIBLIOGRAPHY

Compiled by Virginia T. McLemore, Kathleen S. Smith, and Carol C. Russell

Reviewed by Mary P. Goldade, Thomas R. Wildeman, and Bradley S. Van Gosen

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DISCLAIMER:

This appendix is an overview of field measurements, sample collection, preparation methods, analytical methods, and geophysical and remote sensing methods. It is intended to give a brief summary of the technique and its uses, limitations, and specific references. Not all methods are included. This appendix is not intended to provide all of the information needed to conduct the specific method, but instead to provide enough information for the reader to understand the specifics of the technique and to compare with results obtained using other appropriate techniques. Please note that the method descriptions do not purport to address safety concerns, if any, associated with the method. It is the responsibility of the user of the method to establish appropriate safety and health practices, to perform analysis using good laboratory practices, and to determine the applicability of regulatory limitations prior to use. To the extent the document mentions or discusses statutory or regulatory authorities, it does so for informational purposes only. This document does not substitute for statutes or regulations, and readers should consult the statutes or regulations themselves to learn what they require. Please also note that regulatory requirements may be more specific than those implied in these method summaries. Any use of trade, firm, or product names is for descriptive purposes only and does not imply endorsement by the U.S. Government. The views expressed in this appendix are those of the authors and editors and do not necessarily represent the views of the U.S. Environmental Protection Agency or of the United States. This appendix has been peer reviewed and approved for publication consistent with U.S. Geological Survey Fundamental Science Practices (http://pubs.usgs.gov/circ/1367, 9/17/13).

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GEOLOGICAL AND LOCATIONAL MEASUREMENTS AND DATA

The purpose of this section is to provide a brief summary of the field observations that the field scientist, especially the exploration geologist, should consider making during any sampling and monitoring activity, especially during the exploration and feasibility stages of the mine.

Global Positioning System (GPS) Georeferenced Data Acquisition

(James A. Russell)

MEDIA: Locational Data

MEASUREMENT OBJECTIVES: The determination of precise locations of sampling sites enhances the quality of the subsequent analyses of data collected in geochemical studies. Data sites and observations can now be as accurate as less than one foot allowing for accurate follow-up studies to retrace earlier work by field crews.

SPECIFIC SAMPLING EQUIPMENT: Global Positioning Systems used in scientific studies rely upon ruggedized professional-grade equipment. This equipment can record not only locational data, but also associated metadata that allows for either real-time corrected positioning or for later post processing to improve positional accuracy of the sample site. Other GPS hardware can also be used to communicate with GPS receivers for improved accuracy.

ANALYTICAL TECHNIQUES: Global Positioning Systems rely upon a constellation of Earth orbiting satellites to locate points on the Earth. The main system in use today is the NAVSTAR (Navigation Satellite Timing and Ranging) Global Positioning System operated by the 50th Space Wing of the United States Air Force. A similar system, GLONASS, (Globalnaya navigatsionnaya sputnikovaya sistema) is operated by the Russian Space Forces for Russia. GPS receivers use signals transmitted by these satellites to triangulate positions on the Earth. Commercial-grade hardware is usually ruggedized with extended life battery life. Accuracy ranges from 10 meters to 10 cm, depending upon equipment and software. Positional data can be recorded for later post processing to improve accuracy. Real time differential correction of data is possible with accuracy in the sub-foot range (<30 cm). Real time differential correction requires the use of portable beacons to improve reception of satellite signals. Decimal accuracy (10 cm) can be achieved with further post processing of real-time differentially corrected data. Software, either off the shelf or custom, purpose-built is used to add attribute data to the positional data for use in later analytical studies.

LIMITATIONS/INTERFERENCES: Positional accuracy is a function of several factors: (1) satellite geometry (position) above the sampling site, (2) multipath signals, (3) extent of visible sky, and (4) canopy cover. Positional accuracy improves when there is a greater spatial distribution of the satellites in the sky. Satellites concentrated in one area of the sky can result in poor quality locational information. Multipath signals are signals that are reflected of ground terrain or refracted by the atmosphere. These competing signals result in inaccurate signals received by the GPS receiver. If the visible sky is restricted by valleys, canyons or mountains, signals from GPS satellites will be

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restricted resulting in no useable signal or poor accuracy. Vegetation with high water content also degrades signal strength. This can result in poor signal quality in areas with a dense vegetative canopy.

DOCUMENTATION/DATA HANDLING: Recorded GPS data should be downloaded from the GPS receiver and saved for further analysis onto digital media or workstations. Care should be taken to do this promptly after a field session if data are stored in volatile memory and may be lost if the unit's battery is discharged. The choice of coordinate system and datum for the GPS should be determined prior to data collection. State plane coordinate systems have traditionally been used by County governments for accuracy purposes whereas coordinate systems such as Universal Transverse Mercator are used for regional or statewide scale data collection efforts.

BIBLIOGRAPHY:

Kaplan, E.D., ed., 1996, Understanding GPS Principles and Applications: Artech House Inc., Norwood.

Kaplan, E.D. and Hegarty, C.J., ed., 2005, Understanding GPS Principles and Applications: Artech House Inc., Norwood, 16 p., http://www.google.com/url?sa=t&rct=j&q=&esrc=s&source=web&cd=2&ved=0CDwQFjAB&url=http%3A%2F%2Fwww.sae.org%2Fservlets%2Fbookstore%2FdownloadMyItem.do%3Fmethod%3DdownloadMyItem%26prodCd%3DB-ART-010%26pubType%3Dapub%26fm%3D1%26bm%3D0&ei=UYI4UviNA8OKjAKb_oGgDQ&usg=AFQjCNHB-IqH-ENIcDoTRpRKuw2xtp2sbw&sig2=ERpwQcXx0sNMPf8pYxA-iw&bvm=bv.52164340,d.cGE, accessed 9/17/13.

Thurston, J., Poiker, T.K., and Moore J.P., 2003, Integrated Geospatial Technologies: A Guide to GPS,GIS, and Data Logging: John Wiley, Hoboken.

Van Sickle, J., 2001, GPS for Land Surveyors: Taylor and Francis, London, New York.

Geographic Information System (GIS)

(James A. Russell)

MEDIA: Spatially Enabled Data

MEASUREMENT OBJECTIVES: A geographic information system (GIS) allows analysts to view, understand, question, interpret, and visualize data in many ways that reveal relationships, patterns, and trends in the form of maps, globes, reports, and charts. Measurements of geographic enabled data are used to evaluate the impact of mining-related activities on the environment and humans and animals that are directly or indirectly impacted by these disturbances. Spatially enabled data allow analysts to:

- Map where things are, map quantities and densities of chemical contamination,
- Determine what is inside defined spatial boundaries,
- Map what is nearby defined spatial boundaries, and
- Map temporal changes in the study area.

SPECIFIC FIELD SAMPLING TECHNIQUES/EQUIPMENT: Sampling and recording techniques that can be used in the field include customizable field data

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collection software in conjunction with Geographic Positioning System hardware and software.

1. *Field data collection hardware*
   Computer hardware that can be used with GIS software to collect field data can be either
   - Windows-based laptops, tablet workstations, Windows Mobile devices and mobile phones,
   - Apple IOS devices such as Ipad tablets, Iphones, and
   - Android operating systems devices such as mobile phones and tablets.

   GPS integration with this hardware varies with the hardware configuration.
2. *Field data collection software*
   Computer software that can be used with the hardware mentioned above include easily customized applications based upon office GIS applications or more sophisticated applications that use Application Programming Interface (API) software development kits targeted at specific hardware. The complexity and functionality of field applications depends upon the computer programming capabilities of the organization and the time allocated to software modification and customization.

SPATIAL OR TEMPORAL CONSIDERATIONS:

Data collection can either be synchronous or asynchronous, that is, either connected into a network through wireless technologies while data collection occurs, or not connected to a network requiring data downloads when returning to the office at the end of the day or when a wireless network is in range.

Data should be stored in a secure manner on the hardware during field collection in either case. The real time transfer of data through wireless connections allows for analysis of the data in the office by others as the field data collection proceeds.

LABORATORY TECHNIQUES:

Data collection efforts should consider the needs of GIS data formatting at the outset of the project. Database design is important in efficient analysis with a GIS. Other considerations to consider in new field collection efforts include the geospatial characteristics of already acquired data. This includes spatial projection and datums, accuracy standards for associated with GPS hardware in past and present collection efforts (differentially corrected or not), and different types of software and hardware. Field work can be tough on equipment. Hardware should meet MIL-Spec (military specification) standards (MIL-STD_810G) for durability if possible.

ADDITIONAL CONSIDERATIONS:

Serious thought should be given to proper field crew training for both the hardware and software. Field data collection software should be target the specific data collection effort in mind. This is not difficult with the most current versions of mobile software. Also be aware of the possibility of being disconnected from wireless networks that require synchronizing data at infrequent intervals throughout the data collection project. Furthermore, difficult terrain conditions may interfere with good GPS signal reception.

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BIBLIOGRAPHY

Arctur, D., Zeiler, M., 2004. Designing Geodatabases Case Studies in GIS Modeling: Redlands, ESRI, 408 p.

ESRI, 2011, GIS: Better decisions through mapping and modeling our world [online]: Available from: http://www.gis.com/, accessed 8/23/13.

ESRI, 2011, ARCGIS Resource Center Mobile GIS [online]: http://resources.arcgis.com/content/mobilegis/about, accessed 8/23/13.

ESRI, 2007, Understanding Coordinate Management in a Geodatabase: Available from: http://downloads2.esri.com/support/whitepapers/ao_/UnderstandingCoordinateManagement_June2007.pdf, accessed 8/23/13.

Cultural Resource Surveys

(Karl Laumbach)

MEASUREMENT OBJECTIVE: Any project that includes plans for disturbing ground on publicly owned land, laws and associated regulations require the evaluation of cultural resources. All artifacts and features that are potentially older than 50 years must be recorded and considered in the cultural resource survey. Project personnel should contact the respective State Historic Preservation Division and the state or federal agency that manages land within or near the proposed project to determine whether a cultural resources survey is required. If required, the project must engage qualified personnel who are permitted by the managing agency to perform cultural resource surveys. If standing historic structures are present, a permitted architectural historian may be required for the recording of the survey. For most mining and processing sites it is highly recommended to conduct a cultural resource survey, even if not required by regulation, to avoid potential public-relation problems in the future.

MEDIUM/PARAMETERS: A cultural resource survey involves a close order (usually 15 meter transect) pedestrian survey of the project area as defined by the area of probable effect (APE). Archaeological sites consist of clusters of artifacts and/or features. Sites are recorded on standardized forms in tandem with the preparation of a site plan map, photographs, and locational data (usually UTM coordinates that will be plotted on a USGS topographic map). Isolated artifacts and features may be recorded as isolates. Isolates are described, photographed, if appropriate, and locational data are recorded. If standing buildings are present, an architectural historian, historic architect or archaeologist who is permitted to record buildings will be required to record the standing architecture.

MEASUREMENT OBJECTIVES: Data acquired from the archaeological/architectural survey will be evaluated by the contracting cultural resource specialist to determine the significance of the sites and buildings. Significance depends on a number of factors that include integrity, temporal and historical associations, and potential to yield additional data important to history. The recorder's evaluation of significance and recommendations concerning appropriate action by the project will be included in the cultural resource report. The report will then be evaluated by the involved land managing agency and/or the State Historic Preservation Office. Those agencies will make the final determinations

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of significance and mandate appropriate action to either avoid or mitigate impacts to significant cultural resources within the project area.

SPECIFIC SAMPLE EQUIPMENT/TECHNIQUES: Except in unusual circumstances, artifacts are not collected during a cultural resources survey. If artifacts are collected, the location is recorded and artifacts are bagged with appropriate labeling relating to project and location. Recording of artifacts in the field depends on artifact densities. If the artifact densities are low, all visible artifacts may be recorded. If densities are high, a sample of the artifacts may be recorded. Typical sampling techniques include transects or gridded units placed so that the observed site variability is reflected in the samples.

ANALYTICAL TECHNIQUES: Field analysis of artifacts and archeological features is dependent upon the nature of the artifacts and features. Typically, prehistoric sites may contain flaked stone, ground stone and ceramic artifacts. Historic sites may include a wide variety of glass, metal, wood, and plastic artifacts related to a wide range of functions. Each artifact category has unique attributes that can be monitored during the analysis. In field analysis is focused on obtaining data that can be used to categorize the artifact assemblage and to aid in the determination of the site's temporality, cultural affiliation, and function. These data will be used to aid in the determination of significance.

LIMITATIONS/INTERFERENCES: Depending on the terrain, geomorphology and vegetation, sites will be more or less visible from the surface. If any of these or other factors interfere with the evaluation of an archaeological site during survey, subsurface testing may be required. Depending on the regulations of the oversight agency, limited testing may be allowed during the initial survey. If the regulations do not allow limited testing during survey or if limited testing will not suffice to adequately document the site, then a more formal testing program will be recommended in the survey report and implemented after review. Formal testing usually requires a separate approval process and/or separate permit.

DOCUMENTATION/DATA HANDLING: As most, if not all, of the survey data are collected in the field, the digitized or paper database will be filed with the cultural resource contractor and will be included in the deliverables to the involved agencies. Each state has a clearing house/storage facility for reports and data.

ADDITIONAL CONSIDERATIONS: If the outcome of the survey requires avoidance or mitigation of significant cultural resources, the project directors will have to evaluate these requirements in relation to the goals of the project. With proper planning, avoidance is often a viable mitigation option; however if avoidance is not feasible, a data recovery plan to mitigate the project impacts must be written, submitted, approved by the permitting agencies, and then satisfactorily completed prior to implementation of the project.

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BIBLIOGRAPHY:

http://www.srifoundation.org/consulting.html, accessed 8/23/13.
http://www.srifoundation.org/pdf/SAACURRI.PDF, accessed 8/23/13.

Photography

(Virginia T. McLemore)

MEASUREMENT: Photographs provide a visual record of site conditions, either prior to, during, and/or following site activities. Among other things, photographs may be used to document sample sites, test pits, trenches or long walls, drill core, lithology, mineralogy, stratigraphy, and internal structure of the rock piles and tailings at field scale as well as microscopic (thin section, electron microprobe). Sampling and monitoring activities can be photographed.

ANALYTICAL METHOD: It is recommended that each photograph include the sample number or some other identification in the field of view when appropriate. Each photograph should include a standardized tool (e.g., ruler) to document scale. Each photograph should have a GPS location. Documentation of the photographs acquired in the field and laboratory will provide information on the photograph and also provide a permanent record of field activities.

LIMITATIONS/INTERFERENCES: Taking photographs with digital cameras or cameras with film can be used. Digital photographs and scans should be taken at high resolution so that publication of the photograph will be possible. Hard copy photography (e.g., slides and prints) should be digitally scanned in order to be stored electronically.

DOCUMENTATION/DATA HANDLING: Maintain a photo log for all photographs taken. At a minimum, each photograph shall contain the following information:

- Photograph number
- Date
- Photographer's initials
- Field identification or station number
- Camera type and number of pixels

ADDITIONAL CONSIDERATIONS: Hard copies of the photographs are recommended.

BIBLIOGRAPHY:

http://www.epa.gov/compliance/resources/policies/monitoring/digitalcameraguide.pdf
(accessed 8/23/13)

Topography and Map Projections

(Virginia T. McLemore)

MEASUREMENT: Topography is the study of the Earth's surface, shape, relief, and arrangement of features (both manmade and natural). Topography is generally studied in two or three dimensions and is measured:

- directly using field surveying techniques

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- by remote sensing techniques
- by aerial and satellite photography (http://edc2.usgs.gov/pubslists/booklets/aerial/aerial.php)
- by photogrammetry(Geodetic Services, Inc., 2011)
- by radar and sonar.

Generally, the topography is displayed as a contour topographic map and as topographic cross section profiles. Contours are imaginary lines that join points of equal elevation on the surface of the land and make it possible to show elevations on a two dimensional planar map. From the contours, the user is able to determine the elevation of most features. Topographic maps can show mountains, valleys, plains, lakes, rivers, vegetation, roads, boundaries, transmission lines, major buildings, among other features. The U.S. Geological Survey has standardized map symbols showing these features (http://egsc.usgs.gov/isb/pubs/booklets/symbols/).

MEDIUM/PARAMETERS:

Map scale. The map scale is located on the map and varies according to the size of the physical map and accuracy of location of map features. The map scale is the relationship between the distance on the map and the distance on the ground and is generally given as a ratio (1:10,000). This ratio means that 1 unit of measure on the map (1 inch or 1 centimeter) represents 10,000 of the same measure on the ground, or 10,000 inches or centimeters on the ground. The larger the second number, the smaller the scale of the map (U.S. Geological Survey, 2002). Bar scales also can be displayed. In the United States, the U.S. Geological Survey publishes topographic maps for the entire country at three major scales, 1:24,000 quadrangle maps, 1:100,000 maps, and 1:250,000 scale maps; other scales are available.

Elevation or altitude: The elevation of a geographic feature is the height above a fixed reference point, generally called a geoid or mathematical model of the earth's surface. Elevation typically is used to refer to the geometric height above the earth's surface, whereas altitude is used for points above the surface (such as aircraft in flight). Depth generally is used for points below the earth's surface.

Slope aspects: Slope aspects refer to the direction in which a mountain slope faces and are important on local climate conditions. In most areas, the afternoon or westerly sun is warmer and therefore the west facing slopes can be warmer. Slope aspects can be obtained from topographic maps. Aspect-slope maps also can be created that shows the aspect (direction) and degree (steepness) of the slopes (Nyssen and Vermeersch, 2010; http://blogs.esri.com/Support/blogs/mappingcenter/archive/2008/05/22/aspect-slope-map.aspx, accessed 8/23/13).

Degree of steepness: The degree of steepness of the slope also is important in understanding water flow and avalanches. Generally, the degree of steepness is measured in percentage.

Map projections: A map projection is used to represent the irregular but round surface of the earth on a flat map. A description of various map projections is on the U.S.

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Geological Survey website (http://pubs.usgs.gov/pp/1453/report.pdf, accessed 8/23/13). There is no best map projection; each projection has advantages and disadvantages, but it is critical that the map projection used on the maps, in surveying, and using GPS be identified.

Digital elevation modeling or DEM: Digital elevation modeling or DEM is a raster-based digital representation of the topography generated from the above listed measurements of topography (U.S. Geological Survey, 2000). DEMs are generally regularly spaced grids of elevation points.

MEASUREMENT OBJECTIVES: Topographic features affect climate, erosion, water flow (McGuire et al., 2005), among others, which can affect the success or failure of a reclamation plan (Alday et al., 2010). Topographic maps are important, because they show the location and spatial arrangement of the surroundings, such as the various components of a mine site and their spatial relationship to other features, such as landmarks, rivers, cities, etc. Understanding the topography is the basis of any sampling and monitoring plan; the topography may define sampling locations and sample density. For example, when sampling stream sediments, denser sampling grids can be necessary in areas of uneven river bottom topography compared to more uniform topographic areas or where the river reach displays many bends in a sinuous watercourse.

LIMITATIONS/INTERFERENCES: Maps are only as good as the scale they are derived from. The larger the scale, the more detailed the map will be.

BIBLIOGRAPHY:

Alday, J.G., Marrs, R.H., and Martinez-Ruiz, C., 2010, The importance of topography and climate on short-term revegetation of coal wastes in Spain: Ecological Engineering, v. 36, issue 4, p. 579-585.

Geodetic Services, Inc., 2011, The basics of photogrammetry: 65 p., http://www.geodetic.com/v-stars/what-is-photogrammetry.aspx, accessed 9/156/13.

McGuire, K.J., McDonnell, J.J., Weiler, M., Kendall, C., McGlynn, B.L., Welker, J.M. and Seibert, J., 2005, The role of topography on catchment-scale water residence time, Water Resources Research, v. 41, 14 p., W05002, doi:10.1029/2004WR003657, http://www.cof.orst.edu/cof/fe/watershd/pdf/2005/McGuire_etal2005.pdf, accessed 9/16/13.

Nyssen J. and Vermeersch, D., 2010, Slope aspect affects geomorphic dynamics of coal mining spoil heaps in Belgium: Geomorphology, v. 123, p. 109-121.

Welker, J. M., Seibert, J., and Snyder, J.P., 1987, Map Projections-A Working Manual: U.S. Geological Survey Professional Paper 1395.

U.S. Geological Survey, 2000, US GeoData Digital Elevation Models: U.S. Geological Survey, Fact Sheet 040-00, http://egsc.usgs.gov/isb//pubs/factsheets/fs04000.pdf, accessed 8/23/13.

U.S. Geological Survey, 2002, Map scales: U.S. Geological Survey, Fact Sheet 015-02, http://pubs.usgs.gov/fs/2002/0015/report.pdf, accessed 8/23/13

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Geomorphology

(Virginia T. McLemore)

MEASUREMENT: Geomorphology is the study of landforms, the processes that form the landforms, and the processes that shape and configure the landforms. Geomorphology significantly influences the hydrology of an area (Vincent, 2008) and is affected by tectonics, climate, ecology, and human activity, such as mining, construction, farming, and other similar activities. Geomorphic processes can affect how metals and other contaminants are transported and stored, especially by changing the flow of water (Miller, 1997; Marcus et al., 2001; Macklin et al., 2006). In some areas, analysis has shown that mining and other human activities have a major influence on landform modification and soil and sediment transport (Rivas et al., 2006).

MEDIUM/PARAMETERS: Most sampling and monitoring plans should include a geomorphic history of the area. Geomorphic features should be mapped and characterized using field techniques, aerial photographs, LANDSAT (Novak and Soulakellis, 2000; Bobrowsky, 2005), and other techniques.

MEASUREMENT OBJECTIVES: Geomorphic monitoring at the sites generally includes identification and characterization of geomorphic features, such as stream terraces, glacier boulder fields, dissected terrains, plateaus, river/stream channels, floodplains, tidal flats, slopes, landslides, etc. Historical data, including older aerial photographs and cultural (archaeological) surveys often provide information on changes of the geomorphologic features over time. Additionally, periodic mapping can illustrate contemporary changes in geomorphology. See http://water.epa.gov/polwaste/nps/watershed/hydrologic_geomorphic.cfm (accessed 9/16/13) for more information.

ANALYTICAL TECHNIQUES: Most geomorphic investigations involve the use of field surveying and examining topographic maps, air photos and other maps of different dates (Rivas et al., 2006). New advances in numerical modeling techniques enables the prediction of the location and extent of contaminated sediment in river systems and their movement with time (Macklin et al., 2006).

Methods include mapping landforms, surveys of the channel and riparian areas, subsurface features and topography under the riparian canopy, and measurements of the size distribution of channel bed materials, among others. Understanding the limits of 100 year, 1000 year, and other flood stages is critical in developing monitoring and sampling programs and ultimately any reclamation program.

Investigations of tailings dam failures can provide insight on the geomorphologic controls of these systems (Marcus et al., 2001). The physical and chemical factors that control the dispersal and storage of sediment-associated contaminants need to be documented (Macklin et al., 2006). Geochemical fingerprinting can aid in identifying and distinguishing sediment from mining sources and non-mining sources and when the contamination occurred.

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LIMITATIONS/INTERFERENCES: The expertise of the geological team can vary resulting in differences in observing, recognizing and defining patterns at different scales, defining the useful scale.

BIBLIOGRAPHY:

Bloom, A.L., 1998, Geomorphology: A Systematic Analysis of Late Cenozoic Landforms–Third Edition*:* Prentice–Hall, Upper Saddle River, NJ, 482 p.

Bobrowsky, P.T., 2005, Geoenvironmental mapping Methods, Theory and Practice: Taylor and Francis Group, Plc., London, UK, 735 p.

Dorava, J.M., Montgomery, D.R., Palcsak, B.B., and Fitzpatrick, F.A., (eds.), 2001, Geomorphic Processes and Riverine Habitat: Water Science and Application 4, American Geophysical Union, Washington, D.C., 253 p.

Fookes, P.G., Lee, E.M., and Griffiths, J.S., 2007, Engineering Geomorphology, Theory and Practice: CRC Press, Whittles Publishing, Dunbeath, Caithness, Scotland, 281 p.

Macklin, M.G., Brewer, P.A., Hudson-Edwards, K.A., Bird, G., Coulthard, T.J., Dennis, I.A., Lechler, P.J., Miller, J.R., and Turner, J.N., 2006, A geomorphological approach to the management of rivers contaminated by metal mining: Geomorphology, v. 79, p. 423-447.

Marcus, W.A., Meyer, G.A., and Nimmo, D.R., 2001, Geomorphic control of persistent mine impacts in a Yellowstone Park stream and implications for the recovery of fluvial systems: Geology, v. 29, p. 355-358.

Miller, J.R., 1997, The role of fluvial geomorphic processes in the transport and storage of heavy metals from mine sites: Journal of Geochemical Exploration, v. 58, p. 259-267.

Novak, I.D. and Soulakellis, N., 2000, Identifying geomorphic features using LANDSAT-5/TM data processing techniques on Lesvos, Greece: Geomorphology, v. 34, issue 1-2, p. 101-109.

Ritter, D.F., Kochel, R.C., and Miller, J.R., 2002, Process Geomorphology–Second Edition: McGraw–Hill Companies, New York, 560 p.

Rivas, V., Cendrero, A., Hurtado, M., Cabral, M., Giménez, J., Forte, L. del Río, L., Cantú, M. and. Becker, A, 2006, Geomorphic consequences of urban development and mining activities; an analysis of study areas in Spain and Argentina: Geomorphology, v. 73, p. 185-206.

Vincent, K.R., 2008, Questa baseline and pre-mining ground-water quality investigation. 17. Geomorphology of the Red River Valley, Taos County, New Mexico, and influence on ground-water flow in the shallow alluvial aquifer: U.S. Geological Survey Scientific Investigations Report 2006–5156, p. 50, http://pubs.usgs.gov/sir/2006/5156/, accessed 8/23/13.

Stratigraphy and Lithology

(Virginia T. McLemore)

MEASUREMENT: Stratigraphy is the study of rock layers and layering and traditionally is used in the study of sedimentary and volcanic rocks (lithostratigraphy) and biologic stratigraphy (biostratigraphy). However, in environmental studies, mine rock piles (or mine dumps) and tailings form layered deposits and stratigraphic principles can

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be used to describe and characterize them throughout the mine-life cycle (Fines et al., 2003; McLemore et al., 2009). The Principle of Superposition is a fundamental principle of stratigraphy and states that rock layers are deposited in a time sequence, with the oldest on the bottom and youngest on top. The Principle of Horizontality is another fundamental principle of stratigraphy and states that layers of sediment are originally deposited horizontally under the force of gravity (Fig. A3.1).

Lithology is the description of the rocks in the field at outcrop and hand specimen scale or with low magnification. It includes characterization parameters such as color, texture, grain size, and composition. The naming of the rock type is based upon identification of one of the three major rock types (sedimentary, igneous, metamorphic) and then classified by the mineralogy and texture. Igneous rock lithologies were identified on the basis of mineralogy and chemistry as defined by LeMaitre (1989). Fine-grained volcanic rocks can be classified according to their chemical composition (Le Bas et al., 1986).

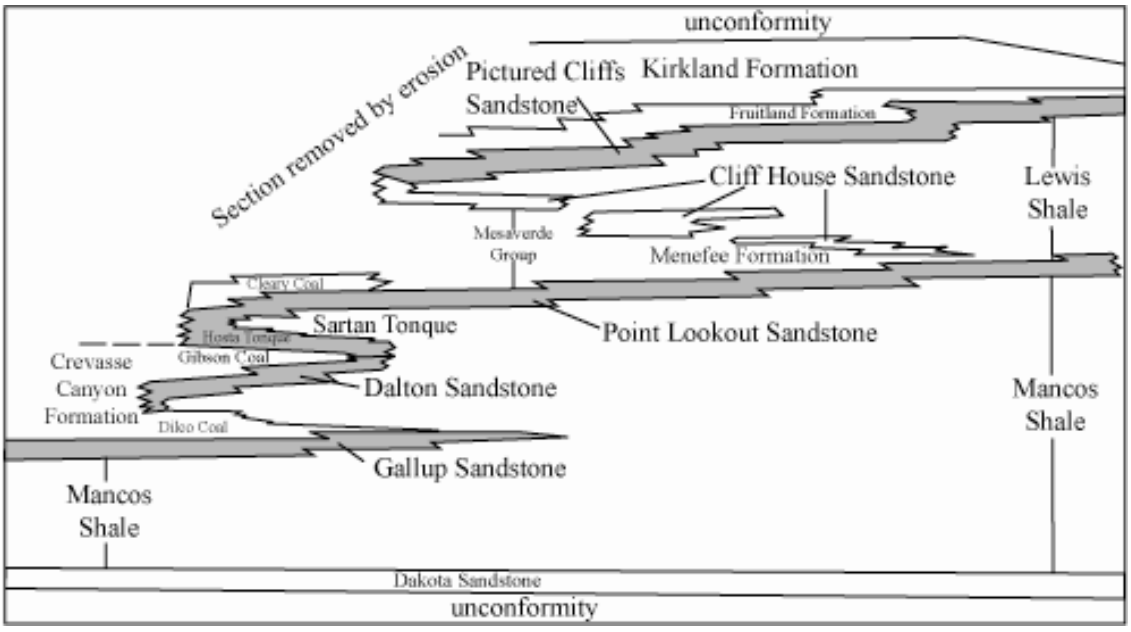


FIGURE A3.1. Simplified stratigraphic sequence of the Late Cretaceous sedimentary rocks in the San Juan Basin (McLemore, 2010). The oldest rocks are at the bottom of the sequence and younging upwards.

MEASUREMENT OBJECTIVES: The measurement objectives are to identify lithology, describe textures, and measure thickness of geologic units. These are useful throughout the mine-life cycle.

SPECIFIC EQUIPMENT/TECHNIQUES: The specific tools required are petrographic microscope, geologic maps, hand lens, hand specimen, and thin sections.

ANALYTICAL TECHNIQUES: Petrographic analysis includes identification of minerals and lithologies, description of alteration, description of texture, grain size, grain

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shape, etc. Description of stratigraphic sections includes composition, texture, thickness, internal structure, etc.

LIMITATIONS/INTERFERENCES: The expertise of the geological team can vary resulting in differences in observing, recognizing and defining patterns at different scales, defining the useful scale.

MEDIUM/PARAMETERS: Parameters that could be described and mapped include:

- Lithology
- Pyrite and other mineralogy on the surface
- Orientation layers and other structure at the surface (strike, dip, UTM northing and easting)
- Estimate range in grain size, texture
- Color
- Paste pH, conductivity, moisture content
- Location surface features, including rills, fractures, vents, cracks, etc.
- Soil consistency (sticky, hard, soft, not sticky)
- Thickness of geologic units
- Internal structure of geologic units

BIBLIOGRAPHY:

Delvigne, J.E., 1998, Atlas of micromorphology of mineral alteration and weathering: Canadian Mineralogist, Special Publication 3, 494 p.

Fines, P., Wilson, G.W., Williams, D.J., Tran, A.B., and Miller, S., 2003, Field characterization of two full-scale waste rock piles; in proceedings of the 6th ICARD: Cairns, Queensland, p. 903-909.

Le Bas, M.J., Le Martre, R.W., Streckusen, A., and Zanettin, B., 1986, A chemical classification of volcanic rocks based on the total alkali-silica diagram: Journal of Petrology, v. 27, p. 745-750.

LeMaitre, R. W., ed., 1989, A classification of igneous rocks and glossary of terms: Blackwell Scientific Publications, Oxford, Great Britain, 193 p.

McLemore, V.T., 2010, Distribution, Origin, and Mineral Resource Potential of Late Cretaceous Heavy Mineral, Beach-Placer Sandstone Deposits, San Juan Basin, New Mexico: New Mexico Geological Society Guidebook 61, p. 197-212, http://nmgs.nmt.edu/publications/guidebooks/downloads/61/61_p0197_p0212.pdf , accessed 9/16/13.

McLemore, V.T., Fakhimi, A., van Zyl, D., Ayakwah, G.F., Boakye, K., Anim, K., Ennin, F., Felli, P., Fredlund, D., Gutierrez, L.A.F., Nunoo, S., Tachie-Menson, S., and Viterbo, V.C., 2009, Literature review of other rock piles: characterization, weathering, and stability: New Mexico Bureau of Geology and Mineral Resources, Open-file Report 517, 101 p., http://geoinfo.nmt.edu/publications/openfile/details.cfml?Volume=517, accessed 8/23/13.

McLemore, V.T., Dickens, A., Boakye, K., Campbell, A., Donahue, K., Dunbar, N., Gutierrez, L., Heizler, L., Lynn, R., Lueth, V., Osantowski, E., Phillips, E., Shannon, H., Smith, M., Tachie-Menson, S., van Dam, R., Viterbo, V.C., Walsh,

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P., and Wilson, G.W., 2008a, Characterization of Goathill North Rock Pile: New Mexico Bureau of Geology and Mineral Resources, Open-file report 523, http://geoinfo.nmt.edu/publications/openfile/details.cfml?Volume=523, accessed 8/23/13.

Neuendorf, K.K.E., Mehl, J.P., Jr., and Jackson, J.A., 2005, Glossary of Geology: American Geological Institute, 5th ed., Alexandria, Virginia, 779 p.

Ragland, P.C., 1989, Basic Analytical Petrology, Oxford University Press, Inc., New York, 369 p.

Williams, H., Turner, F.J., and Gilbert, C.M., 1954, Petrography; an introduction to the study of rocks in thin sections, W.H. Freeman, San Francisco, 406 p.

Structural Geology

(Virginia T. McLemore)

MEASUREMENT: Structural geology in environmental studies uses measurements of modern rock geometries to characterize: (1) the history of deformation of the rocks in the project area, (2) the structural evolution (i.e., history) of the project area with respect to regionally widespread patterns of rock deformation, and (3) how these structures affect the environmental processes. A fundamental principle of structural geology is the principle of cross-cutting relationships, which states that an igneous intrusion, fault, or vein is always younger than the rock it cuts across (Fig. A3.2).

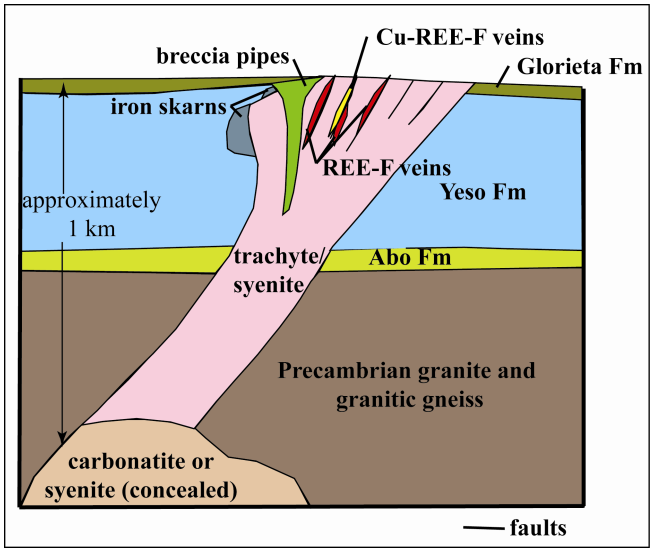


FIGURE A3.2. Cross cutting relationships. Gallinas Mountains, Lincoln County, New Mexico (McLemore, 2010). The oldest rocks are at the bottom of the section (Precambrian) and the youngest rocks are at the top and the entire section is cut by the cross cutting trachyte/syenite.

MEDIUM/PARAMETERS: Measurements of geologic structures such as bedding planes, faults, fractures, lineations, fold axis, channels, joints, geologic contacts, and other features are important in environmental studies. Not only can these features control mineralization and alteration in the project area, but they also can control the flow of

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water and seepage of potentially toxic materials into the environment. The inclination of a plane is measured by strike (the line of intersection between the planar feature and the horizontal plane) and dip (the magnitude of the inclination).

MEASUREMENT OBJECTIVES: Geologists use a variety of methods to measure rock geometries, reconstruct their deformational histories, calculate the stress field that resulted in that deformation, and define the effect of these deformations on the environmental processes. Folded and faulted rocks commonly provide conduits for the movement of mineralizing fluids during the formation of ore deposits and during modern day flow of potentially contaminated fluids into the environment.

SPECIFIC SAMPLE EQUIPMENT/TECHNIQUES: The primary tools of a geologist are geologic maps (pictures of the distribution and patterns of rocks and structural features on the surface) and cross sections (pictures of the distribution and patterns of rocks and structural features in the subsurface).

ANALYTICAL TECHNIQUES: Two basic geologic principles are (1) superposition (generally, the oldest layer will be on the bottom and the layers become progressively younger above, unless strata have been overturned by folding or other geologic processes are in effect) and (2) cross cutting relationships (a fault or intrusion that offsets or cuts across another fault or intrusion is always the younger because the original fault or intrusion had to exist before it could be offset by a fault or cut by an intrusion). Stereographic projection of structural features (projects strike and dip measurements onto a sphere) is a powerful method for analyzing the nature and orientation of deformation stresses, lithological units, and penetrative fabrics. From the geologic maps and cross sections, geologic conceptual models are developed that illustrate the geologic processes involved in the project area, including ore formation, alteration haloes, and past and modern fluid flow. These models then can be used to define the boundary conditions for numerical modeling. Contour maps of structures, isopach maps of thicknesses of stratigraphic units, and other specialized techniques can aid in understanding of the overall geology of the project area.

LIMITATIONS/INTERFERENCES: The expertise of the geological team can vary resulting in differences in observing, recognizing and defining patterns at different scales, defining the useful scale.

DOCUMENTATION/DATA HANDLING: Detailed geologic maps and cross sections with field notes are critical but must be simplified for most reports. Measurements of structural features should be in a database.

BIBLIOGRAPHY:

McLemore, V.T., 2010, Geology and mineral deposits of the Gallinas Mountains, Lincoln and Torrance Counties, New Mexico; preliminary report: New Mexico Bureau of Geology and Mineral Resources, Open-file report OF-532, 92 p., http://geoinfo.nmt.edu/publications/openfile/downloads/OFR500-599/526-550/532/ofr_532.pdf, accessed 8/23/13.

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Pollard, D.P. and Raymond C. Fletcher, R.C., 2005, Fundamentals of structural geology: Cambridge University Press, New York, 500 p. http://www.eas.slu.edu/People/KChauff/earth_history/Correlsquenceevent-posted.pdf, accessed 8/23/13.

Documenting Previous Exploration, Mining and Processing Activities

(Virginia T. McLemore)

MEASUREMENT: It is important to identify, locate, and map all historical features, including mines, shafts, adits, prospect pits, quarries, mills, smelters, etc. because these features can affect water quality, public safety, aquatic life, and the environment (Buxton et al., 1997). Not all water quality issues in a specific river/stream or drainage basin are attributed to a single point source, but can be the result of a combination of drainage from numerous features found along the river/stream or drainage basin. A watershed or basin approach is one method of organizing this information (Buxton et al., 1997; for examples see http://amli.usgs.gov/data/boulder/ and http://amli.usgs.gov/data/animas/, accessed 8/23/13). Some of these features could have historical or archaeological significance and could be classified as historic, archaeological, or cultural preservation sites by local, state, and federal agencies.

MEDIUM/PARAMETERS: Information, such as location, size and extent of workings, production, reserves, resource potential, significant deposits, geology, geochemistry (rock, water, etc.), well data, historical and recent photographs, mining methods, maps, ownership, and other data, should be collected from literature surveys, especially of historic unpublished records, and field observations. Mine rock piles, tailings, slag piles, and other mine features need to measured, mapped, examined, and sampled.

MEASUREMENT OBJECTIVES: Measurement objectives include:

- Identify areas where historical mining and mineral processing activities took place
- Identify and characterize the effect of past mining and mineral processing activities on water quality, aquatic health, human health and the environment
- Provide data to aid in establishing pre-mining background and current baseline conditions for mining areas
- Provide information that could be useful in exploration of new deposits

SPECIFIC SAMPLE EQUIPMENT/TECHNIQUES: Use GPS and plot on topographic maps. Some sites could require more detailed mapping.

ANALYTICAL TECHNIQUES: Air photos and remote sensing techniques could be employed to aid in identifying field sites.

LIMITATIONS/INTERFERENCES: The expertise of the geological team can vary resulting in differences in observing, recognizing and defining patterns at different scales, defining the useful scale.

DOCUMENTATION/DATA HANDLING: A database is an excellent media to store this information (McLemore et al., 2005a, b). The most difficult task is maintaining

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quality control of both the data being entered into the database and of the database. Quality control (QC) is a system of procedures, checks, audits, and corrective actions to ensure that data are collected, stored, and analyzed in an acceptable manner, and that technical aspects and reporting are adequate. The main objectives of a database QC program are to:

- Document the procedures for data collection, preparation, and analysis
- Provide assurance as to precision and accuracy of chemical analyses using replicate samples, cross-laboratory checks, and certified reference materials
- Provide information regarding data analysis and interpretation
- Define an acceptable magnitude of error on chemical analyses
- Determine completeness of the data set
- Define the decision-making process to be used
- Provide a chain of custody of samples when required
- All of these objectives can be met with relational databases.

ADDITIONAL CONSIDERATIONS: One of the concerns about releasing these data is that the general public will have ready access to locations of inactive mines. **Recreation in or around inactive mine sites is extremely dangerous, and can result in serious injury or death.**

BIBLIOGRAPHY:

Buxton, H.T., Nimick, D.A., von Guerard, P., Church, S.E., Frazier, A.G., Gray, J.R., Lipin, B.R., March, S.P., Woodward, D.F., Kimball, B.A., Finger, S.E., Ischinger, L.S., Fordham, J.C., Power, M.S., Bunck, C.M., and Jones, J.W., 1997, A Science-based, Watershed Strategy to Support Effective Remediation of Abandoned Mine Lands: Fourth International Conference on Acid Rock Drainage (ICARD) May 30-June 6, 1997, Vancouver, British Columbia, Canada, http://amli.usgs.gov/reports/icard97/icard97.html, accessed 8/23/13.

Kimball, B.A., Church, S.E., and Besser, J.M., 2006, Lessons Learned from the U.S. Geological Survey Abandoned Mine Lands Initiative—1997-2002; *in* Barnheisel, R.I., ed., Proceedings of the 7th International Conference on Acid Rock Drainage ICARD, St. Louis, MO, p. 944-962, http://amli.usgs.gov/reports/Kimball_Church_and_Besser_2006.pdf, accessed 8/23/13.

McLemore, V.T., Hoffman, G., Smith, M., Mansell, M., and Wilks, M., 2005a, Mining districts of New Mexico: New Mexico Bureau of Geology and Mineral Resources, Open-file Report 494, CD-ROM, http://geoinfo.nmt.edu/publications/openfile/details.cfml?Volume=494, accessed 8/23/13.

McLemore, V.T., Krueger, C.B., Johnson, P., Raugust, J.S., Jones, G.E., Hoffman, G.K., and Wilks, M., 2005b, New Mexico Mines Database: Mining Engineering, February, p. 42-49.

Nimick, D.A., and von Guerard, P., 1998, Science for Watershed Decisions on Abandoned Mine Lands: Review of Preliminary Results, Denver, Colorado, February 4-5, 1998: U.S. Geological Survey Open-File Report 98-0297, http://pubs.usgs.gov/of/1998/0297/, accessed 8/23/13.

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U.S. Bureau of Land Management, Abandoned Mine Lands portal: http://www.abandonedmines.gov/, accessed 8/23/13.

Alteration and Mineralized Outcrops

(Virginia T. McLemore)

MEASUREMENT: Mineral deposits are typically surrounded by a zoned halo of altered rocks, which aid in their discovery. These altered rocks can contain sulfides, especially pyrite, and other minerals that could affect regional drainage quality even if these areas are not disturbed during mining. These rocks typically form the overburden or rock surrounding the ore deposit that have to be removed in order to access the ore. Characterization of these rocks early in the mine life cycle is important because:

- The distribution and characterization of the alteration mineral assemblage is important in identifying the ore deposit and distribution of the ore minerals.
- These rocks could adversely affect drainage quality naturally and if so, should be evaluated in order to determine natural background and/or baseline conditions prior to mining.
- In some locations, these rocks will have to be removed as overburden or mine waste and should be characterized in order to be properly managed during disposal, typically in waste rock piles (also known as mine dumps).
- Some of these areas contain sufficient metals of economic interest (i.e. copper, gold, uranium, etc.) that they could be economically heap leached during times when the commodity price is high.

MEDIUM/PARAMETERS: *Alteration* is a general term describing the changes in mineralogy, texture, and chemistry of a rock as a result of a change in the physical, thermal, and chemical environment in the presence of water, steam, or gas, including those changes produced by weathering (Henley and Ellis, 1983; Reed, 1997; Neuendorf et al., 2005; Ludington et al., 2005). The nature of the alteration depends upon (Browne, 1978; Henley and Ellis, 1983; Ludington et al., 2005):

- Temperature and pressure (including amount of overburden or depth) at the alteration site
- Composition of the parent rock
- Quantity and composition of the altering (invading) fluids
- Permeability of the parent rock
- Duration and intensity of the alteration process (i.e. time)
- Distance from the alteration source, resulting in alteration zoning
- Climate.

Hydrothermal alteration is the change in original composition of rock in place by hydrothermal (warm to hot) solutions during or after the mineralization. In this study, hydrothermal alteration refers to pre-mining conditions. Alteration includes hypogene or hydrothermal (primary), supergene (secondary), and weathering alteration. *Hypogene alteration* occurred below the surface during the formation of the ore body by upwelling (ascending), hydrothermal or warm to hot fluids (Neuendorf, et al., 2005). *Supergene alteration* is the natural weathering, before mining, of the ore body, at low temperatures at and near the Earth's surface by descending fluids (Neuendorf, et al., 2005).

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Weathering is the set of physical and chemical changes, up to and including disintegration of rock by physical, chemical, and/or biological processes, occurring at or near the earth's surface (e.g. in the vadose zone at temperatures less than or equal to approximately 70°C) that result in reductions of grain size, changes in cohesion or cementation, and changes in mineralogical composition. According to the AGI Geologic Glossary (Neuendorf et al., 2005), weathering is "the destructive process or group of processes by which earthy and rocky materials on exposure to atmospheric agents at or near the earth's surface are changed in color, texture, composition firmness, of form with little or no transport of the loosen or altered material; specifically the physical disintegration and chemical decomposition of rock that produce an in-situ mantle of waste…" Weathering is simply the consequence of exposing rocks to the conditions at the Earth's surface as a result of fairly low temperatures, low pressures, organic activity, and chemically active substances such as water and the gases of the atmosphere. There are two general types of weathering that must be examined during this project, physical weathering and chemical weathering. *Physical weathering* involves the physical breakup of a rock by mechanical processes. *Chemical weathering* encompasses those changes to the structure and composition of the rock fragments due to geochemical and biogeochemical reactions. Chemical weathering encompasses changes to the texture, structure, and composition of the rock fragments due to geochemical and biogeochemical reactions. Physical and chemical weathering processes overlap in time and space and it is often difficult to determine which process is most responsible for the observed weathered effects. Many scientists study these processes in terms of geologic scale. The general expression of weathering in natural environments is three-fold:

- Transformations of minerals from their pre-depositional character to new compositions and structures in better accord with the new energy environment. For example, pyrite oxidizes in the presence of molecular oxygen and water; the iron and sulfate released in the dissolution process may be re-precipitated elsewhere as secondary minerals, such as jarosite or goethite.
- Changes in rock texture due to dissolution, precipitation, or solid-state transformations (McLemore et al., 2009a). For example, a rock containing stockwork veins with pyrite and calcite can weather under oxidizing conditions as the pyrite oxidizes, calcite dissolves, and secondary gypsum and iron oxide precipitate; the mineralogical changes may be observed microscopically or even macroscopically. Especially if there is low confining stress (i.e., near the surface of the rock pile), the large molar volume of gypsum compared to the molar volumes of the parent minerals could fracture the rock along veinlets where this reaction is occurring.
- Changes in rock texture and particle size due to mechanical strain. Frost-wedging of fractures by ice during winter conditions is a comparable, entirely physical process that can fracture (in fact, "frost-shatter") rocks. If the stress of the overlying column of rock is sufficiently great and lateral confining pressures are low, it also is possible for crushing to occur deep in a rock pile. A special case that is relevant to Questa rock piles is the physical breakup of the rock by the volume change resulting from transformation of anhydrite to gypsum or other crystal growth along fractures (Campbell and Lueth, 2008). Crystallization of

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mineral phases also can break up the rock, for example the crystallization of salt increases the volume by as much as 5%.

Diagenesis is a broad term related to the chemical, physical, and biological changes affecting sediment after its initial deposition and during and after its lithification. Diagenesis does not include weathering or metamorphism of the sediment, but does include pedogenesis or the processes that form soils (Neuendorf et al., 2005). Weathering and diagenetic processes overlap in time and space, especially in cementation of the rock-pile material. Cementation is the diagenetic process where sediments become lithified or consolidated or the binding-together of particles into hard, compacted rocks (Neuendorf et al., 2005). In this operational definition, there is no clear distinction between weathering and diagenesis, although the coarse-grained and subaerial nature of the mined rock pile restricts the nature of "diagenetic" reactions compared to, for example, those that might occur in a submarine, saturated system.

Weathering is the result of complex climatic, geological, biological, and physical processes as well as geography and climate (Fookes et al., 1971, 1988, Lan, et al, 2003) and the composition of the resulting products are likely to be heterogeneous, especially since the composition of the rock-pile material is heterogeneous. Weathering can change the original texture, structure, mineralogy, and chemistry of the material and tends to change a material from rock-like to soil-like material. Rocks of similar composition in the same area can weather differently depending upon these processes. Different lithologies (i.e. rock compositions) and hydrothermal alteration assemblages can have different weathering rates under similar physical, chemical, and biological processes and therefore have different geological and engineering properties (Lan et al., 2003). The weathering environment and duration of weathering can be variable. Furthermore, weathering is not entirely a subtractive process, but additional elements can be added from precipitation of solutions, such as pore fluids. Other factors, summarized below, significantly affect the changes observed during weathering and can in fact, have more effect on the engineering properties than mineralogy and chemistry.

A characteristic alteration zonation surrounds most porphyry molybdenum systems and exhibits a distinctive district alteration zoning (Fig. A3.3; Martineau et al., 1977; Loucks et al., 1977; Molling, 1989; Meyer and Leonardson, 1990; Ross et al., 2002; Ludington et al., 2005). Supergene alteration is commonly superimposed on the hypogene alteration, especially in near-surface rocks mined early in the history of Questa. In some places, supergene weathering along open fracture systems can extend to considerable depths. Weathering is superimposed on both the hypogene and supergene alteration.

MEASUREMENT OBJECTIVES: Define, characterize, and map the alteration, weathering, and diagenesis in time and space and determine the effect or the potential impact on the environmental geology and geochemistry.

SPECIFIC SAMPLE EQUIPMENT/TECHNIQUES: Geologic maps and cross sections are the main tools to understand alteration. Various geochemical techniques can provide insight as to the paragenesis and conditions of formation.

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ANALYTICAL TECHNIQUES: geologic maps and cross sections must be compiled.

LIMITATIONS/INTERFERENCES: The expertise of the geological team can vary resulting in differences in observing, recognizing and defining patterns at different scales, defining the useful scale. Some minerals found in altered areas are too small to be determined in the field and must be identified by laboratory techniques.

DOCUMENTATION/DATA HANDLING: All field observations must be part of the project database.

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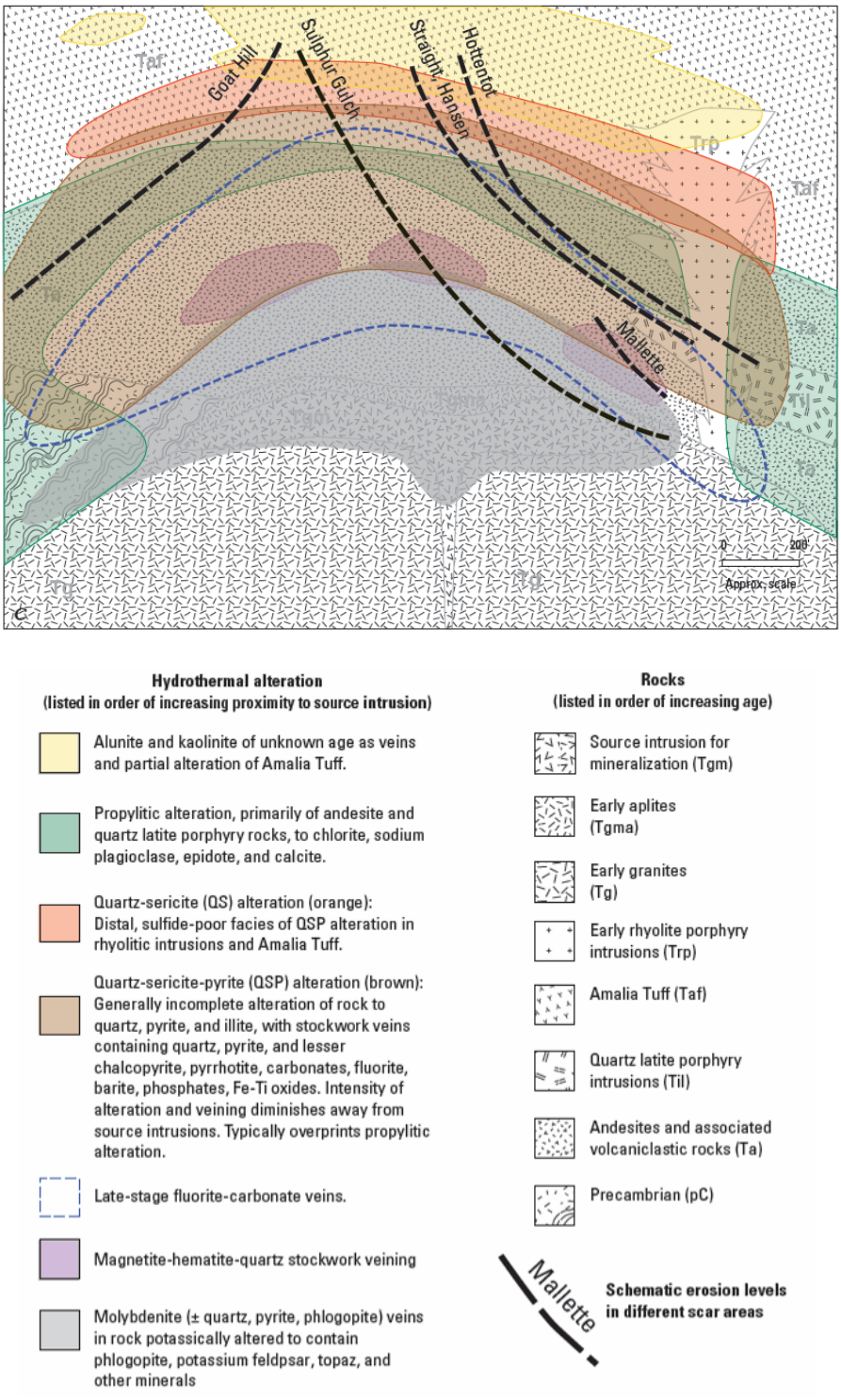


FIGURE A3.3. Cross section showing schematic spatial distributions of lithologies, alteration assemblages, and alteration scars in the Red River valley, New Mexico (taken from Ludington et al., 2005, which is modified from Martineau et al., 1977).

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BIBLIOGRAPHY:

Browne, P.R.L., 1978, Hydrothermal alteration in active geothermal fields: Annual Reviews of Earth and Planetary Science, v. 6, p. 229–250.

Campbell, A.R. and Lueth, V.W., 2008, Isotopic and textural discrimination between hypogene, ancient supergene, and modern sulfates at the Questa Mine, New Mexico: Applied Geochemistry, v. 23, p. 308-319.

Fookes, P. G., C. S. Gourley, et al., 1988, Rock weathering in engineering time: Quarterly Journal of Engineering Geology, v. 21, p. 33-57.

Henley, R.W. and Ellis, A.J., 1983, Geothermal systems, ancient and modern: a geochemical review: Earth Science Reviews, v. 19, p. 1–50.

Lan, H.X., Hu, R.L., Yue, Z.Q., Lee, C.F., and Wang, S. J., 2003, Engineering and geological characteristics of granite weathering profiles in South China: Journal of Asian Earth Sciences, v. 21, p. 353-364.

Loucks, T.A., Phillips, J.S., and Newell, R.A., 1977, Exploration potential of the Questa East area: unpublished report for Kennecott Copper Corporation.

Ludington, S., Plumlee, G., Caine, J., Bove, D., Holloway, J., and Livo, E., 2005, Questa baseline and pre-mining ground-water quality Investigation, 10. Geologic influences on ground and surface waters in the lower Red River watershed, New Mexico: U.S. Geological Survey, Scientific Investigations Report 2004-5245, 46 p.

Martineau, M.P., Heinemeyer, G.R., Craig, S.D., and McAndrews, K.P., 1977, Geological report: Questa Project 1975-1977: unpublished mine report, Questa Molybdenum Company.

McLemore, V., Heizler, L., Donahue, K., and Dunbar, N., 2009a, Characterization of Weathering of Mine Rock Piles: Example from the Questa Mine, New Mexico, USA: paper presented at the 8th ICARD, June 2009, 10 p., http://geoinfo.nmt.edu/staff/mclemore/documents/Virginia_McLemore_P_T2_Characterization-of-weathering-of-mine-rock-piles-example-from-the-Qu.pdf, accessed 8/23/13.

McLemore, V., Sweeney, D., Dunbar, N., Heizler, L. and Phillips, E., 2009b, Determining quantitative mineralogy using a combination of petrographic techniques, whole rock chemistry, and MODAN: Society of Mining, Metallurgy and Exploration Annual Convention, preprint 09-20, 19 p.

Meyer, J.W., and Leonardson, R.W., 1990, Tectonic, hydrothermal and geomorphic controls on alteration scar formation near Questa, New Mexico: New Mexico Geological Society, Guidebook 41, p. 417-422, http://nmgs.nmt.edu/publications/guidebooks/downloads/41/41_p0417_p0422.pdf , accessed 9/16/13.

Molling, P.A., 1989, Applications of the reaction progress variable to hydrothermal alteration associated with the deposition of the Questa molybdenite deposit, NM: PhD dissertation, Johns Hopkins University, 227 p.

Neuendorf, K.K.E., Mehl, J.P., Jr., and Jackson, J.A., 2005, Glossary of Geology: American Geological Institute, 5th ed., Alexandria, Virginia, 779 p.

Reed, M.H., 1997, Hydrothermal alteration and its relationship to ore fluid composition; in Barnes, H.L., ed., Geochemistry of hydrothermal ore deposits: 3rd edition, John Wiley and Sons, New York, New York, p. 303-365.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Ross, P.S., Jebrak, M., and Walker, B.M., 2002, Discharge of hydrothermal fluids from a magma chamber and concomitant formation of a stratified breccia zone at the Questa porphyry molybdenum deposit, New Mexico: Economic Geology, v. 97, p. 1679-1699.

Ore Deposit Types–A Fundamental Consideration for the Mining Environmental Geochemist

(Ron L. Schmiermund)

INTRODUCTION: The science of ore deposits is a dramatically expanding and increasingly complex field of study. The evolution of the journal *Economic Geology*, in continuous publication since 1905, provides documentation of this expansion, which is ostensibly driven by the desire to increase the rate of new discoveries and to enhance the efficiency of their exploitation. Only recently has this vast knowledge base been brought to bear on the environmental consequences of mining and the increasing requirement for responsible mining. Utilizing the classification of ore deposits is the first step toward sharing this information.

MEDIUM/PARAMETERS: Define ore deposit, differentiate between economic ore deposit, mineral deposit, mineral occurrence; define geo-environmental aspects of ore deposits; define baseline and background.

OBJECTIVES: Numerous compilations of ore deposit characteristics, typically organized into types and sub-types (a.k.a. models), have been prepared based on differing philosophies. Classic texts (Guilbert and Park, 1986) have been followed by more concise treatments, including Roberts and Sheahan (1988), Cox and Singer (1986), Sheahan and Cherry (1993), Eckstrand (1984), and in-depth monograph collections (Hedenquist et al., 2005). A review of even these very few works reveals the remarkable evolution in thinking and synthesis about ore deposits, as exemplified by Sillitoe (2010). The physical, chemical and mineralogical characteristics of ore deposits, combined with their locations and environments, dictate much of the consequences of their exploitation. The concept of geo-environmental models of ore deposits (du Bray, 1995) was built upon the model nomenclature of Cox and Singer (1986) and sought to embrace all aspects of mining of ore deposits and processing of ores as they pertain to the environment.

By considering an ore deposit's type and understanding the current models for that type, the environmental geochemist can obtain important insight and perspective into the design of environmental sampling programs and the interpretation of the resulting information. Ore deposits are often extremely complex and rarely fully understood when environmental studies are commissioned for permitting purposes. Although the best source of information in this regard is likely the exploration and mining geologists, they have specific agendas that do not necessarily serve environmental programs. Consider the simple fact that their interest is ore, and that of the environmental geochemist is waste rock. For example, the pyrite content of waste rock may be declared "negligible" relative to the ore zone, but in reality may be entirely sufficient to produce substantial amounts of acid rock drainage (ARD). Similarly, the "oxide zone" of a supergene-altered epigenetic gold deposit is that part that can be economically heap leached from the metallurgist's

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perspective, not necessarily the part with no potential for ARD because all pyrite has been oxidized. Knowledge of the characteristics of similar ore deposits will prompt such statements to be examined critically from an environmental perspective.

A mine plan, which is in part dependent on the ore deposits characteristics, is a highly dynamic and complex construct and mine planners have specific objectives—all production based. These objectives are not necessarily in line with those of the environmental geochemist. For example, a non-ore carbonate country rock adjacent to an ore-bearing intrusion may be mined as waste to achieve proper pit slopes. The same rock may also be ideal for road construction because of its durability, or for drain lining because of its carbonate content, resulting in an economically beneficial coincidence. However, consideration of the ore deposit model may lead to valid environmental concerns about that waste rock, for example, the possibility that leachable non-ore metals of potential environmental concern may be enriched at the contact in a skarn zone. Failure to recognize the rock's unsuitability in a timely fashion can lead to placement of the contaminated rock in environmentally-risky locations and/or significant and very expensive changes to the mine plan upon realization of the problem. Frequently the situation is logistically and financially irreversible and may result in "treatment in perpetuity" of poor quality runoff.

Baseline studies can benefit from a good understanding of the ore deposit in question through the recognition of local genetically-associated but non-commercial mineralization of environmental significance. Selection of the appropriate suite of elements to be considered in a baseline study will be influenced by the ore deposit type, as will be the scope of the study. Although a mine's formal environmental liability may be confined to the immediate mining area, many ore deposits are now known to be genetically related to much larger geologic features with broad elemental haloes and may be associated with diverse satellite deposits. Documenting such conditions outside the immediate mining area is important for limiting or allocating future liability. Consideration of historic mining activities is also important to assembling a holistic view of the regional impacts of mineralization. In addition, such broad elemental enrichments and past mining may be responsible for pre-existing bio-concentration of toxic elements in local food chains with resulting health issues.

The language of ore deposits is quite involved, and may sometimes seem arcane, contradictory and obtuse to the non-economic geologist. The science, however, is quite sophisticated and the literature is vast, dispersed and ever changing. For these reasons it is not reasonable for the environmental geochemist to act alone in assimilating all the relevant information. Consultation with project geologists, mine planners, mine engineers and historical information sources is mandatory. However, neither is it responsible to take any of these sources at face value without testing the information against environmental requirements. It is incumbent upon the environmental geochemist to become sufficiently familiar with the ore deposit type of concern in order to take advantage of the added perspective and awareness that such knowledge provides.

BIBLIOGRAPHY:

Cox, D.P., and Singer, D.A. 1986. *Mineral Deposit Models*. Bulletin 1693. Reston, VA: U.S. Geological Survey.

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du Bray, E. A. (ed.) 1995. Preliminary Compilation of Descriptive Geoenvironmental Mineral Deposit Models: U.S. Geological Survey. Open-File Report 95-831.
Eckstrand, O.R. 1984. *Canadian Mineral Deposit Types: A Geological Synopsis*. Economic Geology Report 36. Geological Survey of Canada, 86 p., http://pubs.usgs.gov/of/1995/ofr-95-0831/, accessed 9/16/13.
Guilbert, J.M., and Park, C.F. 1986. *The Geology of Ore Deposits*. New York: W.H. Freeman, xiv, 985 p.
Hedenquist, J.W., Thompson, J.F.H., Goldfarb, R.J., and Richards, J.P. (eds.) 2005. *Economic Geology—One Hundredth Anniversary Volume*. Littleton, CO: Society of Economic Geologists, Inc., 1136 p.
Roberts, R.G., and Sheahan, P.A. (eds.) 1988. *Ore Deposit Models*. Reprint Series 3. St. Johns, NL: Geological Association of Canada, 194 p.
Sheahan, P.A., and Cherry, M.E. (eds.) 1993. *Ore Deposit Models*, Vol. II. Reprint Series 6. St. Johns, NL: Geological Association of Canada, 154 p.
Sillitoe, R.H. 2010. Porphyry copper systems. *Economic Geology* 105(1):3-41.

CLIMATOLOGICAL INFORMATION AND AIR MEASUREMENTS AND DATA

Meteorological Data

(Carol Russell)

MEDIA: Meteorological Measurements

MEASUREMENT OBJECTIVES:

Meteorological observations at mining sites may provide critical information in the development of a water balance, input for water quality/quantity projections for mine design and for evaluation of potential pathways for potential toxic impacts to human health or to the environment. This information may also be useful for energy conservation; stormwater, wind engineering and tailings cap design; and insurance and emergency measures. Meteorological data inform decisions for all stages of the mine life cycle, and therefore should be collected at each stage of mining.

As with all data collection efforts, the key to obtaining useful meteorological data is careful attention to the purpose of the data being collected. Of primary importance is the proper placement of the weather station and respective instruments. The overwhelming majority of these sites are located on airport property and follow guidelines laid out in the Federal Meteorological Handbook. A sensor placed at the surface monitors only a portion of its surroundings, the source area. Sensors such as thermometers, hygrometers, and anemometers monitor properties such as temperature, humidity, atmospheric gases, wind speed, and direction. Generally speaking the weather station should be situated in a representative location as closely as possible to the mine waste areas but away from obstructions such as trees and buildings. Ten meter towers are often used to raise measurement heights above low-lying obstructions. Calibration of the instruments is very important (NCore Calibration and Accuracy Criteria).

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SPECIFIC SAMPLING TECHNIQUE(S)/EQUIPMENT:

Ambient Temperature (Thermistor) ***and Relative Humidity*** (Psychrometer/Hygrometer)
The sensor (resistance temperature detectors also known as a thermistor) should be housed in a ventilated radiation shield. The USEPA recommends the sensor be no closer than four times an obstruction's height, at least 30 m from large paved areas, and located in an open level area that's at least 9 m in diameter. The open areas should be covered by short grass, or where grass does not grow, the natural earth.

Wind Speed and Direction (Anemometer cup or vane)
The cup anemometer has cups that relate the rate of rotation and the wind speed. The wind vane is a fin tied to a vertical shaft to measure direction. Wind sensors should be located over open level terrain. The USEPA recommends the wind sensor be a distance of at least ten times the height of nearby buildings, trees or other obstructions. Wind speed sensor bearings deteriorate and should be checked.

Solar Radiation (Pyranometer and UV Radiometer)
Pyranometers should be mounted away from shadows, reflective surfaces, and sources of artificial radiation. Mounting the pyranometer on the southernmost (northern hemisphere) part (either crossarm or pyranometer mounting arm) of the weather station should minimize shading from the other weather station structures. The height the sensor is mounted is not critical for the accuracy of the measurement. However, pyranometers mounted at heights of 3 m or less are easier to level and clean.

Precipitation (Tipping bucket)
Precipitation is defined as, "the total amount of precipitation which reaches the ground in a stated period is expressed as the depth to which it would cover a horizontal projection of the earth's surface if there were no loss by evaporation or run-off and if any part of the precipitation falling as now or ice were melted." The AASC and USEPA suggest tipping buckets no closer than four times the height of an obstruction. The orifice of the gage must be in a horizontal plane, open to the sky, and above the level of in-splashing and snow accumulation. Typically, tipping buckets are sited on level ground covered with short grass or gravel. Wind shields, such as those used by the National Weather Service, are recommended for open areas.

Analytical Techniques & Limitations
Each of the meteorological parameters has specific Meteorological Measurement Quality Objectives as described on pages 8-14 of the third reference in the bibliography.

> "The placement of the tower system is more important than the placement of individual sensors. Site selection for tower placement should address the question, "Is the site (are the data) representative?" Representativeness is defined as "the extent to which a set of measurements taken in a space-time domain reflects the actual conditions in the same or different space-time domain taken on a scale appropriate for a specific application." In general, the location of the tower should be representative of meteorological conditions in the "area of interest."

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Sampling strategies vary depending on the variable being measured, the sensor employed, and the accuracy required in the end use of the data. The recommended sampling averaging times for wind speed and wind direction measurements is 1-5 seconds; for temperature and temperature difference measurements, the recommended sample averaging time is 30 seconds for air quality modeling. However, if the data needed is for the design of a tailings facility to ensure a robust design for potential flooding the time period of reference might be the maximum probable flood based on all recorded data. The value of annual and/or seasonal estimates is in relationship to the resource identified for protection.

BIBLIOGRAPHY:

U.S. Department of Commerce National Oceanic and Atmospheric Administration 2005. *Federal Meteorological Handbook No. 1* (FCM-H1- 2005) *Surface Weather Observations and Reports* September 2005, www.ofcm.gov, accessed 8/23/13.

U.S. Environmental Protection Agency, 2000, *Meteorological Monitoring Guidance for Regulatory Modeling Applications* Office of Air Quality EPA-454/R-99-005Research Triangle Park, NC 27711 February 2000, http://www.epa.gov/ttn/amtic/files/ambient/met/Volume%20IV_Meteorological_Measurements.pdf, accessed 8/23/13.

World Meteorological Organization 2008 –*Guide to Meteorological Instruments and Methods of Observation and Guide to Meteorological Instrument*s *and Methods of Observation of the World Meteorological Organization; Geneva Switzerland*. WMO-No. 8 Seventh edition, http://www.wmo.int/pages/prog/gcos/documents/gruanmanuals/CIMO/CIMO_Guide-7th_Edition-2008.pdf, accessed 8/23/13.

Mercury Methods

(Carol C. Russell, Charles N. Alpers, and Sandra Spence)

MEDIA: Air, Water, Soil, and Fish Tissue

MEASUREMENT OBJECTIVES:

Mercury is a naturally occurring element toxic to humans when it is ingested or inhaled. However, methylmercury is the form of greatest importance for human exposure. Methylmercury can be formed from inorganic forms of mercury by microbial action in sediment and soils (Furutani and Rudd 1980; Gilmour et al. 1987). Once formed, methylmercury can be taken up by aquatic organisms and bioaccumulates up the aquatic food web (USEPA 1997). Addressing mercury releases to the air is important because mercury in the air can be deposited to water, converted to methylmercury, and taken up by fish. Mercury can also be released directly to water from wastewater treatment plants, industrial facilities, and from areas affected by current and historical mining activities. The use of mercury for gold and silver amalgamation by artisanal placer miners is of particular international concern. Of special importance is an understanding of the reduction-oxidation balance between aquatic mercury and atmospheric mercury, and the effect of this cycling on the total mercury presence in freshwater and marine systems.

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Measurement objectives focus on the magnitude, concentration distribution, and speciation of mercury in sources, pathways and receptors to assess and manage mercury and methylmercury risks. Because of the complex interactions of mercury species in the atmosphere and the relatively low levels involved, sample collection and analysis of atmospheric mercury is expensive. Sampling and analysis protocols are strict, and laboratories with capabilities to successfully analyze low levels of mercury are limited.

SAMPLING AND ANALYTICAL TECHNIQUES:

***Air*—**Atmospheric transport and fate of mercury have relative contributions from local, regional, and global sources (AMAP/UNEP 2008). Therefore the methodology and instrumentation to make semi-continuous ambient measurements that distinguish among mercury forms—elemental gaseous mercury, divalent mercury, and particulate phase mercury—are important. Mercury associated with ore and waste rock volatilizes at ambient air temperatures (Eckley et al. 2010, 2011a, b). Stack emissions from mineral processing facilities (roasting or autoclaving) are commonly sampled using continuous emission monitors. Obtaining data from the nearest Mercury Deposition Network site prior to initiating site specific monitoring is recommended.

Because mercury exhibits dynamic cycling in the environment and atmospheric deposition is a major source for terrestrial and aquatic systems, it is important to estimate the expected local atmospheric contribution when characterizing a watershed and evaluating non-atmospheric source contributions. Soil and water samples from reference watersheds may be used to determine expected background concentrations as well as direct collection of wet and dry deposition samples for analysis. Also, atmospheric deposition models may be used to estimate atmospheric loads, site specifically (e.g., www.epa.gov/AMD/Tools/wdt.html, accessed 9/28/13).

Potential equipment for mercury air sampling includes: a direct mercury analyzer (USEPA method 7473) such as the Tekran 2537A Gaseous Mercury Analyzer fitted with a 1130 (RGM) and a 1135 (Hg(p)) speciation unit, plus micrometeorological equipment for measuring other routine ambient air quality parameters (Utah Department of Environmental Quality 2008).

***Soil/Sediment*—**Generally, total mercury is measured in soil and is compared to an action limit for cleanup; however, several forms of mercury can be expected to make up the total mercury pool in the soil. For contaminated soils, the form of mercury found is dependent upon several factors including the form in which it was released, soil chemistry and characteristics, moisture content, organic content, microbial community, sulfur content and form, and colloid content (Davis et al. 1997; USEPA 2000). In relatively uncontaminated soil, the vast majority (98%) of mercury is found as Hg(II) complexes and compounds (Bloom et al. 1994; Hultberg et al. 1994; Mason et al. 1994; Yin et al. 1996; Davis et al. 1997).

The most common mercury minerals (cinnabar and metacinnabar, HgS) are not very soluble but may be reduced to Hg(0) by photolysis or contact with amines, aldehydes, and ketones (Davis et al. 1997). This mobilizes mercury into the vapor phase and allows for either transport of elemental mercury out of soils or provides for additional transformations within the soil. Oxidation of Hg(0) produces Hg(I) that tends to oxidize to Hg(II) under neutral conditions or remain as Hg(I) under reducing (and dark acidic)

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conditions (Davis et al. 1997). Organic mercury compounds found in sediments typically include monomethylmercury and dimethylmercury with limited reports of ethylmercury. Of the organic mercury compounds, monomethylmercury dominates (Barkay et al. 2003).

Dynamic flux chambers are commonly used for emissions from a wide range of surface substrate, such as mine waste; however, there is no existing standard method or protocol. USEPA SW846 Method 7471 and USEPA Method 245.5 are widely used laboratory methods for mercury in sediment and soil samples, with Method 7471 being the preferred method. Method 7471 allows detection of all forms of mercury including the relatively insoluble and least toxic mercury sulfide, HgS. The milder solvents used in Method 245.5 do not solubilize HgS and the method estimates the more soluble, more toxic forms of mercury. Neither of these methods is considered to be a speciation method, but comparison of the same samples analyzed by both methods can give an estimate on the fraction of mercury that is more toxic. Method 7471 is approved for measuring total mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials.

***Water*—**Generally, total mercury and sometimes methylmercury are measured in the water column and the values are compared to USEPA or state water-quality criteria. However, water-column mercury tends to be present in very low concentrations and is difficult and costly to sample and analyze (one may need to use ultra-clean techniques—see below). The USGS provides guidelines for collecting and processing water samples for analysis of mercury at ultra-trace levels (sub-nanogram per liter) that are applicable to precipitation samples as well as surface waters (Wilde et al. 2004; USGS 2006). Another recommended resource regarding water sampling techniques is USEPA Method 1669, which is applicable to sample collection for trace metals (including mercury) in ambient water at USEPA water quality criteria limits. For all sampling, risk of sample contamination is high when working at such low mercury levels. Unless extraordinary measures are taken to prevent contamination, great expenses could be incurred for worthless data. The clean hands/dirty hands protocol (Wilde et al. 2004) should be followed rigorously (e.g. Alpers et al. 2000). This also includes wearing lint-free, particle-free clothing and double gloves, using double-bagged, scrupulously pre-cleaned containers, and avoiding breathing over the samples.

For mercury in surface water, the preferred laboratory methods are USEPA SW846 Method 1631e for low levels and USEPA Method 245.1 for moderate levels (USEPA 2007). The ease of contaminating ambient water samples with mercury and interfering substances cannot be overemphasized. The detection limit and minimum level of quantitation in this method typically are dependent on the level of interferences rather than instrumental limitations. The method detection limit (MDL) for mercury has been determined to be 0.2 ng/L when no interferences are present. The minimum level of quantitation has been established as 0.5 ng/L. An MDL as low as 0.05 ng/L can be achieved for low-mercury samples by using a larger sample volume, a lower bromine monochloride (BrCl) level (0.2%), and extra caution in sample handling. For methylmercury, the preferred laboratory method is USEPA Method 1630. A typical method detection limit for methylmercury is 0.025 ng/L.

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***Fish Tissue*—**One should check for local, state, and national fish advisories. USEPA is moving toward fish-tissue-based water-quality standards in which methylmercury is measured in either fillets or whole bodies (USEPA 2001, 2010). As methylmercury tends to bioaccumulate, the concentration in fish tissue is usually orders of magnitude higher than in the water column and is easier and less expensive to analyze. In addition, methylmercury may be measured in other biota, such as macroinvertebrates, for risk assessment purposes. Because it is more costly to measure methylmercury than total mercury in tissue samples, total mercury may be analyzed with the assumption that the vast majority of mercury present in biological tissues (especially in fish fillet tissue) is methylated. Alternatively, literature values may be used to estimate the expected ratio of methylmercury to total mercury in various organisms; however this ratio varies widely for macroinvertebrates. Fish and invertebrate sampling locations should be collocated with any surface water and sediment sampling program. This is important in the overall understanding of fate and transport processes for mercury in the system being investigated. To a degree, temporally and spatially collocating biotic and abiotic samples assists in examining contaminants detected between sites with differing environmental contaminant conditions by removing a level of uncertainty. Selection of the most appropriate sampling period is very important. Preliminary recommendations (Utah Department of Environmental Quality 2008) are to complete sampling from late summer to early fall (i.e., August to October), when the amount of surface water dilution from high spring melt flows is lowest, and spawning periods have ceased. The lower water levels are also more conducive for sampling activities. It is important to research species spawning information because conditions vary by region and species.

SPATIAL OR TEMPORAL CONSIDERATIONS:

Soil moisture enhances mercury flux from soil: furthermore, diel and seasonal variations in mercury releases have been observed. Special protocols have been developed by USEPA for coal combustion residues to establish mercury-leaching potential and thermal stability for the range of environmental conditions (USEPA 2006). Diel variations in methylmercury have also been observed in some systems, in part caused by photodegradation of methylmercury that occurs only during daylight hours (e.g., Fleck et al. 2013). Food web studies involving phytoplankton and zooplankton should have a seasonal component because of the complex dynamics at the base of the food web (e.g., Stewart et al. 2008).

BIBLIOGRAPHY:

Alpers, C.N., Taylor, H.E., and Domagalski, J.L. (eds.), 2000, Metals transport in the Sacramento River, California, 1996-97. Volume 1. Methods and data. U.S. Geological Survey Water-Resources Investigations Report 99-4286, 428 p. http://pubs.usgs.gov/wri/wrir_994286/ (accessed 9/28/13)

AMAP/UNEP, 2008, Technical Background Report to the Global Atmospheric Mercury Assessment. Arctic Monitoring and Assessment Programme/UNEP Chemicals Branch, 159 p. www.chem.unep.ch/mercury/Atmospheric_Emissions/Technical_background_report.pdf (accessed 9/28/13)

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Barkay T., Miller SM, and Summers AO, 2003. Bacterial Mercury Resistances from Atoms to Ecosystems. FEMS Microbiol Rev. 27:355-384.

Bloom NS, and Porcella DB, 1994. Less Mercury. Nature. 357:694-695.

Davis A., Bloom N.S., and Que Hee, S.S., 1997. The environmental geochemistry and bioaccessibility of mercury in soils and sediments: A review, Risk Analysis. 17 (5): 557–569.

Eckley, C.S., Gustin, M., C-J Lin, Miller, M.B., 2010, *The influence of dynamic chamber design and operating parameters on calculated surface-to-air mercury fluxes,* Atmospheric Environment 44:194-203.

Eckley, C.S., Gustin, M., Marsik, F., Miller, M.B., 2011a, *Measurement of surface mercury fluxes at active gold mines in Nevada (USA).* Science of the Total Environment 409:514-522.

Eckley, C.S., Gustin, M., Miller, M.B., and Marsik, F., 2011b, Scaling non-point-source mercury emissions from two active industrial gold mines: Influential variables and annual emission estimates. Environmental Science and Technology 45:392-399.

Fleck, J.A., Gill, G., Downing, B.D., Bergamaschi, B.A., and Alpers, C.N., 2013, Concurrent photolytic degradation of dissolved aqueous methylmercury and dissolved organic matter: Science of the Total Environment. http://dx.doi.org/10.1016/j.scitotenv.2013.03.107 (accessed 9/28/13)

Furutani A., and Rudd J.W.M., 1980. Measurement of mercury methylation in lake water and sediment samples. Appl, Environ. Microbiol. 40: 770-776.

Gilmour C.C., Henry E.A., Mitchell R., 1992. Sulfate stimulation of mercury methylation in freshwater sediments. Environ. Sci. Technol. 26: 2281-2287.

Hultberg H, Iverfeldt A, Lee YH, 1994. Methylmercury Input/Output and Accumulation in Forested Catchments and Critical Loads for Lakes in Southwestern Sweden. In CJ Watras and J Huckabee (eds.) Mercury Pollution Integration and Synthesis. (Lewis Publishers. Boca Raton, FL.) 313 - 332.

Mason RP, Fitzgerald WF, Morel FM, 1994. The Biogeochemical Cycling of Elemental Mercury: Anthropogenic Influences. Geochimica et Cosmochimica Acta. 58:3191-3198.

Stewart, A.R., Saiki, M.K., Kuwabara, J.S., Alpers, C.N., Marvin-DiPasquale, M., and Krabbenhoft, D.P., 2008, Influence of plankton mercury dynamics and trophic pathways on mercury concentrations of top predator fish of a mining-impacted reservoir. Canadian Journal of Fisheries and Aquatic Sciences, v. 65, p. 2351–2366. http://dx.doi.org/10.1139/F08-140 (accessed 9/28/13)

USEPA (U.S. Environmental Protection Agency). 1997. Mercury Study Report to Congress, Volumes I – VIII. EPA-452/R-97-004. www.epa.gov/hg/report.htm (accessed 9/28/13

USEPA (U.S. Environmental Protection Agency). 2000, *2000 Progress Report: Processes Controlling the Chemical/Isotopic Speciation and Distribution of Mercury from Contaminated Mine Sites.* http://cfpub.epa.gov/ncer_abstracts/index.cfm/fuseaction/display.abstractDetail/abstract/250/report/2000 (accessed 9/28/13)

USEPA (U.S. Environmental Protection Agency). 2001, *Water quality criterion for the protection of human health: Methylmercury*. EPA-823-R-01-001; January 2001.

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http://water.epa.gov/scitech/swguidance/standards/criteria/aqlife/methylmercury/upload/2009_01_15_criteria_methylmercury_mercury-criterion.pdf (accessed 9/28/13)

USEPA (U.S. Environmental Protection Agency). 2006, *EPA's Roadmap for Mercury – VI Conducting Mercury Research and Monitoring.* E*PA-HQ-OPPT-2005-0013.* www.epa.gov/mercury/roadmap.htm (accessed 9/28/13)

USEPA (U.S. Environmental Protection Agency). 2007, *Analytical Methods for Mercury in National Pollutant Discharge Elimination System (NPDES) Permits.* Memorandum signed August 23, 2007. http://water.epa.gov/scitech/methods/cwa/metals/mercury/upload/2007_10_02_pubs_mercurymemo_analyticalmethods.pdf (accessed 9/28/13)

USEPA (U.S. Environmental Protection Agency). 2010, *Guidance for Implementing the January 2001 Methylmercury Water Quality Criterion*. EPA 823-R-10-001. U.S. Environmental Protection Agency, Office of Water, Washington, D.C. http://water.epa.gov/scitech/swguidance/standards/criteria/aqlife/methylmercury/upload/mercury2010.pdf (accessed 9/28/13)

USGS (U.S. Geological Survey). 2006. Collection of water samples (ver. 2.0): U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A4, September 2006. Available at: http://pubs.water.usgs.gov/twri9A4/ (accessed 9/28/13)

Utah Department of Environmental Quality, 2008, *Mercury Source Protocol,* http://utah.ptfs.com/Data/Library2/publications/dc000216.pdf (accessed 9/28/13)

Wilde, F.D., Radtke, D.B., Gibs, Jacob, and Iwatsubo, R.T., eds., 2004 with updates through 2009, Processing of water samples (ver. 2.2): U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chap. A5, April 2004. http://pubs.water.usgs.gov/twri9A5/ (accessed 9/28/13)

Yin Y., Allen H.E., Li Y., Huang C.P., Sanders P.F., 1996. Adsorption of mercury (II) by soil: Effects of pH, chloride, and organic matter. J. Environ. Qual. 25: 837-844.

Air Particulates as PM2.5

(Michael Copeland)

MEDIA: Ambient Air

MEASUREMENT OBJECTIVES: The data quality objectives (DQOs) have been established for PM2.5 monitoring by regulation. The PM-2.5 standard includes particles with a diameter of 2.5 micrometers or less. The goal for acceptable measurement uncertainty has been defined as 10 percent coefficient of variation (CV) for total precision and ±10 percent for total bias. This guidance is primarily applicable to PM2.5 ambient air monitoring with reference methods carried out by State and local air monitoring agencies in their State and local air monitoring stations' (SLAMS's) air surveillance networks under the air monitoring requirements of 40 CFR Part 58. This guidance is also applicable to other organizations required to carry out SLAMS or SLAMS-related PM2.5 monitoring and to any PM2.5 monitoring activity for which the PM2.5 reference method measurements will be gathered and/or entered into the Aerometric Quality System (AQS) database.

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SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): A "method" for PM2.5 generally consists of the following:

- A PM2.5 sampler or analyzer designed, built, and sold by a particular manufacturer
- An operation or instruction manual provided by the instrument manufacturer that describes the proper use and operation of the sampler or analyzer
- The other operational and quality assurance requirements necessary to obtain reliable PM2.5 concentration measurements.

Methods used for monitoring PM2.5 in SLAMS or SLAMS-related air monitoring are required to be either reference or equivalent methods as designated by USEPA under the requirements and provisions of 40 CFR Part 53.

Depending on the method chosen for monitoring, samples are either gathered on a 46.2mm Teflon® filter placed within the air sample stream, run for a 24-hour sampling and averaging period, and tare-weighed to derive a mass per unit time, or particulate in the air is measured "real-time" using various proprietary techniques, averaged hourly, and then averaged over a 24-hour period. Both methods are compared to the 24-hour and annual USEPA standard for PM2.5 in the ambient air. The choice between a filter-based method and a continuous "real-time" monitor should be weighed with the consideration of whether post-analysis of filters for speciation of contaminants is desired. The former, filter-based, method would be required to enable this post-analysis processing of filters.

ANALYTICAL TECHNIQUE(S):

A. An example of filter-based sampling technique is as follows:

I. Prepare for Site Visit on Scheduled Date/Time

Be sure to take the following equipment and supplies to each of the sites:

- Preweighed sampling filter(s) in cassette(s), packed in labeled containers. Spares.
- Preweighed field blank filters in cassettes, packed in labeled containers, if a field blank study is scheduled.
- PM2.5 sampler run data sheet for each sampler. Use of electronic data sheets is encouraged. Add comments to the data sheets. Site notebook and calculator.

II. Install Filter/Cassette and Begin Sampler Operations

Note: If a filter is still in the sampler, follow the instructions for removing the sample/cassette. Refer to Part III. Also, if the schedule calls for any of the following procedures, perform them before installing a new sampling filter/cassette:

- Impactor well replacement or cleaning (after every fifth sampling event)
- Sampler flow verification check (every 4 weeks)
- Sampler ambient pressure and temperature checks (every 4 weeks)
- Sampler verification/calibration (temperature, pressure, flow rate)
- Cleaning of sampler inlet head and downtube (monthly)
- Leak check (after every fifth sampling event)
- Field blanks.

Perform the following steps to install the filter/cassette and begin sampler operations:

- Be sure the sampler is not operating.
- Fill in initial information on the data sheet.

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- Remove the new filter/cassette from its protective container and visually inspect the filter/cassette for flaws. Verify that this is the correct filter for this sampler, site, and run date.
- Remove the sampler's filter holder assembly (if required by the manufacturer's instructions).
- Inspect the O-rings inside the filter holder; service them if necessary.
- Install the filter/cassette in the filter holder assembly and then install the loaded filter holder assembly in the sampler based on the manufacturer's instructions. If you touch or scratch the filter, void the filter, record this fact, and get another one from the set of extra filters brought to the site.
- Program the sampler to turn on at the beginning of a sampling period (consult the sampler's instruction manual). The sampling day begins at midnight.
- Make independent measurements of ambient temperature (T_a) and ambient pressure (P_a) using transfer standards. Record these values and the T_a and P_a values indicated by the sampler on the data sheet.

III. Remove Filter/Cassette and End Sampler Operations

Perform the following steps to remove the filter/cassette and end sampler operations:

- The filter/ cassette must be removed within 96 hours after the collection period has ended.
- Determine Pressure and Temperature using transfer standards. Enter the values on the data sheet.
- Based on review of the data, record the stop time; total elapsed time; initial and final flow rates; total volume sampled; T_a; P_a; and so on the data sheet.
- After each completed run, download data from the sampler data port to a laptop or alternative data storage device.
- Open the filter holder assembly (consult the sampler's instruction manual); remove the used filter/cassette; quickly visually inspect the filter for tears, oil, insects, moisture, and so on; and record your observations on the data sheet.
- Place the filter/cassette inside a properly labeled protective container. Verify the container's label versus the site name, date, and so on.
- Place the container inside a cooled storage chest. Do not allow the protective container to come into contact with ice or water. Sealed cooling blocks are recommended. Protect the containers from condensed water.
- Inspect the interior of the filter housing. Note any abnormalities.
- Make a final check of the site, and observe and record the presence of any activity that may have affected the particulate loading of the sample.
- Keep the container holding the filter/cassette at a temperature below 25^{o}C (preferably cooled to 4^{o}C), and promptly deliver or ship it and the data sheet (with comments) to the sample custodian or balance operator in the weighing laboratory.

B. Continuous "real-time" sampling techniques utilize a monitor capable of monitoring without the necessity of a lab-based analysis of filters. Sampler setup, calibration, and regular QA/QC checks are required and regular downloading and analysis of data to ensure that the instrument is performing correctly. As stated previously, there is no filter

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involved in this sampling method, and no post-sampling analysis of filters for speciation purposes would be possible.

LIMITATIONS/INTERFERENCES: One limitation is maintaining the specified ambient air flow rate through the sampling inlet and filter assembly. The flow rate determines the size of the particles that are collected. The effects of this limitation are minimized by following sampler construction requirements and by employing procedures and checks to ensure the proper flow rate is maintained within close tolerances.

Another important limitation involves changes in the weight of a collected sample due to mishandling, chemical reactions, and volatilization. Handling procedures, choice of filter media, humidity and temperature control of the filter and sample during collection and subsequent processing, and promptness in weighing the sample following collection all help control filter artifacts. The chemical makeup of the PM2.5 particulate matter will vary with sampling location and source. Thus, the magnitude of PM2.5 weight changes due to chemical and physical processes will also vary with site location.

DOCUMENTATION/DATA HANDLING: A quality assurance project plan (QAPP) and SOPs for various aspects of the PM2.5 sampling program should be prepared. Data to be gathered consists of the following:

- Siting documentation (upon installation and any changes thereafter)
- PM2.5 concentration data, μg/m3, or sample weight and volume sampled to allow electronic calculations (entered electronically into the AIRS database)
- Information calculated and provided by the sampler. This information is retrieved from the data port of each sampler. It is generally downloaded in the field to a laptop computer or another data storage device and later processed in the laboratory or office. Data outputs to be provided to the AIRS database are indicated in table 12-1 by the bullet symbol.
- Results of all valid precision, bias, and accuracy tests performed during the quarter.

BIBLIOGRAPHY:

USEPA. 1998. Quality Assurance Guidance Document 2.12, Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods. U.S. Environmental Protection Agency. Publication 2.12, USEPA Human Exposure and Atmospheric Sciences Division, National Exposure Research Laboratory, Washington, DC. November.

USEPA. 2006a. Reference method for the determination of fine particulate matter as PM2.5 in the atmosphere. U.S. Environmental Protection Agency. 40 CFR Part 50, Appendix L, as amended October 17, 2006.

USEPA. 2006b. Ambient air quality surveillance. U.S. Environmental Protection Agency. 40 CFR Part 58, as amended October 17, 2006.

USEPA. 2006c. Ambient air quality monitoring methodology. U.S. Environmental Protection Agency. 40 CFR Part 58, Appendix C, as amended October 17, 2006.

USEPA. 2006d. Ambient air monitoring reference and equivalent methods. U.S. Environmental Protection Agency. 40 CFR Part 53, as amended October 17, 2006.

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Air Particulates as PM10

(Michael Copeland)

MEDIA: Ambient Air

MEASUREMENT OBJECTIVES: The data quality objectives (DQOs) have been established for PM10 monitoring by regulation. The goal for acceptable measurement uncertainty has been defined as 10 percent coefficient of variation (CV) for total precision and ±10 percent for total bias. This guidance is primarily applicable to PM10 ambient air monitoring with reference methods carried out by State and local air monitoring agencies in their State and local air monitoring stations' (SLAMS's) air surveillance networks under the air monitoring requirements of 40 CFR Part 58. This guidance is also applicable to other organizations required to carry out SLAMS or SLAMS-related PM10 monitoring and to any PM10 monitoring activity for which the PM10 reference method measurements will be gathered and/or entered into the Aerometric Quality System (AQS) database.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): A "method" for PM10 generally consists of the following:

- A PM10 sampler or analyzer designed, built, and sold by a particular manufacturer
- An operation or instruction manual provided by the instrument manufacturer that describes the proper use and operation of the sampler or analyzer
- The other operational and quality assurance requirements necessary to obtain reliable PM10 concentration measurements.

Methods used for monitoring PM10 in SLAMS or SLAMS-related air monitoring are required to be either reference or equivalent methods as designated by USEPA under the requirements and provisions of 40 CFR Part 53.

Depending on the method chosen for monitoring, samples are either gathered on an 8 inch by 10 inch Microquartz Fiber filter (high-volume) or a 46.2mm Teflon® filter (low-volume), placed within the air sample stream, run for a 24-hour sampling and averaging period, and tare-weighed to derive a mass per unit time; or particulate in the air is measured "real-time" using various proprietary techniques, averaged hourly, and then averaged over a 24-hour period. Both methods are compared to the 24-hour and annual USEPA standard for PM10 in the ambient air. The choice between a filter-based method and a continuous "real-time" monitor should be weighed with the consideration of whether post-analysis of filters for speciation of contaminants is desired. The former, filter-based, method would be required to enable this post-analysis processing of filters.

ANALYTICAL TECHNIQUE(S):

A. An example of filter-based sampling technique is as follows:

I. Prepare for Site Visit on Scheduled Date/Time

Be sure to take the following equipment and supplies to each of the sites:

- Pre-weighed sampling filter(s) in cassette(s), packed in labeled containers. Spares.

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- Pre-weighed field or trip blank filters in cassettes, packed in labeled containers, if a field or trip blank study scheduled.
- PM10 sampler run data sheet for each sampler. Use of electronic data sheets is encouraged. Add comments to the data sheets.
- Site notebook and calculator.

II. Install Filter/Cassette and Begin Sampler Operations

Note: If a filter is still in the sampler, follow the instructions for removing the sample/cassette. Refer to Part III. Also, if the schedule calls for any of the following procedures, perform them before installing a new sampling filter/cassette:

- Sampler flow verification check (every 4 weeks)
- Sampler ambient pressure and temperature checks (every 4 weeks)
- Sampler verification/calibration (temperature, pressure, flow rate)
- Cleaning of sampler down-tube and/or inlet head (monthly)
- Leak check (after every fifth sampling event)
- Field blanks.

Perform the following steps to install the filter/cassette and begin sampler operations:

- Be sure the sampler is not operating.
- Fill in initial information on the data sheet.
- Remove the new filter/cassette from its protective container and visually inspect the filter/cassette for flaws. Verify that this is the correct filter for this sampler, site, and run date.
- Remove the sampler's filter holder assembly (if required by the manufacturer's instructions).
- Inspect the O-rings inside the filter holder; service them if necessary.
- Install the filter/cassette in the filter holder assembly and then install the loaded filter holder assembly in the sampler based on the manufacturer's instructions. If you touch or scratch the filter, void the filter, record this fact, and get another one from the set of extra filters brought to the site.
- Program the sampler to turn on at the beginning of a sampling period (consult the sampler's instruction manual). The sampling day begins at midnight.
- Make independent measurements of ambient temperature (T_a) and ambient pressure (P_a) using transfer standards. Record these values and the T_a and P_a values indicated by the sampler on the data sheet.

III. Remove Filter/Cassette and End Sampler Operations

Perform the following steps to remove the filter/cassette and end sampler operations:

- The filter/ cassette must be removed as soon as possible after the collection period has ended. Particle loss or filter damage will result if the filter is left in the sampler for extended periods.
- Determine Pressure and Temperature using transfer standards. Enter the values on the data sheet.
- Based on review of the data, record the stop time; total elapsed time; initial and final flow rates; total volume sampled; T_a; P_a; and other parameters as needed on the data sheet. Final flow rate may have to be determined post operation by running the sampler post-sampling with the filter installed for high-volume samplers.

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- After each completed run, download data from the sampler data port to a laptop or alternative data storage device (if applicable).
- Open the filter holder assembly (consult the sampler's instruction manual); remove the used filter/cassette; quickly visually inspect the filter for tears, oil, insects, moisture, and so on; and record your observations on the data sheet.
- Place the filter/cassette inside a properly labeled protective container. Verify the container's label versus the site name, date, and so on.
- Inspect the interior of the filter housing. Note any abnormalities.
- Make a final check of the site, and observe and record the presence of any activity that may have affected the particulate loading of the sample.
- The sampler may now be readied for the next sampling period if necessary.

B. Continuous "real-time" sampling techniques utilize a monitor capable of monitoring without the necessity of a lab-based analysis of filters. Sampler setup, calibration, and regular QA/QC checks are required and regular downloading and analysis of data to ensure that the instrument is performing correctly. As stated previously, there is no filter involved in this sampling method, and no post-sampling analysis of filters for speciation purposes would be possible.

LIMITATIONS/INTERFERENCES: One limitation is maintaining the specified ambient air flow rate through the sampling inlet and filter assembly. The flow rate determines the size of the particles that are collected. The effects of this limitation are minimized by following sampler manufacturer requirements and by employing procedures and checks to ensure the proper flow rate is maintained within close tolerances.

Another important limitation of filter-based particulate sampling involves changes in the weight of a collected sample due to mishandling, chemical reactions, and volatilization. Handling procedures, choice of filter media, humidity and temperature control of the filter and sample during collection and subsequent processing, and promptness in weighing the sample following collection all help control filter artifacts. The chemical makeup of the PM10 particulate matter will vary with sampling location and sources. Thus, the magnitude of PM10 weight changes due to chemical and physical processes will also vary with site location.

DOCUMENTATION/DATA HANDLING: A quality assurance project plan (QAPP) and SOPs for various aspects of the PM10 sampling program should be prepared. Data to be gathered consists of the following:

- Siting documentation (upon installation and any changes thereafter)
- PM10 concentration data (μg/m3), or sample weight and volume of air sampled to allow electronic calculations (entered electronically into the AQS database)
- Information calculated and provided by the sampler if applicable. This information is retrieved from the data port of each sampler. It is generally downloaded in the field to a laptop computer or another data storage device and later processed in the laboratory or office.
- Results of all valid precision, bias, and accuracy tests performed during the quarter.

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BIBLIOGRAPHY:

USEPA (U.S. Environmental Protection Agency). 1997. Quality Assurance Guidance Document 2.10, Monitoring PM10 in Ambient Air Using a Dichotomous Sampler. U.S. Environmental Protection Agency. Publication EPA-600/4-77-027a, USEPA National Exposure Research Laboratory, Research Triangle Park, NC. September 1997 Second Draft.

USEPA (U.S. Environmental Protection Agency). 1994. Quality Assurance Guidance Document Volume II Part II Ambient Air Specific Methods Section 2.11, Monitoring PM10 in Ambient Air Using a High Volume Sampler. U.S. Environmental Protection Agency. Publication EPA-600/R-9/038b, USEPA Office of Research and Development, Washington, DC. April 1994.

USEPA (U.S. Environmental Protection Agency). 1987. Reference Method for the Determination of Fine Particulate Matter as PM10 in the Atmosphere. U.S. Environmental Protection Agency. 40 CFR Part 50, Appendix J, August 7, 1987.

USEPA (U.S. Environmental Protection Agency). 2006a. Ambient air quality surveillance. U.S. Environmental Protection Agency. 40 CFR Part 58, as amended October 17, 2006.

USEPA (U.S. Environmental Protection Agency). 2006b. Ambient air quality monitoring methodology. U.S. Environmental Protection Agency. 40 CFR Part 58, Appendix C, as amended October 17, 2006.

USEPA (U.S. Environmental Protection Agency). 2006c. Ambient air monitoring reference and equivalent methods. U.S. Environmental Protection Agency. 40 CFR Part 53, as amended October 17, 2006.

Asbestos and other Elongated Mineral Particles

(Mary Goldade)

INTRODUCTION: Asbestos can be an important, but often overlooked, contaminant in mine life cycle analysis. This may be due to the long-standing, but incorrect belief that asbestos exposures of concern are limited to occupational exposures in asbestos mining, asbestos fabrication, and work with commercial asbestos products. An investigative news article, published in 1999 (Schneider, 1999) publicized the tragedy of asbestos exposures to residents of Libby, Montana, due to a nearby vermiculite mine and milling facility in which asbestos minerals were intergrown with the vermiculite. This situation highlighted cases of asbestos-related illnesses in non-occupational exposure settings. Since that time, conditions at Libby and other places throughout the United States and the world have highlighted the problem of non-occupational, environmental exposures to asbestos and some other elongate mineral particles.

The term “asbestos” is fraught with differing definitions amongst mineralogical, commercial, analytical, toxicological, and regulatory institutions. Classically, the definition of asbestos has been limited to a generic industrial-commercial term consisting of six commercial mineral types: chrysotile, actinolite, amosite, anthophyllite, crocidolite, and tremolite. In recent years, the term “elongate mineral particles,” or EMP, has evolved to represent an understanding that some asbestos-like minerals, not specifically regulated as “asbestos,” also may cause disease (National Institute for Occupational Safety and Health, 2011). Because asbestos/EMP is a mineral or a series of minerals, it is feasible that asbestos/EMP may be present in mining locations—areas

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where mineral excavation occurs. Thus, asbestos/EMP should be considered as a potential contaminant of concern when performing mine life cycle evaluations in appropriate geologic settings (Van Gosen, 2007).

As with any environmental sampling program, appropriate sample collection techniques must be applied to ensure proper characterization of the site conditions. Prior to sampling, a conceptual site model must be prepared that describes the site conditions and potential asbestos/EMP exposures to human and/or ecological receptors. Both the array of sampling locations and temporal sampling considerations are critical to success of the investigation. However, proper sampling design is not addressed here. Specific guidance for asbestos/EMP sampling and screening is given in *Framework for Investigating Asbestos-Contaminated Superfund Sites* (U.S. Environmental Protection Agency, 2008). Due to the complexities associated with scientifically sound asbestos/EMP sampling and analysis for environmental samples, designers are encouraged to discuss sampling and analysis needs and approach with the experts listed in that document (U.S. Environmental Protection Agency, 2008). Information presented here focuses on sampling asbestos/EMP for a variety of media that may be encountered during the mine life cycle analyses. For convenience, general information about asbestos/EMP sampling and analysis is summarized below. Guidelines specific to each medium (air, water, soil/bulk material and settled dust) are presented separately in the following sections.

GENERAL MEASUREMENT OBJECTIVES: Environmental asbestos/EMP samples are collected to identify the mineral type and to quantify the amount of asbestos/EMP fibers that are present under certain exposure scenarios, such as: air inhalation (respirable zone), inhalation of air above disturbed solid media, or water ingestion. In addition, it is often helpful to identify the asbestos/EMP present in the host solid media, such as soil, dust, rock, or other bulk materials. Sampling equipment and technique(s) for asbestos/EMP testing often differ amongst the media types. The media of interest, along with their respective sampling conditions, are described in the respective media-specific sections.

GENERAL SAMPLING EQUIPMENT/TECHNIQUE(S): Proper sampling equipment and techniques are a function of both the type of media to be sampled as well as the respective measurement objectives. Refer to the media-specific sections of this chapter for the appropriate sampling approach. It must be emphasized that whenever performing sampling and analysis for asbestos, proper health and safety precautions must be followed. Refer to OSHA (Occupational Safety and Health Administration) 29 CFR 1910.1001 (CFR, Code of Federal Regulations, 1986) for further details on required training, certification, and personal protective equipment.

GENERAL ANALYTICAL METHOD(S): Sampling and analysis methods to test environmental asbestos/EMP are cited in subsequent sections. Research continues towards the procedures of sampling and analyses of asbestos; also, several methods are currently under development and/or revision and rewrite, which are not available as of publication of this document. The reader is encouraged to look for more recent editions of the cited methods. As is true of the sampling approach, appropriate analytical methods

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are a function of both the type of media to be sampled as well as the respective measurement objectives. Most of the standard analyses for asbestos are performed using either light or electron microscopy techniques, which require analysts to view the samples and visually estimate the amount of asbestos/EMP present. Refer to the media-specific sections of this chapter for more information on the appropriate asbestos analytical method(s) for a particular medium.

GENERAL LIMITATIONS/INTERFERENCES: Traditional chemical and optical (microscopic) methods are similar in that accurate asbestos/EMP measurements depend on proper sample preparation and instrument operation, maintenance, and calibration. Although most chemical methods generate measurement readings via objective instrumental analysis, optical methods introduce a subjective component that is typically not present in traditional chemical analysis. That is, optical methods rely on visual observation and interpretation of prepared samples by a certified microscopist. Although microscopists are highly trained, the necessity for analysts to count and record visual observations of asbestos/EMP introduces subjectivity into the optical methods. Analyst fatigue becomes a factor in asbestos/EMP testing.

In addition, although the actual sample size may vary among microscopic techniques, the amount of sample used in microscopic analysis is usually very small, which may lead to bias resulting in inaccurate results (biased high or low). This is especially true if the sample is not completely homogenized. Method-specific guidance for asbestos sample preparation techniques are summarized here. Broad guidance for application of statistical sampling techniques to reduce sampling error is given in *Guidance on Choosing a Sampling Design for Environmental Data Collection* (U.S. Environmental Protection Agency, 2002).

GENERAL BIBLIOGRAPHY:

Code of Federal Regulations 1986. Asbestos in General Industry Standard. 29 Code of Federal Regulations Part 1910.1001. June 20, 1986.

Occupational Safety and Health Administration 1986. Asbestos in General Industry Standard. 29 Code of Federal Regulations Part 1910.1001. June 20, 1986.

Schneider, A. 1999. A Town Left to Die. Seattle Post-Intelligencer. November 18, 1999.

U.S. Environmental Protection Agency 2002. Guidance on Choosing a Sampling Design for Environmental Data Collection. **QA/G-5S.** EPA/240/R-02/005. December 2002.

U.S. Environmental Protection Agency 2003. Guidance for Geospatial Data Quality Assurance Project Plans (QA/G-5G). EPA/240/R-03/003. 2003.

U.S. Environmental Protection Agency 2006. Guidance on Systematic Planning using the Data Quality Objectives Process **QA/G-4.** EPA/240/B-06/001. February 2006.

U.S. Environmental Protection Agency, 2008, Framework for Investigating Asbestos-Contaminated Superfund Sites. Prepared by the Asbestos Committee Technical Review Workgroup of the Office of Solid Waste and Emergency Response. OSWER DIRECTIVE #9200.0-68. September 2008.

Van Gosen, B.S., 2007, The geology of asbestos in the United States and its practical applications: Environmental & Engineering Geoscience, v. 13, no. 1, p. 55-68.

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MEDIA: Air (Stationary, Personal and Activity-Based Sampling)

MEASUREMENT OBJECTIVES: Although emerging science in human health and ecological receptors reveals that air is not the only exposure pathway, the respiration pathway is still considered the primary route for asbestos/EMP exposure. There are three key forms of air sampling, each with different measurement objectives: (1) Stationary air samples measure asbestos/EMP levels in ambient air conditions, which can occur both indoors and outdoors. (2) Personal air samples generally refer to samples collected as part of a health and safety program for workers that handle asbestos/EMP, as prescribed by the Occupational Safety and Health Administration (OSHA) Asbestos Standard 1910.1001 (Code of Federal Regulations, 1986). Personal air samples are worn near the breathing zone of a worker to monitor the levels of asbestos/EMP to which workers are exposed. (3) Activity-based sampling air samples, similar to personal air samples, are collected in the breathing zone of a person wearing a sampler while they performs a disturbance activity that is representative of activities carried out by potential receptors (as examples, sweeping, digging, raking, driving recreational vehicles on unsurfaced roads). Air sample measurements report the number of asbestos/EMP particles present in a known volume of air (elongate structures per cubic centimeters, s/cm^3).

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): Asbestos/EMP in air is sampled using a pump to draw a known volume of air through a non-conductive, three-piece cassette holding a filter (U.S. Environmental Protection Agency, 1994). The solid particles present in the air are thereby trapped onto the filter surface. The filter commonly used for sample collection is a mixed-cellulose ester material containing small holes that allow the air to pass through the filter while trapping particles onto the filter. The holes, referred to as pores, are generally 0.45 μm or 0.8 μm in diameter. The volume of air drawn through the filter is the product of the pump air flow rate and the total time the pump is run. The analytical sensitivity achieved for a particular sample depends in part on the amount of air passed through the filter and on the amount of filter area examined under the microscope. Therefore, the larger the volume of air that is passed through the filter without overloading the filter with material, the better.

There are two general categories for air sample collection: stationary and breathing zone. Stationary air sample collection consists of static placement of the filter cassette and pump apparatus. To collect breathing zone samples (personal or activity-based samples), an individual wears both the pump and the filter cassette, with the opening of the filter cassette positioned near the mouth and nose. In addition to the U.S. Environmental Protection Agency Environmental Response Team methods for sampling stationary and personal air sample collection (U.S. Environmental Protection Agency, 1994), the National Institute of Occupational Safety and Health Method 7400 (National Institute for Occupational Safety and Health, 1994a) also describes personal air sampling. Specific guidance for activity-based air sampling is given in *Framework for Investigating Asbestos-Contaminated Superfund Sites* (U. S. Environmental Protection Agency 2008).

ANALYTICAL TECHNIQUE(S): The recommended analytical techniques vary for each measurement objective. At present, the most common methods used to analyze air samples for asbestos/EMP are phase contrast microscopy (PCM) followed by

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transmission election microscopy (TEM). Environmental samples—any air sample collected for purposes other than for OSHA health and safety regulations—are analyzed by TEM. In most cases, the air filter is prepared for examination without disturbing the particles deposited onto the filter surface. This is referred to as "direct analytical preparation." Whenever feasible, the analysis should be performed using direct analytical preparation via the modified International Organization for Standardization 10312 method (International Organization for Standardization, 1995). In cases where the amount of material collected on the filter is too large to allow for reliable examination, termed "overloaded," the material on the filter may be re-suspended in water; a portion of the water is then applied to a second filter. This is referred to as "indirect analytical preparation." The advantage of indirect preparation is the ability to adjust the loading on the secondary filter to an optimal level for examination. If indirect analytical preparation is necessary, modified procedure ISO 13704 (International Organization for Standardization, 1999) is used.

Transmission electron microscopy emits electrons that are focused using electromagnetic lenses to visualize the specimen. A fully informative asbestos/EMP TEM analysis includes characterization of the mineralogy, morphology (size and shape), and crystal structure of each elongate particle counted and recorded. Thus, an asbestos/EMP analysis must include information about a particle's length, width, aspect ratio, mineral type and diffraction pattern. Refer to the next section for more detail on particle counting protocols.

Particle Counting Protocols:

Given that the general approach to TEM analysis is reasonably consistent among the many published analytical methods, the associated literature includes a surprisingly large variation in particle counting protocols. Seemingly minor differences among the counting protocols can result in significant differences in the interpretation of analytical data. Differences include rules for when and how to ascribe particle counts for fibers, bundles of fibers, and complex structures. To avoid the pitfalls with the differences in particle counting protocols, use of a modified method for International Organization for Standardization (ISO 10312) is recommended for any TEM analysis of environmental asbestos samples. The key modification to the written ISO method pertains to the particle counting protocol for aspect ratio. That is, the analytical laboratory should be instructed to identify, count, and record particles as prescribed in the ISO method for the length, width, and chemical composition for all asbestos and EMP particles of length $\geq$0.5 μm, regardless of the particles width. In addition, the laboratory must be directed to count and record all particles with an **aspect ratio ≥3:1**. In brief, the reason for counting the shorter particles is for comparability with data measured by using the PCM method, which has less specificity and less sensitivity. In addition, this length and aspect ratio is recommended in the *Asbestos Fibers and Other Elongate Mineral Particles: State of the Science and Roadmap for Research* (National Institute for Occupational Safety and Health, 2011).

Analytical Sensitivity:

As with any other contaminant analysis, thoughtful consideration for assignment of appropriate target analytical sensitivity is paramount to obtaining usable data. Typically,

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better analytical sensitivities may be required for residential exposure situations than in occupational exposure settings due in part to longer exposure durations. The laboratory must be informed up front of the target analytical sensitivity for each sample delivery group. That is, analytical sensitivity is a function of the volume of sample collected during the sampling and the amount of the filter that is observed under the microscope during analysis.

The greater the volume collected, the smaller the area necessary to be viewed during analysis, while still achieving the target analytical sensitivity. This is important because the cost of TEM sample analysis is related to the amount of time an analyst spends viewing the sample.

Samples collected for the Occupational Safety and Health Administration's health and safety regulations use phase contrast microscopy (PCM) as the primary screening tool. For this microscope illumination technique, light passes through a particle, which becomes "out-of-phase" (phase contrast) when compared with light passing next to the particle. PCM uses this increased contrast to visualize particles that would otherwise be very difficult to observe under ordinary light microscopy. The limit of visual resolution using PCM is about 0.25 μm. Therefore, particles thinner than about 0.25 μm in width cannot be observed by PCM. PCM cannot discriminate the mineralogy of particles; therefore, confirmation analysis using TEM (National Institute for Occupational Safety and Health, 1994b) is performed, if the number of fibers counted per cm^2 by PCM is above the Occupational Safety and Health Administration's trigger for further analysis.

LIMITATIONS/INTERFERENCES:

Sampling: Care should be taken whenever possible to avoid filter overloading. Examples of situations that cause overloading are smoke, such as cigarette or fireplace smoke, and very dusty conditions. Weather conditions such as rain, foggy mist, or other precipitation may also impact the filter's integrity.

Indirect Analytical Preparation:

The main concern for indirect analytical preparation compared to direct analytical preparation is that suspension and sonication of asbestos/EMP particles in water may alter the physical state of some particles on the original air filter. For example, sonication may break apart particles or particle bundles that were originally intact, thereby potentially increasing the number of particles observed.

Limitations on PCM:

PCM uses morphology to discriminate particles. Therefore, information about a particle's mineralogy and crystal structure are not obtained using PCM. Consequently, particles that are counted by PCM may include a variety of non-asbestos minerals, which can also occur in the form of long, thin, fiber-like shapes, plus organic particles can be included, such as animal hair or carpet fibers. This is especially true in nonindustrial (environmental) settings. When these non-asbestos particles are present, PCM tends to overestimate the true concentration of asbestos present in the sample. Conversely, when very thin asbestos fibers are present, PCM can tend to underestimate the true asbestos content of a sample because particles that are thinner than 0.25 μm cannot be observed. For these reasons, TEM is the method of analytical choice for environmental air samples.

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DOCUMENTATION/DATA HANDLING: The U.S. Environmental Protection Agency has developed a standardized electronic reporting form for asbestos/EMP in air data entry, referred as the National Asbestos Data Entry Spreadsheet for TEM and PCM air testing. Analytical data are entered into the spreadsheet, which includes a report form. In addition to this documentation, the laboratory should provide copies of the following documentation for investigative and quality control samples: handwritten benchsheets, preparation logbook pages, internal chain-of-custody forms, and chain-of-custody forms for the samples.

BIBLIOGRAPHY:

Code of Federal Regulations, 1986, Asbestos in General Industry Standard. 29 Code of Federal Regulations Part 1910.1001. June 20, 1986.

International Organization for Standardization, 1995, Ambient air—Determination of asbestos fibres—Direct transfer transmission electron microscopy method. International Organization for Standardization. ISO 10312:1995.

International Organization for Standardization, 1999, Ambient air—Determination of asbestos fibres—Indirect transfer transmission electron microscopy method. International Organization for Standardization. ISO 13794:1999.

National Institute for Occupational Safety and Health, 1994a, Asbestos and Other Fibers by PCM, Issue 2. NIOSH Method 7400. NIOSH Manual of Analytical Methods, 4th edition. August 15, 1994.

National Institute for Occupational Safety and Health, 1994b, Asbestos by TEM, Issue 2. NIOSH Method 7402. NIOSH Manual of Analytical Methods, 4th edition. August 15, 1994.

National Institute for Occupational Safety and Health, 2011, Asbestos Fibers and Other Elongate Mineral Particles: State of the Science and Roadmap for Research, Revision. Department of Human Health Services Publication No. 2011–159, April 2011.

U.S. Environmental Protection Agency, 1994, Asbestos Sampling. ERT SOP #2015. Prepared by the USEPA Environmental Response Team. November 17, 1994.

U.S. Environmental Protection Agency, 2008, Framework for Investigating Asbestos-Contaminated Superfund Sites. Prepared by the Asbestos Committee Technical Review Workgroup of the Office of Solid Waste and Emergency Response. OSWER DIRECTIVE #9200.0-68. September 2008.

MEDIA: Water

MEASUREMENT OBJECTIVES: Water samples collected for asbestos/EMP testing are obtained predominantly from drinking water sources. The U.S. Environmental Protection Agency's maximum contaminant level for asbestos in drinking water is 10 million fibers greater than 10 μm in length per liter of water. However, in a mine life cycle setting there are likely to be other objectives for collecting water samples beyond regulatory requirements for drinking water. Sampling of surface water, ground water, wastewater, pore water, or other waters may be necessary to satisfy objectives, such as: characterizing the nature and extent of asbestos/EMP contamination

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in the waterways, asbestos/EMP source attribution, ecological receptor risk assessments, and studies. Water sample measurements for asbestos report the number and type of asbestos/EMP particles that are present within a known volume of water (for example, structures per liter (s/L)).

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): Waters sampled for asbestos/EMP are collected in a manner similar to that of any other chemical of concern. A representative water sample is collected in a sterilized, pre-cleaned, screw-capped flint glass or polyethylene container that can hold about 1 liter of liquid. A water sample collected for asbestos/EMP testing must be filtered at the analytical laboratory within 48 hours of sampling to avoid possible changes in the sample make-up due to microbial activity (U.S. Environmental Protection Agency, 1983a, 1983b).

ANALYTICAL TECHNIQUE(S): Water samples are analyzed using transmission electron microscopy (TEM). Upon receipt by the laboratory, all water samples must be filtered. In preparing for analysis, a known amount of the water sample is passed through a filter membrane (pore size 0.1 to 0.22 μm) (U.S. Environmental Protection Agency, 1994). That filter is then prepared for TEM analysis.

Transmission electron microscopy functions by emitting electrons that are focused using electromagnetic lenses to visualize the specimen. A fully informative asbestos/EMP TEM analysis requires characterization of the mineralogy, morphology (size and shape), and crystal structure for each particle counted. Thus, an asbestos/EMP analysis must include information about a particle's length, width, aspect ratio (length divided by width), chemical composition, and x-ray diffraction pattern. For drinking water samples, U.S. Environmental Protection Agency Method 100.2 is commonly used (U.S. Environmental Protection Agency, 1994). U.S. Environmental Protection Agency Method 100.2 is unique in that only fibers with length greater than 10 μm are counted and recorded. This is an important point, especially if water samples are collected for a different measurement objective than for drinking water regulations. That is, if the sampling goal is to characterize the nature and extent of asbestos/EMP levels in surface water, it may be appropriate to request an alternative particle counting protocol—modified International Organization for Standardization Method 10312—where particles of all lengths ≥0.5 μm and with an aspect ratio of ≥3:1 are counted and recorded. The general particle counting rules are outlined in the ISO 10312 method (International Organization for Standardization, 1995) with one exception. The exception is that regardless of the particle's width, the analytical laboratory must be instructed to identify, count, and record particles as prescribed in the ISO method for the length, width, and chemical composition for all asbestos and EMP particles of length ≥0.5 μm. In addition, the laboratory must be directed to count and record all particles with an **aspect ratio ≥3:1**. In brief, the reason for counting the shorter particles is for comparability with data measured by using the PCM method, which has less specificity and less sensitivity. In addition, this length and aspect ratio is recommended in the *Asbestos Fibers and Other Elongate Mineral Particles: State of the Science and Roadmap for Research* (National Institute for Occupational Safety and Health, 2011).

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LIMITATIONS/INTERFERENCES:

Sampling:

A significant concern for analysis of asbestos/EMP in water is "fiber loss" due adhesion to the sample container. Studies have shown that asbestos adheres to microbiological material present in unsterilized bottles, making those fibers unavailable for analysis and thereby "lost" (U.S. Environmental Protection Agency, 1983a). Similarly, organic material in the raw waters can also serve as a sink for asbestos/EMP (U.S. Environmental Protection Agency, 1983a), carrying mineral fibers down and out of the water. Some analytical preparation methods, such as treatment using ozonation, ultra-violet light, and sonication, reduce organic matter and liberate asbestos/EMP for easier TEM measurement.

Analysis:

U.S. Environmental Protection Agency Methods 100.1 and 100.2 for asbestos in drinking water report different units of measure than is typical for most asbestos/EMP testing methods. Only particles >10 μm in length are counted. If measurement objectives are not only drinking water regulations, consider requiring the analytical laboratory to perform a different particle counting protocol using modified ISO 10312.

DOCUMENTATION/DATA HANDLING: The U.S. Environmental Protection Agency has developed a standardized electronic reporting form for asbestos/EMP in water data entry, referred as the National Asbestos Data Entry Spreadsheet for TEM water testing. Analytical data are entered into the spreadsheet, which includes a report form. In addition to this documentation, the laboratory should provide copies of the following documentation for investigative and quality control samples: handwritten benchsheets, preparation logbook pages, internal chain-of-custody forms, and sample chain-of-custody forms.

BIBLIOGRAPHY:

International Organization for Standardization,1995, Ambient air—Determination of asbestos fibres—Direct transfer transmission electron microscopy method. International Organization for Standardization. ISO 10312:1995.

National Institute for Occupational Safety and Health, 2011, Asbestos Fibers and Other Elongate Mineral Particles: State of the Science and Roadmap for Research, Revision. Department of Human Health Services Publication No. 2011–159, April 2011.

U.S. Environmental Protection Agency, 1983a, Development of Improved Analytical Techniques for Determination of Asbestos in Water Samples. EPA/600/4-83/042. Prepared by E. Chatfield, M. Dillon, and W. Stott.

U.S. Environmental Protection Agency, 1983b, Analytical Method for Determination of Asbestos Fibers in Water. USEPA Method 100.1. EPA 600/4-83-043. September 1983. Prepared by E. Chatfield, M. Dillon.

U.S. Environmental Protection Agency, 1994, Determination of Asbestos Structures over 10μm in Length in Drinking Water. USEPA Method 100.2. June 1994. Prepared by K. Brackett, J. Millette.

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MEDIA: Soil and Bulk Material

MEASUREMENT OBJECTIVES: Generally, asbestos/EMP testing in soil and bulk materials is very useful for screening purposes. Soil and bulk material screening for moderate levels of asbestos/EMP is particularly useful in the early stages of mine-cycle investigations to identify and locate contamination or to guide more detailed future sampling efforts (U.S. Environmental Protection Agency, 2008a). Soil sample and bulk material measurements estimate the area fraction of asbestos/EMP present in a sample and are generally reported in units of area percent (%).

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S):

Soil

As with any soil sampling activity, obtaining accurate results for asbestos/EMP-contaminated soils requires representative sampling and proper homogenization. Sampling designs for asbestos/EMP in soil should emphasize representativeness to the intended measurement objective. That is, multi-point composite samples may be necessary represent the average asbestos/EMP levels in soil over the sampled area. Likewise, if the question to be answered is how much asbestos/EMP is available for air entrainment, a sample design might focus on the topmost portion of the surface soil (for example, 1 cm). General guidance for soil sampling techniques and sampling design are described in U.S. Environmental Protection Agency (2002, 2003).

Bulk Material

Bulk material is a "catch-all" category that generally includes National Emission Standards for Hazardous Air Pollutants (NESHAP)-defined asbestos-containing materials (ACMs). NESHAP-defined ACMs are any materials and/or products that contain greater than 1% asbestos by volume. Under NESHAP regulations, asbestos is currently defined as six commercial mineral types: chrysotile, actinolite, amosite, anthophyllite, crocidolite, and tremolite. This definition is expanded to other inorganic mineral fibers under other regulations, such as EPA's Comprehensive Environmental Response, Compensation, and Liability Act and the Clean Air Act. Examples of bulk material are building materials and building products, such as wall board, wall joint compound, vermiculite attic insulation, vermiculite, pipe insulation, acoustic insulation, roofing materials, cements, and mastics. Bulk sampling to support NESHAP regulations is clearly defined and will not be reiterated here. If a portion of the mine life cycle investigation falls under the purview of NESHAP, refer to the Code of Federal Regulations (Code of Federal Regulations, 1984). For bulk materials such as vermiculite-containing materials that are not regulated by the NESHAP regulations, sampling considerations (homogeneity and representativeness) are similar to those discussed for soil sampling and the definition of ACM do not apply. Refer to the *Framework for Investigating Asbestos-Contaminated Superfund Sites* for more information (U.S. Environmental Protection Agency 2008a).

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ANALYTICAL TECHNIQUE(S):

Soil Preparation

Soil is generally composed of particles that are heterogeneous in size and mineralogy; therefore, the distribution of asbestos/EMP particles within the soil may likewise be highly heterogeneous. Heterogeneity greatly increases the analytical variability of any sample analysis and reduces the certainty of the asbestos measurement. One of the special issues regarding soil samples undergoing asbestos/EMP testing is whether the sample should be ground prior to analysis. Grinding can substantially increase the homogeneity or representative character of the sample, and thus, increase the accuracy of any analytical procedure in terms of identifying the total fibers present in the soil sample. The downside to grinding is that the particle size distribution within a sample may be altered, thereby limiting the ability to draw conclusions about the amount of asbestos/EMP that is available for release into the air exposure pathway.

For screening purposes, a sample preparation step that includes drying and grinding of soil samples is often recommended (U.S. Environmental Protection Agency, 2008b, 2008c; California Air Resources Board, 1991) to obtain a more reproducible estimate of total asbestos/EMP present in the soil; however, the data user is cautioned that the ground sample may not represent the particle size distribution of the asbestos/EMP that may be entrained into air. Currently, research activities are underway to investigate this uncertainty.

Bulk Material Preparation

Bulk material samples often require sample preparation that is specialized for the particular product or material being tested. Typically, the asbestos/EMP is further manually separated from other non-asbestos materials. Portions of the separated sample may be analyzed by PLM and/or TEM. The details for sample preparation are given in the NESHAP regulations (Code of Federal Regulations, 1984), National Institute for Occupational Safety and Health Method 9002 (National Institute for Occupational Safety and Health, 1994), and USEPA 600/R-93/116 (U.S. Environmental Protection Agency, 1993). Vermiculite attic insulation requires special sample preparation, using a procedure that suspends the vermiculite attic insulation in water allowing the sample to separate using simple gravity or centrifugation (U.S. Environmental Protection Agency, 2004).

Soil & Bulk Material Analysis

It is noteworthy that few analytical methods have been validated specifically for the analysis of asbestos in soil, whereas multiple methods exist for analyzing commercial materials and products for asbestos content. This is due to the now outdated view that only commercial asbestos materials are of environmental concern. Soil and bulk material analytical methods use stereoscope microscopy followed by PLM. Both techniques are microscopy tools, which are based on the principle that light passing through a mineral particle allows the analysts to visualize and identify mineral particles. PLM utilizes polarization of the transmitted light to distinguish the mineralogy and, therefore, to classify different types of asbestos/EMP.

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Two different strategies are employed to quantify the amount of asbestos/EMP in a soil sample using PLM: (1) visual area estimation, and (2) point counting. In the visual area estimation approach, the microscopist examines several fields of view and estimates the area fraction comprised by asbestos/EMP particles compared to the total area of all particles observed in each field of view. Methods that employ visual area estimation are U.S. Environmental Protection Agency Method 600/R-93/116 (U.S. Environmental Protection Agency, 1993) and Libby Asbestos Method Standard Operating Procedure Libby-SRC-03 (U.S. Environmental Protection Agency, 2008c). For point counting, a grid is superimposed on each field of view through the microscope. If a particle lies on a grid node, it is classified either as asbestos/EMP or non-asbestos. After classifying a specified number of particles (usually 400), the area fraction is calculated. Methods that employ point counting are U.S. Environmental Protection Agency Method 600/R-93/116 (U.S. Environmental Protection Agency, 1993) and CARB 435 (California Air Resources Board, 1991). Both visual area estimation and point counting approaches result in an estimate of the asbestos/EMP present in the sample. For both quantification strategies, the detection limit of the method is about 0.2% and the practical quantitation limit is 1% or higher, providing that a reference standard material is used that consists of the asbestos/EMP present in the samples.

LIMITATIONS/INTERFERENCES:

Steps must be taken to reduce electrostatic effects when sampling soil for asbestos/EMP. Limit the use of plastic, whenever possible.

Screening Tool Only:
Soil and bulk material testing methods are valuable screening tools for identifying the presence of moderate levels of asbestos/EMP. However, polarized light microscopy (PLM) is neither sufficiently quantitative nor sensitive to confirm the absence of asbestos/EMP at levels below about 1%. Some programs have developed validated reference materials to improve the detection limit of this technique to about 0.2% (U.S. Environmental Protection Agency, 2008c). However, reportedly, laboratories have been citing lower practical quantitation limits and/or detection limits for soil (1% or higher and 0.2%, respectively) that cannot be validated. Readers are cautioned to require laboratories to maintain an inventory of validated/certified reference materials for the asbestos/EMP of interest at level commensurate with the laboratory's reporting level. And, the reference materials used must include the asbestos/EMP type found in the samples as not all asbestos/EMP looks and acts the same.

Lack of Soil-to-air or Bulk material-to-air Correlations:
Measurements in solid media do not directly relate to exposures in air; therefore, asbestos/EMP estimations in solid materials are excellent screening tools, but results must be used with caution when relating them for use in a risk assessment.

Visual Area Estimation vs. Point Counting:
Results reported for both methods of sample fiber analyses—visual area estimation and point counting methods—are given as a number. Often the visual area estimation

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value is either a whole number or a concentration range. In contrast, point counting values are often reported out to 2 decimal places, which implies a level of certainty not available. Both methods should be considered estimates and never precise to more than 1 decimal place. For levels of asbestos/EMP reported below 1%, in which validated reference materials were not used, the error bars around the reported value may be so large that only the presence of asbestos/EMP can be confirmed and not a precise quantity.

Use of Electron Microscopy:
TEM and scanning electron microscopy (SEM) are useful tools to identify the presence of asbestos/EMP, usually at levels lower than what can be measured by PLM. That is, TEM and SEM are valuable qualitative tools. However, no U.S. Environmental Protection Agency method validation is currently available for TEM and SEM methods to confirm their use as quantitative tools for ultra-low concentrations of asbestos/EMP.

DOCUMENTATION/DATA HANDLING: The U.S. Environmental Protection Agency has developed a standardized electronic reporting form for asbestos/EMP in soil data entry, referred as National Asbestos Data Entry Spreadsheet for PLM testing. Analytical data are entered into the spreadsheet, which also includes a report form. In addition to this documentation, the laboratory should provide copies of the following documentation for investigative and quality control samples: handwritten benchsheets, preparation logbook pages, internal chain-of-custody forms, and sample chain-of-custody forms.

BIBLIOGRAPHY:

California Air Resources Board,1991, Determination of Asbestos Content of Serpentine Aggregate. Method CARB 435. California Air Resources Board. June 5, 1991.

Code of Federal Regulations, 1984, National Emission Standards for Hazardous Air Pollutants. National Air Emission Standard for Asbestos. 40 CFR Part 61, Subpart M. Original Source: 49 FR 13661, Apr. 5, 1984. eCFR website: **Error! Hyperlink reference not valid.**

National Institute for Occupational Safety and Health, 1994, Asbestos (bulk) by PLM. NIOSH 9002. NIOSH Manual of Analytical Methods, 4th edition. August 15, 1994.

U.S. Environmental Protection Agency, 1993, Method for the Determination of Asbestos in Bulk Building Materials. EPA/600/R-93/116

U.S. Environmental Protection Agency, 2002, Guidance on Choosing a Sampling Design for Environmental Data Collection. QA/G-5S. EPA/240/R-02/005. December 2002.

U.S. Environmental Protection Agency, 2003, Guidance for Geospatial Data Quality Assurance Project Plans (QA/G-5G). EPA/240/R-03/003. 2003.

U.S. Environmental Protection Agency, 2004, Research Method for Sampling and Analysis of Fibrous Amphibole in Vermiculite Attic Insulation—Cincinnati Method, EPA 600/R-04/004. January 2004.

U.S. Environmental Protection Agency, 2008a, Framework for Investigating Asbestos-Contaminated Superfund Sites. Prepared by the Asbestos Committee Technical Review Workgroup of the Office of Solid Waste and Emergency Response. OSWER DIRECTIVE #9200.0-68. September 2008.

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U.S. Environmental Protection Agency, 2008b, Soil Sample Preparation. SOP ISSI-Libby-01 Revision 10. 2008.
U.S. Environmental Protection Agency, 2008c. Analysis of Asbestos Fibers in Soil by Polarized Light Microscopy. SOP Libby-SRC-03 Revision 2. October 10, 2008.

MEDIA: Settled Dust

MEASUREMENT OBJECTIVES: Generally, asbestos/EMP testing in settled dust may be useful for screening purposes, particularly if buildings or other structures are present onsite that will remain intact and/or be used in the future. In addition, settled dust samples may be useful in the early stages of mine-cycle investigations to identify and locate contamination or to guide more detailed future sampling efforts (U.S. Environmental Protection Agency, 2008a). Settled dust sample measurements estimate the number of asbestos/EMP particles present across an area sampled of known surface area (for example, structures per square centimeter, s/cm^2).

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): The term "settled dust" refers to accumulations of solid particles that have fallen out of the air (Millette and Hay, 1994). There are several methods available for measuring asbestos/EMP in dust such as: dust wipes, adhesive tape lifts, microvacuum samples, passive dust monitors, and bulk dust samples. However, for environmental sampling not under the purview of NESHAP, settled dust sampling is most often conducted using a "microvacuum." A microvacuum is a small air pump fitted with tubing that vacuums a defined area of a surface (Millette and Hay, 1994; American Society for Testing and Materials, 1995, 2003). The material picked up by the vacuum is captured onto a filter, housed in a three-piece cassette holding a filter. The filter commonly used for sample collection is a mixed-cellulose ester (MCE) material containing small holes to allow the air to pass through the filter while trapping particles onto the filter. The holes, referred to as pores, are generally 0.45 μm in diameter. Bulk dust samples (sometimes referred as "scrape and scoop" samples) should be treated in the same manner as bulk material, which is described in the previous section.

ANALYTICAL TECHNIQUE(S):

Settled Dust Sample Preparation

Because dust samples contain a much higher amount of debris than air samples, all dust samples are prepared for examination using the indirect analytical preparation technique (American Society for Testing and Materials, 1995, 2003). For indirect analytical preparation, the material on the filter is re-suspended in water and a portion of the water is then applied to a second filter. It is sometimes possible to concentrate asbestos/EMP fibers present on the filter by removing some of the interfering materials. This may be done by adding chemical reagents or ashing. In cases where the raw dust samples contain a large fraction of organic material, pet hair for example, ashing may provide a significant improvement in analytical sensitivity because more of the ashed sample may be applied to the secondary filter without overloading with irrelevant fibers. Chemical reagents, such as acids, can be added to remove inorganic materials and thereby further increase analytical sensitivity.

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Settled Dust Sample Analysis

Settled dust samples are most commonly analyzed by transmission electron microscopy (TEM). The analytical method referenced in the American Society for Testing and Materials methods (American Society for Testing and Materials, 1995, 2003) is the Asbestos Hazard Emergency Response Act particle counting protocol, which records numbers of particles ≥0.5 µm in length that also have an aspect ratio of ≥5:1 (Code of Federal Regulations, 1987). However, the counting rules of the Asbestos Hazard Emergency Response Act limits the usability of the data because the dimensions of individual particles are not captured, but instead binned into 2 categories: <5 µm and ≥5 µm. Data users may decide to collect and record the individual particle dimensions, and if so, the laboratory must be requested to follow specified particle counting rules, such as those outlined in method International Organization for Standardization 10312 (International Organization for Standardization, 1995); this modified combination of rules ensures that asbestos/EMP particles of aspect ratio ≥3:1 are counted and recorded.

For most situations analytical sensitivity of 500-5000 s/cm^2 is recommended, but this target sensitivity may change (increase or decrease) depending on the particular site conditions and measurement objectives. Always notify the laboratory of the required analytical sensitivity at the time of sample delivery to ensure that sufficient filter area is analyzed.

LIMITATIONS/INTERFERENCES:

Screening Tool:

It bears repeating here that settled dust testing is a valuable screening tool for locating the presence of asbestos/EMP, estimating the amount present, and guiding future sampling efforts (for example, activity-based sampling) (U.S. Environmental Protection Agency, 2008). Average asbestos levels for chrysotile found in urban environments are published in the literature (Millette and Hays, 1994). These measurement comparisons are useful guides, but must be used thoughtfully by considering their applicability to site conditions and data use. Considerations may include, but are not limited to, comparability of: asbestos/EMP mineral type, site conditions, likelihood that a "background" level of the mineral exists, potential exposure scenarios for current and/or future receptors, and other examples.

Lack of Soil-to-air or Bulk material-to-air Correlations:

Measurements in solid media do not directly relate to exposures in air; therefore, settled dust sample analyses are excellent screening tools, but results must be used with caution.

DOCUMENTATION/DATA HANDLING: The U.S. Environmental Protection Agency has developed a standardized electronic reporting form for asbestos/EMP in dust data entry, referred as National Asbestos Data Entry Spreadsheet. Analytical data are entered into the spreadsheet, and includes a report form. In addition to this documentation, the laboratory should provide copies of the following documentation

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for investigative and quality control samples: handwritten benchsheets, preparation logbook pages, internal chain-of-custody forms, and sample chain-of-custody forms.

BIBLIOGRAPHY:

American Society for Testing and Materials, 1995, ASTM Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading. ASTM D5755-95. ASTM Subcommittee D22.07.

American Society for Testing and Materials, 2003, ASTM Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading. ASTM D5755-03. ASTM Subcommittee D22.07.

Code of Federal Regulations, 1987, Asbestos Hazard Emergency Response Act. — Interim Transmission Electron Microscopy Analytical Methods—Mandatory and Nonmandatory—and Mandatory Section To Determine Completion of Response Actions. 40CFR Part 763 Subpart E, Appendix A. Original Source: 52 FR 41846, Oct. 30, 1987. eCFR website: http://ecfr.gpoaccess.gov, accessed 9/16/13.

International Organization for Standardization, 1995, Ambient air—Determination of asbestos fibres—Direct transfer transmission electron microscopy method. International Organization for Standardization. ISO 10312:1995.

Millette, J.R. and S.M. Hay, 1994, Settled Asbestos Dust Sampling and Analysis. 1st ed. Lewis Publishers of CRC Press LLC.

U.S. Environmental Protection Agency, 2008, Framework for Investigating Asbestos-Contaminated Superfund Sites. Prepared by the Asbestos Committee Technical Review Workgroup of the Office of Solid Waste and Emergency Response. OSWER DIRECTIVE #9200.0-68. September 2008.

Radon and Daughter Products

(Angelique D. Diaz)

MEDIA: Air

MEASUREMENT OBJECTIVES: The measurement of radon-222 (Rn-222) and radon daughters at underground mines is useful in determining (and preventing) exposure to both workers and the public. It is also necessary to show compliance with various regulatory requirements. This document serves as a general guide to radon and radon daughter monitoring methods.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): Radon-222 in the air is measured in picocuries per liter (pCi/L) and radon daughters are commonly measured in working levels (WL). Some methods for radon measurement are: scintillation cells, alpha track detectors, activated charcoal, and electret ion chambers. A common method for Rn-222 daughter measurement is to collect daughters from a known volume of air on a filter and then to analyze the alpha activity on the filter with a scintillation-type counter. Working levels are most often determined using the Kusnetz procedure (ANSI 1973, Kusnetz 1956).

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ANALYTICAL TECHNIQUE(S): Scintillation cells can be used as a continuous gas monitor for radon-222 (U.S. Environmental Protection Agency, 1989; 40 Code of Federal Regulations, 2012a). The decay of radon and radon daughters result in the production of light pulses which are amplified by a photo multiplier tube which generates electrical pulses. The electrical pulses are converted to pCi/L. Alpha track detectors act like photographic paper recording a microscopic "track" on the detector as alpha decay occurs (U.S. Environmental Protection Agency, 1989). The tracks are then quantified to determine the radon or radon daughter activity. Activated charcoal is used to adsorb radon-222 gas it comes in contact with (U.S. Environmental Protection Agency, 1989; Code of Federal Regulations, 2012b). The adsorbed radon undergoes radioactive decay and it is then analyzed in a laboratory for gamma rays, which are a result of the decay of the radon daughters. The gamma activity measured is used to determine the radon activity present on the charcoal. Electret ion chambers act as a capacitor and record the drop in voltage that occurs when ions produced by radioactive decay are drawn to the surface of the electret (U.S. Environmental Protection Agency, 1989; Code of Federal Regulations, 2012c). When calibrated correctly, the voltage drop can then be correlated to radon-222 activity.

LIMITATIONS/INTERFERENCES: The decay of radon and radon daughters can be measured using multiple methods. It is important that each method is understood so that you are aware of factors that may interfere with the analysis (e.g., dust and moisture) as well as lower and upper limits of detection. The equipment being used must be properly calibrated to the sampling conditions to account for limitations and interferences. Radon-220 (thoron) is another isotope of radon, produced by the radioactive decay of thorium-234. The gas has a half-life of approximately 56 seconds. There is a possibility that the presence of thoron can interfere with the Rn-222 measurement. You must be aware of how thoron should be treated based on the purpose of his measurements.

DOCUMENTATION/DATA HANDLING: During the analysis of your data, be aware that a background radiation level is present. It is important that you know the regulatory/procedure requirements on how to handle background radon and radon daughter values in your data analysis. The times associated with the radon analysis must be recorded so that the necessary corrections can be made for holding time.

BIBLIOGRAPHY:

40 Code of Federal Regulations (C.F.R.) Part 61 Appendix B, Method 114, 3.1.6, Method A-6, Radon-222 Continuous Gas Monitor (2012a)

40 Code of Federal Regulations (C.F.R.) Part 61 Appendix B, Method 114, 3.1.7, Method A-7, Radon-222 Alpha Track Detectors (2012b)

40 Code of Federal Regulations (C.F.R.) Part 61 Appendix B, Method 115, 2. *Radon-222 Emissions from uranium Mill Tailings Piles* (2012c)

Hartley, J.N. and Freeman, H.D. (1986), "Radon Flux Measurements on Gardinier and Royster Phosphogypsum Piles Near Tampa and Mulberry, Florida," U.S. Environmental Protection Agency Report, EPA 520/5-85-029.

Kusnetz, H. L., (1956), "Radon Daughters in Mine Atmospheres – A field Method for Determining Concentrations, " Am. Ind. Hyg. Assoc. J., Vol. 17:85-88.

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Salaymeh, S.R. (1999). Evaluation of Electret Ion Chambers for Measurement of Surface Alpha Contamination in Preparation for SRS-LSDDP. Westinghouse Savannah River Company, WSRC-MS-99-00332.
U.S. Environmental Protection Agency (1989), "Indoor Radon and Radon Decay Product Measurement Protocols," EPA 520/1-89-009.
http://epa.gov/radon/pubs/devprot4.html, accessed 8/23/13.

WATER-GENERAL INFORMATION AND MEASUREMENT TECHNIQUES

Groundwater

(Richard T. Wilkin and Carol C. Russell)

SAMPLING OBJECTIVES: The overall goal of any groundwater sampling program is to collect water samples from defined subsurface locations with no alteration in water chemistry. Monitoring objectives at ARD impacted sites are of three main types: detection, assessment, and remedial performance evaluation. Monitoring objectives may change as contamination or water quality problems are discovered. Initial groundwater monitoring objectives typically include consideration of a site conceptual model that incorporates local site descriptions and a hydrogeologic framework, collection of high-quality data, and subsequent refinement of initial conceptual models.

SPECIFIC SAMPLING EQUIPMENTS/TECHNIQUES: The disturbance of the subsurface environment as a result of well construction and sampling procedures presents serious obstacles to the interpretation of groundwater quality results (Backhus et al., 1993). Groundwater samples may be collected using a variety of methods: bailers, syringe samplers, suction-lift pumping, submersible pumps, and air-lift pumps (Parker, 1994). Waters from unsaturated zones may be collected using suction lysimeters or core squeezing methods. Selection of the appropriate technique(s) for any situation will depend on specific data quality objectives (DQOs). Low-flow purge methods are outlined here and are appropriate for groundwater monitoring at AMD impacted sites (Puls and Barcelona, 1991).

SPATIAL OR TEMPORAL CONSIDERATIONS: If sampling objectives include an assessment of the magnitude and extent of subsurface contamination over time and/or assessment of remedial performance, then information regarding plume delineation in three-dimensions is necessary. Information on groundwater flow velocity, direction, and vertical and horizontal variability are essential baseline data requirements for any subsurface assessment and monitoring program. For high-resolution sampling needs, screens less than 1 m should be used.

ANALYTICAL/SAMPLING TECHNIQUES: The objective of low-flow sampling techniques is to pump water in a manner that minimizes stress (drawdown, mobilization of particulates) to the system. Typically flow rates on the order of 0.1 to 0.5 mL/min are optimal. The flow rate used will rely on site-specific and well-specific parameters. In general, higher flow rates are appropriate in coarse-grained aquifers; aquifers containing

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fine-grained materials will require lower flow rates. Stabilization of parameters such as pH, specific conductance, concentration of dissolved oxygen, oxidation-reduction potential, and turbidity is appropriately used to determine when formation water is being sampled. If the sampling device is left in place (i.e., dedicated sampling systems), then the time and purge volume needed for stabilization is much shorter. Turbidity is generally the last parameter to stabilize. Ideally, turbidity will stabilize at a value <5 NTUs (nephelometric turbidity units); however, it should be noted that natural turbidity levels in groundwater may exceed 10 NTUs.

CONTAINER: For inorganic constituents including major cations and metals (unfiltered and filtered), plastic bottles are recommended. For anionic constituents, amber plastic bottles are recommended.

CONTAINER PRECLEANING: All sample containers should be either certified precleaned by the lab or with 5% HCl or HNO_3 (overnight soak), followed by three rinses with deionized (DI) water. Just prior to groundwater sample collection, all containers should be single rinsed with sample water. Special considerations for precleaning groundwater sample containers are described below:

- Sample containers intended for determination of anions must **not** be pre-cleaned with acid. Three rinses with DI water are typically not enough to remove residual chloride (Cl^-) or nitrate (NO_3^-). If the container is washed with metal-free, non-ionic detergent solution, well rinsed with tap water and three times with DI water, then in the field is rinsed three times with the sample, this is sufficient for major cations and major and minor anions.
- For trace metal analyses the sample container should be washed as above; rinsed with tap water; soaked in acid solution, then rinsed carefully with DI water. Then give the container a final rinse with 18 mega-ohm water (RO water).

PRESERVATION: For inorganic constituents including major cations and metals (unfiltered and filtered), concentrated nitric acid (HNO_3) is added to adjust the sample pH<2 (generally, 1 mL of concentrated HNO_3 per 100 mL of sample, but volume of acid necessary will depend on sample alkalinity). All samples should be kept refrigerated (4ºC±2°C) until the time of analysis. Sample bottles should be tightly capped to prevent leakage. In general, preservation is not necessary for samples on which major anions will be determined. If nitrate determination is needed and can't be done within 48 hours, a separate sample should be preserved with a small amount of chloroform. Samples for trace and ultra-trace metals determination should be acidified to pH=2 with trace-pure nitric acid. Hydrochloric acid (HCl) should not be used if flame atomic absorption spectroscopy (FAA), GFAA, ICP-AES or ICP-MS analytical techniques will be used.

HANDLING AND PREPARATION: Samples should be collected immediately after geochemical parameters have stabilized. Decisions to filter samples should be dictated by sampling objectives rather than to correct for poor sampling practices where excessive particulate loads are pumped to the surface (Puls and Barcelona, 1991). For assessment of truly dissolved (as opposed to operationally defined fractions, i.e., 0.45 micrometer filters), 0.1 micrometer filters are recommended although regulatory programs normally

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call for 0.45 micrometer filter papers (Kennedy et al., 1974). For assessment of potentially mobile contaminants, unfiltered groundwater samples should be collected. Although filtration may be appropriate, filtration may cause a number of unintended changes to occur (i.e., oxidation, adsorption) possibly leading to filtration-induced artifacts during sample analysis and uncertainty (Danielsson, 1982). In-line filtration is generally recommended because it provides better consistency through less sample handling and minimizes sample exposure to the atmosphere.

ADDITIONAL CONSIDERATIONS: The following are practical recommendations to be considered before, during, and after sampling (Puls and Barcelona, 1991):

- Use low-flow rates (<0.5 L/min) during purging and sampling to maintain minimal drawdown and disturbance;
- Maximize tubing wall thickness, minimize tubing length;
- Place the sampling device intake at the desired sampling point;
- Minimize disturbances of the stagnant water column above the screened interval during water level measurement;
- Make adjustments to stabilize flow rates as soon as possible after the initiation of pumping;
- Monitor water quality indicators during purging;
- Collect unfiltered samples to estimate contaminant loading and transport potential in the subsurface system.

BIBLIOGRAPHY:

Backhus, D.A., Ryan, J.N., Groher, D.M., MacFarlane, J.K., Gschwend, P.M. 1993. Sampling colloids and colloid-associated contaminants in ground water. *Ground Water* 31:466-479.

Danielsson, L.G. 1982. On the use of filters for distinguishing between dissolved and particulate fractions in natural waters. *Water Research* 16:179-182.

Kennedy, V.C., Zellweger, G.W., and Jones, B.F. 1974. Filter pore-size effects on the analysis of Al, Fe, Mn, and Ti in water. *Water Resources Research*: v. 10, p. 785-790.

Parker, L.V. 1994. The effects of ground water sampling devices on water quality: A literature review. *Ground Water Monitoring and Remediation* 14:130-141.

Puls, R.W., and Barcelona, M.J. 1991. Low-flow (minimal drawdown) ground-water sampling procedures. Ground Water Issue Paper, U. S. Environmental Protection Agency, 12 p., http://www.epa.gov/superfund/remedytech/tsp/download/lwflw2a.pdf, accessed 9/16/13.

Tremblay, G.A., and Hogan, C.M. (eds.) 2001. MEND Manual Volume 2—Sampling and Analysis. MEND document 5.4.2b. Ottawa, Ontario: Natural Resources Canada, 160 p. http://www.mend-nedem.org/reports/details-e.aspx?pub_id=5.4.2b. accessed 8/23/13.

USEPA (U.S. Environmental Protection Agency). 1989. *Ground-Water Monitoring in Karst Terranes*. EPA/600/X-89/050. Las Vegas, Nevada: Environmental Monitoring Systems Laboratory. http://www.epa.gov/swerust1/cat/gwkarst.pdf, accessed 8/23/13.

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USEPA (U.S. Environmental Protection Agency). 1994. *Ground Water Sampling—A Workshop Summary*. EPA/600/R-94/205. Ada, Oklahoma: Robert S. Kerr Environmental Research Laboratory. http://www.epa.gov/swerust1/cat/gwwkshop.pdf, accessed 8/23/13.

TABLE A3.1. Summary ground water sample collection methods (Tremblay and Hogan, 2001).

Type	Application	Limitations
Saturated zone		
Bailers	Simple, inexpensive method for sample collection.	Samples are disturbed and must be extensively handled during filtration. Can be time consuming in small diameter wells.
Depth-specific bailer	Simple, inexpensive method less disruptive than bailer.	Requires relatively large diameter wells.
Syringe sampler	Allows collection of samples without exposure to the atmosphere and allows filtration down-the-hole Can collect samples from any depth	Only small volumes of pre water are collected in each syringe Requires piezometers greater than 3cm in diameter
Suction-lift pump	For collection of relatively large volumes of water from shallow depth Permits use of inline filters to reduce sample handling	Sampling limited to water depth less than 8m
Submersible pumps	For collection of large volume samples from depths greater than 8m	Requires relatively large diameter wells. Costly
Air-lift	For collection of water samples from conventional wells or special air-lift samplers No limitation on depth of water	Samples can be highly disturbed. Will not work for volatile organics
Inertia pumping system (Waterra)	Low cost, unlimited depth, no power requirements, dedicated installment (no cross-contamination), direct filtering capabilities	
Unsaturated zone		
Suction lysimeters	For collection of pore water samples from unsaturated tailing solids samples	Reliable only in relatively coarse sandy materials
Core squeezing	For collection of pore water form tailings solids samples by squeezing or displacement	Destructive sampling, monitoring precisely the same location is not possible.

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Surface Water

(Kathleen S. Smith)

SAMPLING OBJECTIVES: The overall objective of surface-water sampling is to collect liquid samples that are representative of the constituents in the water body. No universal sampling procedure can be recommended because appropriate sampling techniques and objectives vary depending upon the specific study objectives.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): It is important to follow current, relevant sampling, preservation, and analytical protocols, to use consistent methodologies employing best-practice techniques and equipment, to be consistent in sampling locations and time of day, and to employ appropriate QA/QC. If samples must be collected from a boat, bridge, or cable, specialized sampling equipment may be required. Sampling devices must be constructed of the proper composition for the sampling application. For example, devices that are free of metal surfaces should be used for collecting samples for metal analyses.

Surface Water Sampling: Depending on surface-water conditions, several types of sampling devices exist with different uses and limitations (see U.S. Geological Survey, variously dated). Sampling devices that can be employed depending upon the sampling objectives include pumping systems, depth samplers, automatic sampling devices, continuous-flow samplers, and integrating samplers. Only experienced personnel should collect water samples. Field parameters (i.e., temperature, pH, specific conductance, dissolved oxygen) should be measured in the field, and samples should be filtered in the field. Surface-water samples should always be collected prior to sediment samples at the same location.

Stream Water Sampling: Considerations for stream-water sampling sites and techniques include sediment transport and mixing characteristics. Generally, it is important that the water being sampled is well mixed. In streams, it is good practice to collect depth-integrated samples that are collected at several verticals along a cross section of the flow so that the entire stream cross section is represented in the final sample (see U.S. Geological Survey, variously dated). Wading can cause resuspension of bottom sediment so samples should be collected while facing upstream.

SPATIAL OR TEMPORAL CONSIDERATIONS: Selection of sampling sites depends on the project objectives. Generally, criteria for selecting stream-water sampling sites include (1) fairly straight channel reach, (2) relatively uniform flow (i.e., not in rippled or sluggish areas), (3) condition of the streambed, (4) avoidance of confluences (to avoid poorly mixed flows or backwater effects), (5) location of point-source and nonpoint-source discharges, (6) avoidance of bridges, culverts, or roads (to avoid potential contamination), and (7) consideration of hydrologic interactions (see Winter et al., 1988). When defining contaminant inflows, care should be taken to collect samples that bracket the inflow and collection procedures should ensure good mixing and representation of the entire cross section of the stream. To ensure spatial and temporal comparability, samples need to be collected at the same cross section throughout a temporal period, and temporal samples need to be collected at the same time of day under the same light conditions because there can be considerable diurnal cycles in water chemistry. To account for temporal variability, nesting short-term studies within longer-

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term studies that encompass variable climatic and hydrologic conditions can provide insights about the importance of short-term diurnal cycles. It may be advantageous to obtain samples throughout the day and night to account for the actual 24-hour variation of constituent concentrations at a given site (see Nimick et al., 2011).

ANALYTICAL/SAMPLING TECHNIQUE(S): It is important to minimize disturbance of the sampling site when collecting surface-water samples. Whenever possible, it is useful to conduct stream-water flow or discharge measurements so that mass load computations can be made (see Kimball et al., 2010; see separate method on stream flow and discharge). Knowledge of meteorological and hydrological information can be useful in data interpretation (see separate method on meteorological data).

CONTAINER: For inorganic constituents, such as major cations and metals, use plastic or Teflon bottles with plastic or Teflon-lined caps. For nutrients use brown or amber plastic containers. For organic carbon use pre-baked amber glass bottles with Teflon-lined caps. For mercury samples use glass bottles with Teflon-lined caps.

CONTAINER PRECLEANING: Certified, pre-cleaned containers are available from supply companies. For inorganic cationic constituents, sample containers should be precleaned with 5% HCl or HNO_3 (overnight soak) followed by three rinses with deionized water. Containers should be rinsed with sample water prior to sample collection. For inorganic anionic constituents and nutrients, sample containers should be well-rinsed with deionized water, but should not be precleaned with acid (see separate method for groundwater for additional information). For organic carbon, glass containers should be baked at 475^{o}C for at least 8 hours, and no detergent should be used in precleaning. Sampling equipment should be decontaminated prior to the collection of additional samples. Powder-free gloves should be worn to minimize contamination. If possible, one member of the sampling team should take all the notes and photographs, fill out tags, etc., while the other members collect the samples (see main text for additional information).

PRESERVATION: It is important to communicate with the laboratory to ensure that adequate sample volumes are collected and that proper sample preservation is performed. For inorganic cationic constituents, it is common practice to add concentrated HNO_3 to the sample to bring the pH <2 (or 1 mL concentrated HNO_3 per 100 mL sample; more acid may be required for alkaline samples). For inorganic anionic constituents and for organic carbon, it is common practice to refrigerate samples (maintained at 4^{o}C). See separate groundwater method and main text for additional information.

HANDLING AND PREPARATION: It is good practice to use a sample splitter to divide the collected sample into subsamples. For inorganic constituents, filtration procedures and equipment vary depending on the objective and application (0.45 μm filter size is commonly used for regulatory purposes; 0.2 μm filter size is commonly used for biological purposes; various filter sizes or microfiltration is used for research applications). For organic carbon, silver or pre-baked glass-fiber filters can be used to collect samples.

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ADDITIONAL CONSIDERATIONS: Precautions must be taken to avoid contamination by sampling equipment or atmospheric particles, and sample bottles should be capped whenever possible. Document time of day, weather conditions, stream-flow conditions, and sampling equipment and techniques used. Take blank samples for cleaned bottles, filters, preservatives, and sampling procedures. Replicate samples can be used to assess sampling precision. Sample high-flow and transient hydrologic events to obtain an estimate of flushing of constituents from soils, mining wastes, hyporheic zones, etc. in a watershed.

BIBLIOGRAPHY:

COAG (Council of Australian Governments). 2000. *Australian Guidelines for Water Quality Monitoring and Reporting, Chapter 4. Field Sampling Program*. http://www.mincos.gov.au/__data/assets/pdf_file/0012/316110/mg-ch4.pdf. Accessed 8/23/13.

INAP (International Network for Acid Prevention). 2009. *Global Acid Rock Drainage Guide (GARD Guide)*. www.gardguide.com/. Accessed 8/23/13.

Kimball, B.A., Runkel, R.L., and Walton-Day, K. 2010. An approach to quantify sources, seasonal change, and biogeochemical processes affecting metal loading in streams: Facilitating decisions for remediation of mine drainage. *Appl. Geochem*. V. 25, p. 728-740.

Nimick, D.A., Gammons, C.H., and Parker, S.R. 2011. Diel biogeochemical processes and their effect on the aqueous chemistry of streams: A review. *Chem. Geol*. V. 283, p. 3-17.

Tremblay, G.A., and Hogan, C.M. (eds.). 2001a. *MEND Manual, Vol. 2—Sampling and Analysis*. MEND document 5.4.2b. Ottawa, Canada: Natural Resources Canada, Mine Environment Neutral Drainage (MEND) Program, 190 p., http://www.mend-nedem.org/reports/files/5.4.2b.pdf, accessed 9/16/13.

Tremblay, G.A., and Hogan, C.M. (eds.). 2001b. *MEND Manual, Vol. 6—Monitoring*. MEND document 5.4.2f. Ottawa, Canada: Natural Resources Canada, Mine Environment Neutral Drainage (MEND) Program, http://www.mend-nedem.org/reports/files/5.4.2f.pdf, accessed 9/16/13.

USGS (U.S. Geological Survey). Variously dated. *National Field Manual for the Collection of Water-Quality Data*. Techniques of Water-Resources Investigations, book 9, chaps. A1-A9. Reston, VA: U.S. Geological Survey. http://water.usgs.gov/owq/FieldManual/. Accessed 8/23/13.

Winter, T.C., Harvey, J.W., Franke, O.L., and Alley, W.M. 1998. *Ground Water and Surface Water—A Single Resource*. Circular 1139. Reston, VA: U.S. Geological Survey. http://water.usgs.gov/pubs/circ/circ1139. Accessed 8/23/13.

Hyporheic Flow And Groundwater/Surface-Water Interactions

(Kathleen S. Smith)

MEDIUM: Water (groundwater/surface-water interface)

SAMPLING OBJECTIVES: Understanding interactions between groundwater and surface water is an important, often overlooked consideration in water management.

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Winter et al. (1998) provide an overview of groundwater/surface-water interactions. Interactions at the groundwater/surface-water interface (the "hyporheic zone") can play an important role in the concentration and load of constituents and can have significant environmental influences on biogeochemical processes (Bencala, 2005). The hyporheic zone is "a subsurface flow path along which water 'recently' from the stream will mix with subsurface water to 'soon' return to the stream" (Harvey and Bencala, 1993). Another commonly cited definition is "the subsurface zone exchanging at least 10% stream water with subsurface water" (Triska et al., 1989). Gandy et al. (2007) provide a review of information about attenuation of mining-derived pollutants in the hyporheic zone. Important processes include sorption, precipitation, redox reactions, and reductive dissolution of iron and manganese oxyhydroxides, and increased contact of dissolved metals with reactive sediments and microbes can enhance metal attenuation in the hyporheic zone (e.g., Fuller and Harvey, 2000).

SPECIFIC SAMPLING EQUIPMENTS/TECHNIQUE(S): Sample collection at the groundwater/surface-water interface poses special problems. Groundwater may discharge at the surface in seeps and springs or in the subsurface (e.g., within a pond or lake). Groundwater discharges generally are not in equilibrium with surface conditions and it is common for groundwater to degas or form precipitates when it discharges at the surface. Sampling situations vary widely and no universal sampling procedures can be recommended (see U.S. Environmental Protection Agency, 1984; U.S. Geological Survey, variously dated). A primary consideration in sampling from springs and seeps is to minimize disturbance of solid materials that may become entrained in the sample matrix. Water samples may be collected by direct sampling or by using a variety of devices including dip samplers, Kemmerer bottles, syringe samplers, and peristaltic pumps. Where mechanical pumps are used to collect water samples, pumping rates should not exceed the point where fine-grained particulates are mobilized. To maintain the chemical integrity of the sample, flow-through cells may be used to minimize exposure to surface conditions.

Hyporheic water can be sampled using shallow piezometers or special sampling devices, such as a Minipoint sampler (Duff et al., 1998). The influence of hyporheic zone interactions can be quantified using tracer-injection studies. Hyporheic zones generally are characterized by steep concentration gradients of reactive constituents, dissolved oxygen, and pH. Selection of the appropriate technique(s) for any situation will depend on specific project objectives and on the hydrologic setting.

SPATIAL OR TEMPORAL CONSIDERATIONS: Because the rate of discharge into seeps and springs can vary widely, monitoring programs will need to be designed to capture seasonal effects. In addition to evaluating the changes of water quality with time, also important is measurement of the discharge rate as a function of time and space. Harvey and Fuller (1998) address the different spatial scales (from cm to km) related to hyporheic flow.

HANDLING AND PREPARATION: Samples should be collected immediately after geochemical parameters have stabilized. For inorganic constituents, filtration procedures and equipment will vary depending on the project objectives. Decisions to filter samples

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should be dictated by sampling objectives and not as a corrective measure for poor sampling practices that result in excessive particulate loads. For assessment of truly dissolved constituents, 0.1 micrometer filters or ultrafiltration are possibilities, although regulatory programs normally call for 0.45 micrometer filters (Kennedy et al., 1974). For assessment of potentially mobile contaminants, unfiltered groundwater samples should be collected. Although filtration may be appropriate, filtration may cause a number of unintended changes to occur (i.e., oxidation, adsorption) possibly leading to filtration-induced artifacts during sample analysis and uncertainty (Danielsson, 1982). In-line filtration generally is recommended because it provides better consistency through less sample handling and minimizes sample exposure to the atmosphere.

ADDITIONAL CONSIDERATIONS: The groundwater/surface-water interface is oftentimes coincident with a steep gradient in redox chemistry. Consequently, special precautions will often be required to preserve redox-sensitive species (e.g., arsenic speciation). Measurements may need to be carried out in the field immediately following sample collection. For example, the possible outgassing of CO_2 can affect alkalinity measurements.

BIBLIOGRAPHY:

Bencala, K.E. 2005. Hyporheic exchange flows. In *Encyclopedia of Hydrological Sciences*, Vol. 3(10). Edited by M.G. Anderson. New York: Wiley, p. 1–7. http://water.usgs.gov/nrp/proj.bib/Publications/2005/bencala.2005.pdf. Accessed 8/23/13.

Danielsson, L.G. 1982. On the use of filters for distinguishing between dissolved and particulate fractions in natural waters. *Water Res* 16:179–182.

Duff, J.H., Murphy, F., Fuller, C.C., Triska, F.J., Harvey, J.W., and Jackman, A.P. 1998. A mini drivepoint sampler for measuring pore water solute concentrations in the hyporheic zone of sand-bottom streams. *Limnol. Ocean*. 43:1378–1383.

Fuller, C.C., and Harvey, J.W. 2000. Reactive uptake of trace metals in the hyporheic zone of a mining-contaminated stream, Pinal Creek, Arizona. *Environ. Sci. Technol*. 34:1150–1155.

Gandy, C.J., Smith, J.W.N., and Jarvis, A.P. 2007. Attenuation of mining derived pollutants in the hyporheic zone: a review. *Sci. Total Environ*. 373:435–446.

Harvey, J.W., and Bencala, K.E. 1993. The effect of streambed topography on surface-subsurface water exchange in mountain catchments. *Water Res. Research*. 29:89–98.

Harvey, J.W., and Fuller, C.C. 1998. Effect of enhanced manganese oxidation in the hyporheic zone on basin-scale geochemical mass balance. *Water Res. Research* 34:623-636.

Kennedy, V.C., Jackman, A.P., Zand, S.M., Zellweger, G.W., and Avanzino, R.J. 1984. Transport and concentration controls for chloride, strontium, potassium and lead in Uvas Creek, a small cobble-bed stream in Santa Clara County, California, U.S.A., 1. Conceptual model. *J. Hydrol*. 75:67-110.

Triska, F.T., Kennedy, V.C., Avanzino, R.J., Zellweger, G.W., and Bencala, K.E. 1989. Retention and transport of nutrients in a third-order stream in northwestern California: Hyporheic processes. *Ecology* 70:1893–1905.

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USEPA (U.S. Environmental Protection Agency). 1984. *Characterization of Hazardous Waste Sites—A Methods Manual*. Vol. II. Available sampling methods, 2nd Ed.: U.S. Environmental Protection Agency, EPA/600/4-84-076.

USGS (U.S. Geological Survey). variously dated. *National Field Manual for the Collection of Water-Quality Data*. Techniques of Water-Resources Investigations, book 9, chaps. A1-A9: U.S. Geological Survey. http://pubs.water.usgs.gov/twri9A. Accessed 8/23/13.

Winter T.C., Harvey J.W., Franke O.L., and Alley W.M. 1998. Ground water and surface water—a single resource. Circular 1139. Reston, VA: U.S. Geological Survey, 79 p. http://water.usgs.gov/pubs/circ/circ1139. Accessed 8/23/13.

Automated Water Samplers

(Thomas P. Chapin)

PRINCIPLE: Automated water samplers are used to sample at frequencies that are impractical with hand sample collection. Commercially available portable automated water samplers have been successfully used for over two decades in mine impacted areas. These battery operated instruments typically are large (~size of a medium garbage can), heavy (>50 lbs.), difficult to conceal, and cost $2500-$4000. A microprocessor controls the sample timing, sample volume, and pump operation and twenty-four 500-1000 mL samples are collected in separate sample bottles. Stabilizing reagents can be added to the sample bottles beforehand and some degree of refrigeration can be attained by packing the sampler in ice. These -automated water samplers are placed on the shore or in an enclosure near a lake/stream and work well for short term (days) deployments (days-weeks) but are not well suited for freezing conditions or long-term deployments (Gammons et al., 2005).

High-sensitivity analytical instruments such as inductively coupled plasma mass spectrometry (ICP-MS) only require a few milliliters of sample per analysis so large sample volumes are often unnecessary. The USGS has developed a new small, light, low-cost, high-capacity, in-situ water sampler (MiniSipper) to overcome the limitations of currently available automated samplers. MiniSipper components are: 1) waterproof housing for microcontroller, micro-pumps, and rechargeable battery; 2) 150 m sample coil; and 3) collapsible bags for an inert gas (N_2) and a stabilizing reagent (HNO_3). The MiniSipper injects discrete or integrated samples (1-20 mL) into the sample coil. Adjacent samples are separated with a gas bubble and a stabilizing reagent can be added. Over 250 5-mL water samples can be collected. After recovery, the MiniSipper is returned to lab and samples are pumped out of the sample coil into sample vials. Samples are then analyzed by high sensitivity multi-element methods.

PRIMARY USES: Commercially available samplers are primarily used in short term field studies lasting a few days. The USGS MiniSipper was initially developed for long-term trace metal water sampling in acid mine drainage areas, but the MiniSipper can be used to sample other water borne constituents. The small size, long duration (>12 months), and in-situ deployment of the MiniSipper are ideal for sampling diel, episodic (storms or spring flush) or long-term (winter low-flow) events in hard to access areas. The MiniSipper can be deployed in freezing areas for over-winter studies, providing samples throughout the entire water year.

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LIMITATIONS: Commercially available portable automated water samplers are limited to collecting 24 samples and cannot easily be used in freezing conditions. These units are relatively large and heavy and difficult to conceal in remote areas. MiniSipper limitations are related to the small sample volumes and long deployment durations. Analytical methods requiring more than 20 mL sample volumes are not practical for MiniSipper samples. The long deployment duration of MiniSippers requires the analyte be stable over time or capable of stabilization with a reagent. Since MiniSipper samples are separated by gas bubbles; shaking or dropping the sample coil can mix up the samples. The MiniSipper can only be submerged to a depth of 1 meter.

SCOPE: Automated water samplers have been deployed in oceans, rivers and lakes to measure a wide range of ion concentrations. MiniSippers have successfully collected over-winter samples under surface ice.

SAMPLE REQUIREMENTS: MiniSipper sample volumes are determined by the sensitivity of the analytical detection method. Sample preservation is optimized for long-duration deployments.

DETECTION LIMITS: Detection limits depend on the analyte of interest and the analytical method used. For example ICP-MS methods have detection limits in the parts per trillion (ng/L) to low parts per billion (µg/L) range.

DATA FORMAT: Information is reported as concentration of analyte.

COST: Commercially available automated water samplers (Teledyne-Isco, Hach, Sigma, Sirco, etc.) cost between $2000 and $4000. The USGS MiniSipper is not commercially available. The raw components of the MiniSipper cost ~$2000 but must be assembled by a researcher with basic electronics and machining skills.

BIBLIOGRAPHY:

Chapin, T.P., and Todd, A.S. 2012. MiniSipper: A new in situ water sampler for high-resolution, long-duration acid mine drainage monitoring. *Science of the Total Environment* 439:343-353.

Gammons, C.H., Nimick, D.A., Parker, S.R., Cleasby, T.E., and McCleskey, R.B. 2005. Diel behavior of iron and other trace metals in a mountain stream with acidic to neutral pH: Fisher Creek, Montana, USA. *Geochim Cosmochim Acta* 69:2505–2516.

Sample Preservation

(Virginia T. McLemore)

PRESERVATION AND STORAGE

Most solid samples generally do not require any special preservation or handling procedures. Any exceptions will be noted in the appropriate SOPs written for the project. Table A3.2 summarizes some of the parameters, preservation methods, and holding times

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acceptable for water samples. Sample filtration of water samples may be required. More details are in specific collection and analytical sections of this appendix.

When in bear country, coolers should be avoided or covered up with blankets, since bears have been known to break into vehicles and attack the coolers looking for food.

TABLE A3.2. Sample preservation and holding times for water samples. Also see http://www.epa.gov/region6/qa/qadevtools/mod5_sops/sample_handling_preservation/r6sample_handling_tables.pdf (accessed 8/23/13) for more parameters.

Parameter	Field Analysis	Glass sample container	Polyethylene sample container	Preservation	Maximum storage/holding time
pH	X			unpreserved	6 hours
Electrical conductance	X			unpreserved	24 hours
Temperature	X				
Dissolved oxygen	X				
TOC					
Redox potential					
TDS		X	X	unpreserved	7 days
Acidity		X	X	unpreserved	24 hours
Alkalinity		X	X	unpreserved	24 hours
Calcium (Ca)			X	HNO3	6 months
Magnesium (Mg)			X	HNO3	6 months
Sodium (Na)			X	HNO3	6 months
Potassium (K)			X	HNO3	6 months
Chloride (Cl)		X	X	unpreserved	7 days
Sulfate(SO4)		X	X	unpreserved	7 days
Nitrate (NO3)		X	X	unpreserved	24 hours
Total iron (Fe)			X	HNO3	6 months
Ferrous Iron			X	HNO3	24 hours
Aluminum (Al)			X	HNO3	6 months
Arsenic (As)			X	HNO3	6 months
Cadmium (Cd)			X	HNO3	6 months
Copper (Cu)			X	HNO3	6 months
Lead (Pb)			X	HNO3	6 months
Nickel (Ni)			X	HNO3	6 months
Zinc (Zn)			X	HNO3	6 months
Mercury (Hg)		X		H2SO4+K2 Cr2O	28 days

SHIPPING

Samples collected are often shipped to the laboratory via a carrier or will be hand delivered to a specific site for preparation and analysis. If the samples are sent by common carrier, an air bill can be used. Receipts of air bills will be retained as part of the permanent chain of custody documentation.

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BIBLIOGRAPHY:

http://nepis.epa.gov/Exe/ZyNET.exe/30000QSA.TXT?ZyActionD=ZyDocument&Client=EPA&Index=1981+Thru+1985&Docs=&Query=&Time=&EndTime=&SearchMethod=1&TocRestrict=n&Toc=&TocEntry=&QField=&QFieldYear=&QFieldMonth=&QFieldDay=&IntQFieldOp=0&ExtQFieldOp=0&XmlQuery=&File=D%3A\zyfiles\Index%20Data\81thru85\Txt\00000001\30000QSA.txt&User=ANONYMOUS&Password=anonymous&SortMethod=h|-&MaximumDocuments=1&FuzzyDegree=0&ImageQuality=r75g8/r75g8/x150y150g16/i425&Display=p|f&DefSeekPage=x&SearchBack=ZyActionL&Back=ZyActionS&BackDesc=Results%20page&MaximumPages=1&ZyEntry=1&SeekPage=x&ZyPURL#

http://sisbl.uga.edu/epatab1.html

http://www.epa.gov/region6/qa/qadevtools/mod5_sops/sample_handling_preservation/r6sample_handling_tables.pdf, accessed 8/23/13.

http://enterprise.astm.org/filtrexx40.cgi?+REDLINE_PAGES/D6517.htm, accessed 8/23/13.

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Alkalinity and Acidity
(Kathleen S. Smith)

MEDIA: Water

MEASUREMENT OBJECTIVES: Alkalinity is a measure of the capacity of water to neutralize acid and is one of the best water measurements to evaluate the sensitivity of a water body to acidic inputs. Alkalinity is commonly used to check the charge balance of a water sample. Levels of acidity or alkalinity are important in determining metal solubility and toxicity. Alkalinity in water can be influenced by underlying rock, soil, wastewater discharges, and input of alkaline substances (e.g., carbonate, bicarbonate, hydroxide, phosphate, silicate, borate, ammonia, organic ligands). To determine total alkalinity, a strong acid (generally sulfuric acid) is incrementally added to a water sample until all the alkalinity is consumed. In most natural water samples, alkalinity is primarily due to the presence of carbonate (CO_3^{2-}) and bicarbonate (HCO_3^-). Figure A3.4 shows how bicarbonate and carbonate in water vary with pH during a titration.

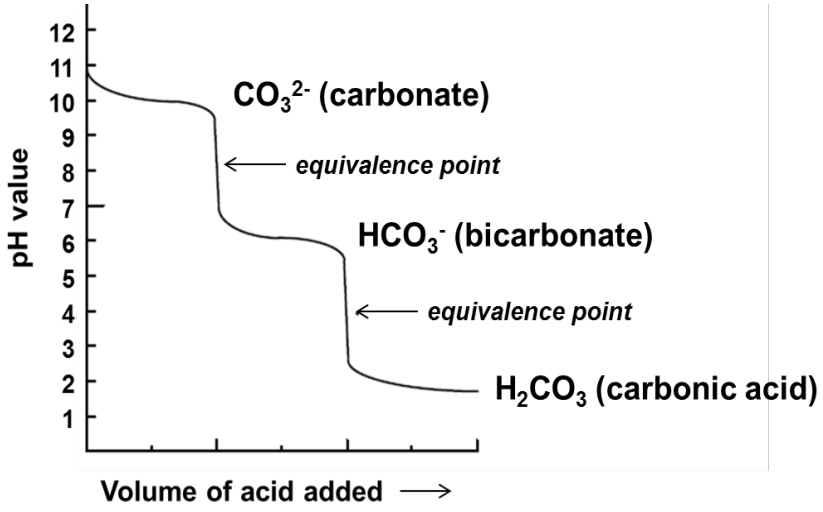


FIGURE A3.4 Titration of carbonate-rich water with sulfuric acid.

In contrast, acidity is measured by determining the amount of a strong base added to a water sample necessary to consume the acidity. The amount of acid or base used in the titration corresponds to the total alkalinity or acidity, respectively, of the water sample. Alkalinity and acidity can be measured in the field or in the laboratory. Alkalinity is sometimes referred to as "acid neutralizing capacity" or ANC. Nomenclature used by the U.S. Geological Survey specifies that alkalinity samples are filtered whereas ANC samples are not filtered prior to titration. Alkalinity and acidity generally are reported as mg $CaCO_3$/L. Net alkalinity (defined as the difference between alkalinity and acidity) and net acidity (defined as the difference between acidity and alkalinity) are common terms in mining applications to determine whether water requires treatment. Kirby and Cravotta (2005) discuss problems with definition and determination of net alkalinity and net acidity.

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SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): Titration equipment is necessary to measure both alkalinity and acidity. Titration is the incremental addition of small, precise quantities of acid or base to the water sample. In the laboratory, burettes are commonly used for titrations. If conducting measurements in the field, digital titrators are more robust than burettes. Field kits also are available, but are less accurate than using a digital titrator. It is helpful to have a pH electrode and meter to determine pH values during the titration, and necessary if the water sample is turbid. A stirring mechanism during titration is suggested to obtain a uniform mixture of sample and titrant. Generally, a 50-mL water sample in a 100-mL beaker is used for the titrations.

ANALYTICAL TECHNIQUE(S): In addition to manual titration methods using burettes or digital titrators, automatic titrators are available for alkalinity and acidity titrations. Burettes generally yield better accuracy than digital titrators. Acids and bases used for titrations must be manually standardized; as an alternative, known (standardized) concentrations of acids and bases appropriate for use in titrations can be purchased. Procedures include inflection point titration (IPT) and Gran plot method (see various references below). Titration to a fixed endpoint pH is an outdated method that is less accurate than the IPT or Gran methods. The IPT method is appropriate for most natural water. The Gran plot method is recommended for low-alkalinity (<20 mg $CaCO_3$/L) or low conductivity (<100 µS/cm) water (Rounds, 2006). Greater accuracy can be achieved by using a lower-concentration titrant, decreasing the volume of the titrant increments, or using a larger sample volume. For the IPT method it is important to carefully add small increments of titrant when approaching the equivalence point(s) because of the large pH changes near the equivalence point(s) (see Figure A3.4). For the Gran method it is important to collect data between and beyond the expected equivalence point(s) because the Gran method requires data that are removed from the equivalence point(s). Hot acidity is determined by measuring the pH of the sample and adding a measured amount of standard acid necessary to lower the pH to a value of 4 or less. Hydrogen peroxide is then added and the solution is boiled, cooled, and titrated with a standard base to pH 8.2 (U.S. Environmental Protection Agency, 1983a).

LIMITATIONS/INTERFERENCES: The measured value depends upon the method used. It is essential to specify the method, filtration, and, if appropriate, the endpoint pH when reporting alkalinity and acidity data. Interferences can include soaps, oily matter, suspended solids, and precipitates that can coat the pH electrode. Dissolved organic carbon can significantly contribute to total alkalinity in organic-rich systems. Aeration of a sample not equilibrated with the atmosphere can cause mineral precipitation, which will alter the alkalinity determination. The pH value of near-neutral pH, metal-laden water that possesses considerable alkalinity and measurable acidity can increase or decrease upon equilibration with the atmosphere, which presents analytical difficulties for acidity and alkalinity determinations (Kirby and Cravotta, 2005). If a water has been subjected to a condition where CO_2 gas is above atmospheric levels, then alkalinity will change as the excess CO_2 exsolves. Generally, the endpoint for alkalinity is well defined (see Figure A3.4). However, the pH endpoint for acidity often depends on the objectives of the study.

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BIBLIOGRAPHY:

ASTM D1067. 2006. *Standard Test Methods for Acidity or Alkalinity of Water*. West Conshohocken, PA: ASTM International. Available from www.astm.org, accessed 8/23/13.

Eaton, A.D., Clesceri , L.S., Rice, E.W., Greenberg, A.E., and Franson, M.A.H. (eds.). 2005. *Standard Methods for the Examination of Water and Wastewater*, 21st ed. Washington, DC: American Public Health Association, American Water Works Association, and Water Environment Federation.

Kirby, C.S., and Cravotta, C.A., III. 2005. Net alkalinity and net acidity 2: Practical considerations: *Appl. Geochem*., 20:1941–1964.

Rounds, S.A. 2006. *Alkalinity and Acid Neutralizing Capacity (version 3.0)*. Techniques of Water-Resources Investigations, book 9, chap. A6., section 6.6. Reston, VA: U.S. Geological Survey. http://water.usgs.gov/owq/FieldManual/Chapter6/section6.6/. Accessed 8/23/13.

USEPA (U.S. Environmental Protection Agency). 1983a. *Method 305.1, Acidity (Titrimetric), in Methods for Chemical Analysis of Water and Wastes:* U.S. Environmental Protection Agency EPA/600/4-79-020.

USEPA (U.S. Environmental Protection Agency). 1983b. *Method 310.1, Alkalinity (Titrimetric), in Methods for Chemical Analysis of Water and Wastes*: U.S. Environmental Protection Agency EPA/600/4-79-020.

Dissolved Inorganic Carbon (DIC)

(Kathleen S. Smith)

MEDIA: Water

MEASUREMENT OBJECTIVES: Dissolved inorganic carbon (DIC) is the sum of inorganic species in a solution and includes carbonic acid (including dissolved carbon dioxide; H_2CO_3), bicarbonate (HCO_3^-), and carbonate (CO_3^{2-}). Each of these species is related by the following pH-dependent equilibria:

$$CO_2 + H_2O \leftrightarrow H_2CO_3 \leftrightarrow HCO_3^- + H^+ \leftrightarrow CO_3^{2-} + 2H^+$$

DIC is the major carbon reservoir in the ocean. Determining inorganic carbon species is important in understanding carbon cycling and tracking the impact of changing levels of carbon dioxide. Photosynthesis (the organic carbon pump) depends on the availability of carbon dioxide according to the equation:

$$CO_2 + H_2O \rightarrow CH_2O + O_2$$

The carbonate "counterpump" relies on production of $CaCO_3$, which releases CO_2 according to the equation:

$$Ca^{2+} + 2HCO_3^- \rightarrow CaCO_3 + CO_2 + H_2O$$

In freshwater environments DIC is produced primarily by weathering of carbonate and silicate rocks and respiration in soils. In study of a lake system, CO_2 availability was shown to vary on diel, episodic, and seasonal scales coupled with significant depletions in DIC (Maberly, 1996). Stable isotopic composition of DIC has been used as a tracer of

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stream flow generation, groundwater inflow, and carbon dynamics in watersheds (e.g., Doctor et al., 2008).

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): Care must be taken to avoid exchange of CO_2 with the atmosphere during sampling. Water samples for DIC analysis should be collected in gas-tight containers (commonly pre-cleaned borosilicate test tubes or bottles with septa caps) and refrigerated in the dark until analysis. The DIC sample containers should be completely filled, minimizing turbulence, and tightly capped so that no headspace or bubbles remain. It is general practice to use mercuric chloride as a preservative for DIC samples in order to prevent biological activity. However, Winslow et al. (2001) demonstrated that sample preservation with copper sulfate is as good as that with mercuric chloride. Filtration details depend on the objectives of the study.

ANALYTICAL TECHNIQUE(S): Most DIC determinations are made using a commercially-available analyzer. A common analytical method is to dispense a known amount of sample into a stripping chamber where it is acidified and purged with CO_2-free gas. The acidification causes the inorganic carbon in the sample to be evolved as CO_2. The amount of CO_2 in the gas stream is determined by trapping the CO_2 in an absorbent containing ethanolamine and titrating coulometrically the hydroxyethylcarbamic acid that is formed. Johnson et al. (1993) developed a system called the Single Operator Multi-parameter Metabolic Analyzer (SOMMA) to interface with a coulometer to make high-precision measurements. Another common detection technique for CO_2 is by infrared spectroscopy.

LIMITATIONS/INTERFERENCES: The presence of solid carbonates interferes with the determination; particulates can be removed by filtration. For samples that contain hydrogen sulfide, a chemical scrubber should be used.

DOCUMENTATION/DATA HANDLING: It is good practice to document background concentrations and blanks by analyzing a laboratory reagent blank and various procedural blanks. Instrument calibration and standardization should be documented for each set of analyses. Use of certified reference materials and sampling replicates help ensure the accuracy of the measurements.

BIBLIOGRAPHY:

ASTM D513. 2006. *Standard Test Methods for Total and Dissolved Carbon Dioxide in Water*. West Conshohocken, PA: ASTM International. Available from www.astm.org. Accessed 8/23/13.

ASTM D5904. 2009. *Standard Test Method for Total Carbon, Inorganic Carbon, and Organic Carbon in Water by Ultraviolet, Persulfate Oxidation, and Membrane Conductivity Detection*. West Conshohocken, PA: ASTM International. Available from www.astm.org. Accessed 8/23/13.

ASTM D6317. 2009. *Standard Test Method for Low Level Determination of Total Carbon, Inorganic Carbon and Organic Carbon in Water by Ultraviolet, Persulfate Oxidation, and Membrane Conductivity Detection*. West

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Conshohocken, PA: ASTM International. Available from www.astm.org. Accessed 8/23/13.
Dickson, A.G., Sabine, C.L., and Christian, J.R. (eds.). 2007. *Guide to Best Practices for Ocean CO_2 Measurements*. PICES Special Publication 3, 191 p.
Doctor, D.H., Kendall, C., Sebestyen, S.D., Shanley, J.B., Ohte, N., and Boyer, E.W. 2008. Carbon isotope fractionation of dissolved inorganic carbon (DIC) due to outgassing of carbon dioxide from a headwater stream. *Hydrol. Proc.* 22:2410–2423.
DOE (U.S. Department of Energy). 1994. *Handbook of Methods for the Analysis of the Various Parameters of the Carbon Dioxide System in Sea Water*, version 2.1. In A.G. Dickson and C. Goyet (eds.). ORNL/CDIAC-74. http://cdiac.ornl.gov/oceans/DOE_94.pdf. Accessed 8/23/13.
Fonyuy, E.W., and Atekwana, E.A. 2008. Effects of acid mine drainage on dissolved inorganic carbon and stable carbon isotopes in receiving streams. *Appl. Geochem*. 23:743–764.
Fonyuy, E.W., and Atekwana, E.A. 2008. Dissolved inorganic carbon evolution and stable carbon isotope fractionation in acid mine drainage contaminated streams: Insights from a laboratory study. *Appl. Geochem*. 23:2634–2648.
Johnson, K.M., Wills, K.D., Butler, D.B., Johnson, W.K., and Wong, C.S. 1993. Coulometric total carbon dioxide analysis for marine studies: maximizing the performance of an automated continuous gas extraction system and coulometric detector. *Mar. Chem*. 44:167–187.
Maberly, S.C. 1996. Diel, episodic and seasonal changes in pH and concentration of inorganic carbon in a productive lake. *Freshwat. Biol*. 35:579-598.
Winslow, S.D., Pepich, B.V., Bassett, M.V., Wendelken, S.C., Munch, D.J., and Sinclair, J.L. 2001. Microbial inhibitors for USEPA drinking water methods for the determination of organic compounds. *Environ. Sci. Technol*. 35:4103–4110.

Dissolved Organic Carbon (DOC)

(Kathleen S. Smith)

MEDIA: Water

MEASUREMENT OBJECTIVES: Dissolved organic carbon (DOC) represents a broad range of organic molecules of varied origin and composition. DOC is determined by first removing inorganic carbon from the sample, and then oxidizing the remaining carbon in the sample and measuring the resulting carbon dioxide (CO_2). DOC is operationally defined by the filtration method used. DOC can be important in the transport and bioavailability of metals in aquatic systems because many metals form strong complexes with DOC, which enhances the metal solubility and reduces the metal bioavailability.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): DOC must be sampled using equipment that does not contribute any organic material to the sample. Samples must be stored in glass (preferably amber) bottles that have been baked at 475°C for at least 8 hours (cool to room temperature before use). The sample bottle should have

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minimal headspace, and the portion of the bottle cap that is in contact with the sample should be coated with Teflon. Do not rinse the baked DOC bottle. Samples need to be refrigerated and stored at or below 4°C without freezing. Samples should be analyzed as soon as possible (28 days is the maximum holding time suggested by the USEPA). Generally, DOC samples are preserved with 1 mL of concentrated phosphoric acid (H_3PO_4) per liter of sample to keep the pH of the sample at or below pH 2. Sulfuric acid can also be used as a preservative. When checking pH, do not place pH paper in the sample bottle as this may introduce contamination; instead, place a drop of the acidified sample onto pH paper. DOC requires filtration through either baked glass microfiber filters (GF/F; generally 1 to 7 μm) or through 0.45 μm disposable capsule membrane filters (e.g., hydrophilic polyethersulfone (PES) or hydrophilic polypropylene; see Karanfil et al. (2003) for detailed information about filter types). Any sample to be analyzed for total particulate carbon must be filtered through a GF/F filter. Filtration blank samples using organic-grade laboratory reagent water should be collected to test for collection procedure contamination and to define the background concentration. The organic-grade laboratory reagent water should be analyzed for organic carbon prior to use in the field. No organic solvents (including methanol) should be used to clean DOC-related equipment. If it is necessary to use soap, then the equipment should be rinsed copiously and a blank should be collected to ensure thorough removal of the soap. When cleaning equipment, it is good practice to wear disposable, powderless gloves and wash everything 3 times with tap water, followed by 3 times with deionized water (or nanopure water), and air drying equipment that cannot be baked.

Specific Ultraviolet Absorbance (SUVA) determinations provide a measure of DOC aromatic content. For $SUVA_{254}$ determinations the sample is transferred into a quartz cell and placed into a spectrophotometer to measure UV absorbance at 254 nm. The $SUVA_{254}$ is calculated by dividing the UV absorbance by the DOC of the sample, and then multiplying by 100. Samples collected for $SUVA_{254}$ determinations are not acidified at the time of collection and should be analyzed within 48 hours of collection. Spencer et al. (2007) discuss effects of pH and freeze/thaw on absorbance properties of DOC.

ANALYTICAL TECHNIQUE(S): For DOC analysis, the organic carbon in the water sample is oxidized to produce CO_2, which is then measured by a detection system. Techniques to oxidize organic carbon include high-temperature combustion (typically 680°C), ultra-violet (UV)-promoted photo-oxidation, and oxidation using persulfate solution. Prior to analysis for organic carbon the sample is acidified and the inorganic carbon is removed by sparging the sample with nitrogen or ultra-pure air. Organic carbon measurements generally are based on calibration with potassium hydrogen phthalate (KHP) standards.

LIMITATIONS/INTERFERENCES: Inorganic carbon interferes with DOC analysis; inorganic carbon is removed during the initial stages of the analytical procedure. Low results may be obtained for samples containing highly volatile organic compounds. Background levels of organic carbon contamination are problematic, and the minimum reporting level depends on the analytical instrument precision and the purity of the organic-grade laboratory reagent water. Values less than about 0.1 mg/L can be quite

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variable. Laboratories located in areas that contain organic aerosols, dust, etc. should use filtered air, and all glassware must be meticulously cleaned. Analysis of samples that contain high concentrations of organic carbon (>10 mg/L) can cause contamination of the instrument system; it is recommended that samples with high concentrations be diluted prior to analysis. Aquatic humic substances precipitate at pH values below 2. If aquatic humic substances precipitate in the sample, it is suggested to (1) shake well and split the sample into two portions, (2) raise the pH in the second portion to pH 5 to 7 to dissolve the humic substances, and (3) analyze both portions. Efficiency of oxidation can be problematic. High chloride concentrations (> 700 mg/L) may interfere with persulfate oxidation methods.

DOCUMENTATION/DATA HANDLING: It is good practice to document background concentrations and blanks by analyzing a laboratory reagent blank and various procedural blanks. Instrument calibration and standardization should be documented for each set of analyses.

BIBLIOGRAPHY:

Aiken, G., Kaplan, L.A., and Weishaar, J. 2002. Assessment of relative accuracy in the determination of organic matter concentrations in aquatic systems. *J. Environ. Monit.* 4:70–74.

ASTM D5904. 2009. *Standard Test Method for Total Carbon, Inorganic Carbon, and Organic Carbon in Water by Ultraviolet, Persulfate Oxidation, and Membrane Conductivity Detection*. West Conshohocken, PA: ASTM International. Available from www.astm.org. Accessed 8/23/13.

ASTM D6317. 2009. *Standard Test Method for Low Level Determination of Total Carbon, Inorganic Carbon and Organic Carbon in Water by Ultraviolet, Persulfate Oxidation, and Membrane Conductivity Detection*. West Conshohocken, PA: ASTM International. Available from www.astm.org. Accessed 8/23/13.

Karanfil, T., Erdogan, I., and Schlautman, M.A. 2003. Selecting filter membranes for measuring DOC and UV_{254}. *J. Am. Water Works Assoc.* 95:86–100.

Spencer, R.G.M., Bolton, L., and Baker, A. 2007. Freeze/thaw and pH effects on freshwater dissolved organic matter fluorescence and absorbance properties from a number of UK locations. *Water Res*. 41:2941-2950.

Urbansky, E.T. 2001. Total organic carbon analyzers as tools for measuring carbonaceous matter in natural waters. *J. Environ. Monit.* 3:102-112.

USEPA (U.S. Environmental Protection Agency). 2009. *Method 415.3-Determination of Total Organic Carbon and Specific UV Absorbance at 254 nm in Source Water and Drinking Water*, revision 1.2 (Sept. 2009). U.S. Environmental Protection Agency Office of Research and Development, National Exposure Research Laboratory, EPA/600/R-09/122. www.epa.gov/nerlcwww/ordmeth.htm. Accessed 8/23/13.

Wilde, F.D., Radtke, D.B., Gibs, Jacob, and Iwatsubo, R.T. eds. 2004 with updates through 2009. *Processing of Water Samples* (version 2.2) Techniques of Water-Resources Investigations (TWRI), book 9, chap. A5, Section 5.2.2.C (Procedures

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for processing samples for carbon analysis). Reston, VA: U.S. Geological Survey. http://pubs.water.usgs.gov/twri9A5/. Accessed 8/23/13.

Dissolved Oxygen (DO)

(Richard T. Wilkin)

MEDIA: Water

MEASUREMENT OBJECTIVES: The objective of dissolved oxygen (DO) measurements is to determine the concentration of free molecular oxygen (O_2) in water samples. The concentration of DO can be used to assess the stability of various trace metals, radionuclides, toxic anionic species, and organic contaminants. Oxygen is an electron acceptor (oxidant) and is often important in biological and non-biological redox reactions. The concentration of DO in groundwater and surface water is controlled by atmospheric interactions, mixing with oxygen-rich meteoric water, microbial respiration, biodegradation of organic matter, and reactions with reduced mineral phases. DO measurements must be made in the field.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): The measurement of DO can be made using Winkler titrimetric methods, colorimetric methods, or membrane-covered electrode methods. Selection of the most suitable analytical technique should follow from an analysis of project objectives. In general, Winkler methods provide the most accurate and precise DO concentrations but require the most effort to carry out. Colorimetric and electrode techniques are generally not as accurate or precise as Winkler methods, but these techniques are much easier to implement in the field. Field analysts should be thoroughly familiar with the sampling and measurement techniques for the specific method to be used.

ANALYTICAL TECHNIQUE(S): Analytical methods will depend on the choice of technique. The modified Winkler techniques involve sample collection in glass-stoppered bottles, addition of prepared reagents, and then titration with a standardized thiosulfate solution (ASTM D888). Colorimetric techniques involve cracking a glass ampoule in an overflow cell and comparing color development of the ampoule contents with sets of liquid color standards or by quantitating color development with a spectrophotometer (ASTM D5543). Electrode measurements of DO should generally be made in flow-through cells or in overflow cells. Manufacturer's recommendations should be closely followed for procedures of calibration and sample measurement. Temperature, barometric pressure, and salinity must also be measured and recorded to determine the degree of oxygen saturation.

LIMITATIONS/INTERFERENCES: All techniques for the measurement of DO are subject to contamination if poor sampling technique allows introduction of air to the sample. Recommendations for sampling must be closely followed, especially with respect to insuring adequate purge volumes before sample measurement and eliminating sample mixing with air.

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DOCUMENTATION/DATA HANDLING: Calibration notes and sample measurements should be recorded in a dedicated notebook.

BIBLIOGRAPHY:

American Public Health Association, American Water Works Association and Water Pollution Control Federation. 1985. *Standard Methods for the Examination of Water and Wastewater,* 16th edition. Washington, D.C.: American Public Health Association.

ASTM D888. 1992. *Standard Test Methods for Dissolved Oxygen in Water*. West Conshohocken, PA: ASTM International. Available from www.astm.org. Accessed 8/23/13.

ASTM D5543. 1995. *Standard Test Methods for Low-level Dissolved Oxygen in Water*. West Conshohocken, PA: ASTM International. Available from www.astm.org. Accessed 8/23/13.

Gilbert, T.W., Behymer, T.D., and Castaneda, H. B. 1982. Determination of dissolved oxygen in natural and wastewaters. *American Laboratory* 14:119-134.

Hitchman, M.L. 1978. *Measurement of Dissolved Oxygen.* John Wiley and Sons, 255 p.

Wilkin, R.T., McNeil, M.S., Adair, C.J., and Wilson, J.T. 2001. Field measurement of dissolved oxygen: A comparison of methods. *Ground Water Monitoring and Remediation* 21:124-132.

Eh/pE/ORP

(Richard T. Wilkin)

MEDIA: Water

MEASUREMENT OBJECTIVES: Eh is the measured potential of a platinum (Pt) electrode referenced to the Standard Hydrogen Electrode (SHE). Eh measurements are a relatively easy approach for determining overall redox conditions, but in most cases must be thought of as qualitative indicators of the state of oxidation or reduction of a natural system (Langmuir, 1971). In some cases, measurements of Eh can be used to correlate with the speciation of redox sensitive elements, such as nitrogen, manganese, and iron. Eh is not the same as the oxidation-reduction potential (ORP). ORP is the direct, uncorrected reading of the reference electrode.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): The measurement of ORP/Eh is normally accomplished with a digital meter and a combination Pt electrode, typically with an Ag/AgCl reference electrode. A selection of redox meters and electrodes is available. Operators should be thoroughly familiar with the recommendations for calibration and operation provided by the manufacturer. The standard method for redox measurement is to record measured potentials with time until a stable value is obtained (generally ±5 mV). Stable readings can be obtained in less than 30 minutes. Longer equilibration times are possible depending on rate of pumping and fluid composition.

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ANALYTICAL TECHNIQUE(S): The recommended procedure for measurement of Eh uses a small-volume flow-through cell into which a temperature and Eh electrode is inserted. After purging the cell with several volumes of water, ORP readings are recorded until they stabilize or a minimum value is obtained. Proper preparation and maintenance of the Pt electrode is required. Proper procedures include frequent polishing of the electrode surface, changing of the internal filling solution, and frequent checks with standard solutions that are maintained at ground water or surface water temperature. The most common standard solutions for reference checks are Zobell's solution (Nordstrom, 1977), Light's solution (Light, 1972), and others recommended by electrode manufacturers. Measurements of ORP must be corrected to reference the Standard Hydrogen Electrode in order to obtain Eh values. To properly reference measurements to the Standard Hydrogen Electrode, correction factors must be available as a function of temperature for the specific type of reference electrode and reference solution used in the measurement assembly. Electrodes should generally be checked in the laboratory before field activities begin. Manufacturer's recommendations should be closely followed for procedures of calibration and sample measurement.

LIMITATIONS/INTERFERENCES: Redox electrodes are notorious for being slow to equilibrate. The rate of electrode response is generally controlled by the concentrations of electroactive species in the sample being measured. So where pH electrodes respond to aqueous concentrations of hydrogen ions, Eh electrodes respond to electron transfers between solutes, e.g., Fe^{2+} and Fe^{3+}. Since ORP electrodes respond to instantaneous electron transfer reactions, it is not always possible to obtain a stable measurement, either because there are no instantaneous reactions or because the oxidized or reduced species is not present. Measuring a low ORP, such as -300 mV, is challenging because oxygen dissolution in the water disrupts the measurement.

DOCUMENTATION/DATA HANDLING: All calibration notes and sample measurements should be recorded in a dedicated notebook.

BIBLIOGRAPHY:

American Public Health Association, American Water Works Association, Water Environment Federation (APHA). 1999. *Standard Methods for the Examination of Water and Wastewater, 20th Edition*. Method 2580 – Oxidation-reduction potential (ORP). Washington, D.C.: American Public Health Association.

Langmuir, D. 1971. Eh-pH determination. In R.E. Carver, ed., *Procedures in Sedimentary Petrology*, Ch. 26, p 597-635. New York: Wiley-Interscience.

Light, T.S. 1972. Standard solution for redox potential measurements. *Analytical Chemistry* 44:1038-1039.

Lindberg, R.D., and Runnells, D.D. 1984. Ground water redox reactions: An analysis of equilibrium state applied to Eh measurements and geochemical modeling. *Science* 225:925-927.

Nordstrom, D.K. 1977. Thermochemical redox equilibria of Zobell's solution. *Geochimica et Cosmochimica Acta* 41:1835-1841.

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Field-Test Kits and Ion-Selective Electrodes

(Kathleen S. Smith)

MEDIA: Water

MEASUREMENT OBJECTIVES: A variety of field-test kits for analysis of water-quality parameters is commercially available. Their use depends upon the study objectives and budget. Field-test kits are useful in remote areas and in countries where laboratory facilities are not readily available. They are a cost effective way of performing screening and prioritization, especially when immediate decisions or actions are required. They also are useful to measure unstable constituents (e.g., dissolved oxygen or sulfide) or to measure sudden changes in water quality. They can be used in routine monitoring, contaminant identification, and for tracking contaminant source(s). Field-test kits generally are quick, simple, robust, and inexpensive, and users do not need to have a high level of expertise. However, their accuracy, precision, and sensitivity generally are inferior to laboratory analytical measurements for the same constituent.

Ion-selective electrodes (ISEs) determine the activity of a particular ion in solution. ISEs can be useful for continuous monitoring to detect changes in the activity of an ion, or in situations (e.g., aquatic metal toxicity investigations) when measurement of a particular ionic form is of interest. It is important to keep in mind that ISEs measure the activity of a particular ion, not the total concentration of an element.

SPECIFIC FIELD SAMPLING EQUIPMENT: General instructions cannot be provided for field-test kits because they use different methodologies and equipment. The user should follow the manufacturer's instructions. There are different types of field-test kits including single-parameter kits, multiple-parameter kits, test strips, drop tests, color-comparison tests, and probe tests. See Table A.3.3 for a summary of methods.

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TABLE A3.3 Some single-parameter field-test kits [Kit methodology: A = appearance/turbidity; C = visual colorimetric; GNG = go no go; P = photometric; T = titrimetric]. Modified from ASTM 2008.

Analyte	Methodology
Acidity	T
Alkalinity	C, P, T
Aluminum	C, P
Ammonia	C, P
Arsenic	C
Boron	C, P
Bromine	C, P, T
Cadmium	C
Calcium	P, T
Carbon dioxide	T
Chloride	A, C, P, T
Chromium(III)	C
Chromium(VI)	C, P, T
Cobalt	C
Copper	C, P, T
Cyanide	C, P, T
Fluoride	P
Hardness	C, GNG, P, T
Hydrogen peroxide	C, P, T
Iron	C, P
Lead	C, P
Manganese	C, P
Magnesium	C, T
Molybdate	C, P, T
Nickel	C, P
Nitrate	C, P
Nitrite	C, P, T
Oxygen (dissolved)	C, P, T
pH	C, P
Phosphate	C, P
Silica	C, P
Silver	P
Sulfate	A, C, P, T
Sulfide	C, P, T
Sulfite	C, P, T
Thiocyanate	C
Tin	C
Vanadium	C
Zinc	C, P, T

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Multi-parameter test kits combine single-parameters test kits for a particular application. For example, test kits are available for acid mine drainage (includes acidity, alkalinity, pH, and Fe) and water quality (includes combinations of hardness, pH, Cl, Fe, Mn, and several other parameters).

Test strips are semi-quantitative and useful for pre-screening and spot-checks. They are easy to use and inexpensive. Test strips are available for alkalinity, hardness, pH, nitrogen species, phosphorus species, As, Cl, Cu, and Fe.

Portable water-quality labs include a portable spectrophotometer that can conduct tests for a variety of parameters including acidity, alkalinity, hardness, pH, suspended solids, nitrogen species, phosphorus species, sulfur species, Ca, Cl, Cr, Cu, Fe, Mn, Mo, and Si. Portable water-quality labs are especially useful when a temporary on-site laboratory can be assembled.

ISEs measure potential in a solution against a reference electrode of constant potential. ISEs respond selectively to ions in the presence of other constituents, and the most commonly used ISE is the pH electrode. To perform ISE measurements it is necessary to have a pH meter that also measures millivolts. Most ISEs have a built-in reference electrode (referred to as a combination electrode); however, some ISEs have a separate reference electrode that requires that the meter have a pin connection to accommodate the reference electrode. Types of ISEs include membrane electrodes, solid-state electrodes, gas-sensing electrodes (to measure dissolved gases), and glass-membrane electrodes. Ions that can be measured include barium(II), bromide, cadmium(II), calcium(II), chloride, copper(II), cyanide (CN^-), fluoride, iodide, lead(II), mercury(II), nitrate, perchlorate, potassium(K^+), silver (Ag^+), sodium (Na^+), sulfide (S^{2-}), thiocyanate (SCN^-), and water hardness.

LIMITATIONS/INTERFERENCES: Test-kit techniques typically have lower accuracy and higher detection limits than do analytical laboratory methods. In addition, calibration frequently is difficult or not adjustable for kits. Sample matrix and the presence of other constituents can cause interferences. It is important that kits be evaluated for their reliability and sensitivity in the desired concentration range and sample matrix in question. The occurrence of false positives and false negatives is fairly common for some types of kits.

Most ISEs require a specific pH range for optimal performance. In addition, response time for ISEs often is long, frequently 15 min or longer per reading. Ionic strength and temperature affect ISE measurements and should be kept constant. The detection limit of ISEs for metal ions is sometimes a limiting factor in natural systems. Many ISEs are subject to fouling over time and require special cleaning and maintenance.

ADDITIONAL CONSIDERATIONS: Test kit reagent refills commonly are available from manufacturers. Refills permit cost savings through reuse of the major test kit components. Field-test kits also are available for soils (e.g., Liebig et al., 1996; USDA, 2001). Development of new methods and applications for field-test kits and probes are an important area of current research (e.g., Steinmaus et al., 2006; Guo et al., 2008).

BIBLIOGRAPHY:

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ASTM D5463. 2008. *Standard Guide for Use of Test Kits to Measure Inorganic Constituents in Water*. West Conshohocken, PA: ASTM International, Available from www.astm.org, accessed 9/16/13.

EPA Recognition of Lead Test Kits—U.S. Environmental Protection Agency. 2012. www.epa.gov/lead/pubs/testkit.htm. Accessed 8/23/13.

Guo, L., Ni, Q., Li, J., Zhang, L., Lin, X., Xie, Z., and Chen, G. 2008. A novel sensor based on the porous plastic probe for determination of dissolved oxygen in seawater. *Talanta* 74:1032–1037.

Ion Selective—Van London-Phoenix Company. 2011. www.vl-pc.com/default/index.cfm/ion-selective. Accessed 8/23/13.

Liebig, M.A., Doran, J.W., and Gardner, J.C. 1996. Evaluation of a field test kit for measuring selected soil quality indicators. *Agron. J.* 88:683–686.

Self-Filling Ampoules for Water Analysis—CHEMetrics. 2010. www.chemetrics.com/index.php?Page=methods&tab=1. Accessed 8/23/13.

Steinmaus, C.M., George, C.M., Kalman, D.A., and Smith, A.H. 2006. Evaluation of two new arsenic field test kits capable of detecting arsenic water concentrations close to 10 µg/L: *Environ. Sci. Technol.* 40:3362–3366.

Test Kits—CLU-IN, U.S. Environmental Protection Agency. 2009. www.clu-in.org/characterization/technologies/color.cfm. Accessed 8/23/13.

Test Kits—Hach Company. 2012. www.hach.com/test-kits/category?productCategoryId=7640199346. Accessed 8/23/13.

USDA (U.S. Department of Agriculture). 2001. Soil Quality Test Kit Guide. Washington, D.C.: USDA Agricultural Research Service. http://soils.usda.gov/sqi/assessment/files/test_kit_complete.pdf. Accessed 8/23/13.

Stream Flow and Discharge

(Kathleen S. Smith and Carol C. Russell)

MEDIUM: Water

OBJECTIVES: Stream flow (or discharge) is the volume of water per unit of time. It is commonly expressed as cubic feet per second (ft^3/s or cfs), acre-feet per day, or cubic meters per second (m^3/s). It cannot be directly measured; instead, it is computed from variables that can be directly measured. Discharge is computed by multiplying the area of water in a channel cross section by the average velocity of water in that cross section (discharge = area x velocity). Accurate stream flow data are important because flow impacts site hydrology, water quality, aquatic biota, habitat, and ecosystems. Stream flow information is critical to computing loads of water constituents, predicting floods, and designing engineering structures and mitigation strategies at mining sites.

SPECIFIC EQUIPMENT/TECHNIQUES: A common way to determine discharge is with a mechanical current meter using the velocity-area method. For this method the stream channel cross section is divided into several vertical segments, and the area for each segment is obtained by measuring the width and depth of the segment. For small streams, width generally is measured using a measuring tape or cable, and depth is

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measured using a wading rod with designated intervals marked on the rod. For large streams and rivers the width is measured with a calibrated cable or reel from a bridge, cableway, or boat, and the depth is measured by suspending a sounding weight into the water. Global positioning system technology also can be used for width measurements. Water velocity is measured for each segment by using a mechanical current meter. A common design for current meters, which are available in different sizes, consists of a wheel with cups that revolve around a vertical axis. The rate that the cups revolve is related to the water velocity, and generally an electronic signal is transmitted for each revolution. Discharge is then computed for each stream segment by multiplying the segment area by the measured velocity, and total discharge is computed by summing the discharge from each segment. Turnipseed and Sauer (2010), U.S. Environmental Protection Agency (2001), and Rantz and others (1982) provide detailed descriptions of discharge measurements, equipment, and calculations.

Hydroacoustic meters (e.g., acoustic Doppler current profilers (ADCP) and acoustic Doppler velocimeters (ADV)) represent a relatively new approach for determining streamflow. The ADCP is rapidly replacing traditional mechanical current meters for streamflow measurements (Muste et al., 2007; Oberg and Mueller, 2007). It sends a sound pulse into the water and measures the change in frequency of the sound pulse. The change in frequency is translated into water velocity using the Doppler Effect. Water depth is determined by measuring the travel time of a sound pulse. Hydroacoustic streamflow measurements are less time-consuming than traditional methods, allow measurements to be made under conditions not amenable to other techniques (e.g., flooding), and provide more detailed streamflow information compared to traditional mechanical current-meter measurements (Simpson, 2001; see USGS OSW Hydroacoustics website).

Discharge measurements made in shallow depths and low velocities are problematic. Under these conditions, use of a weir or a Parshall flume is a preferable method to determine discharge. Weirs generally are less expensive than flumes and are more sensitive at low flows. However, weirs tend to trap sediment, and they are not suitable in steep or unstable channels. V-notch weirs often are used in stormwater diversions around waste piles and tailings to verify design specifications.

The most accurate way to determine very small discharges is the volumetric method. This method involves measuring the time it takes to fill a container of known capacity. The container should be calibrated either by volume or weight of fluid. Float measurements are another way to obtain discharge in low-velocity streams. For this method two cross sections are selected along a straight stream reach, and the travel time is measured for a float to move from one section to the other (for a minimum travel time of 20 seconds).

Discharge also can be determined using tracers, which relies on determining the degree of dilution of an added tracer solution or an in-situ tracer by the flowing water. Tracer discharge measurements are described in separate method write-ups.

Stream stage (or stage or gage height) is the height of the water surface above a specified elevation. Stream stage can be used to compute discharge once a relationship is established between stream stage and discharge at a particular site under a variety of hydrologic conditions. This relationship is developed by conducting frequent direct-discharge measurements at the site for a range of stream stages and creating a rating

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curve, which is a chart of stage versus streamflow. Various methods can be used to measure stream stage. The simplest method is to install a permanent staff gage, similar to a measuring stick, in the stream that cannot be dislodged or moved. The stage can then be read directly from the staff gage. Another common method to measure stream stage is to install a stilling well, which allows water from the stream to enter the well through underwater pipes such that the water in the well is at the same elevation as the water in the stream. The stage is measured inside the stilling well using a float, pressure, optical, or acoustic sensor. Sauer and Turnipseed (2010) provide detailed descriptions of stage measurements at gaging stations.

SPATIAL OR TEMPORAL CONSIDERATIONS: Streamflow changes during different seasons of the year, generally decreasing during the summer months when evaporation and evapotranspiration are higher, and increasing during spring runoff.

ADDITIONAL CONSIDERATIONS: Streamflow is related to the amount of water moving off the catchment area and is affected by weather conditions. Flow also can be influenced by withdrawal of water for irrigation or other purposes. Care must be taken in choosing a site to determine discharge. Important criteria include having (1) a straight stream reach with a well-defined channel cross section, (2) flow that is confined to the channel, (3) a streambed free of aquatic growth, and (4) no large rocks or obstructions nearby. Channel roughness can be taken into account using the Manning equation, which relates velocity to total bed resistance or friction, to calculate flow velocity.

DATA AVAILABILITY AND HANDLING: The U.S. Geological Survey operates stream-gaging stations across the United States (see USGS NSIP website; see USGS Surface-water Data for the Nation website). Digital data can be downloaded from these sites. Turnipseed and Sauer (2010), USEPA (2001), and Rantz and others (1982) discuss considerations necessary to obtain quality discharge measurements.

BIBLIOGRAPHY:

ASTM D3858. 2008. *Standard Test Method for Open-Channel Flow Measurement of Water by Velocity-Area Method*. West Conshohocken, PA: ASTM International. Available from www.astm.org. Accessed 8/23/13.

ASTM D4409. 2008. *Standard Test Method for Velocity Measurements of Water in Open Channels with Rotating Element Current Meters*. West Conshohocken, PA: ASTM International. Available from www.astm.org. Accessed 8/23/13.

ASTM D5089. 2008. *Standard Test Method for Velocity Measurements of Water in Open Channels with Electromagnetic Current Meters*. West Conshohocken, PA: ASTM International. Available from www.astm.org. Accessed 8/23/13.

ASTM D5388. 2007. *Standard Test Method for Indirect Measurements of Discharge by Step-Backwater Method*. West Conshohocken, PA: ASTM International. Available from www.astm.org. Accessed 8/23/13.

Muste, M., Vermeyen, T.R., Hotchkiss, R., and Oberg, K.A. 2007. Acoustic velocimetry for riverine environments. *J. Hydraul. Eng.* 133:1297–1298.

Oberg, K.A., and Mueller, D.S. 2007. Validation of streamflow measurements made with Acoustic Doppler Current Profilers. *J. Hydraul. Eng.* 133:1421–1432.

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OSW Hydroacoustics Home Page—U.S. Geological Survey. 2012. http://hydroacoustics.usgs.gov/. Accessed 8/23/13.

Rantz, S.E., and others. 1982. *Measurement and Computation of Streamflow: Volume 1. Measurement of Stage and Discharge. Volume 2. Computation of Discharge*. Water-Supply Paper 2175. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/wsp/wsp2175/. Accessed 8/23/13.

Sauer, V.B., and Turnipseed, D.P. 2010. *Stage Measurement at Gaging Stations*. Techniques and Methods, book 3, chap. A7. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/tm/tm3-a7/. Accessed 8/23/13.

Simpson, M. 2001. *Discharge Measurements Using a Broad-Band Acoustic Doppler Current Profiler*. Open-File Report 01-01. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/of/2001/ofr0101/. Accessed 8/23/13.

Turnipseed, D.P., and Sauer, V.B. 2010. *Discharge Measurements at Gaging Stations*. Techniques and Methods book 3, chap. A8. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/tm/tm3-a8/. Accessed August 8/23/13.

USEPA (U.S. Environmental Protection Agency). 2001. *Performing Quality Flow Measurements at Mine Sites*. U.S. Environmental Protection Agency, EPA/600/R-01/043.

USGS National Streamflow Information Program (NSIP)—U.S. Geological Survey. 2012. http://water.usgs.gov/nsip/. Accessed August 2012.

USGS Surface-Water Data for the Nation—U.S. Geological Survey. 2012. http://waterdata.usgs.gov/usa/nwis/sw. Accessed 8/23/13.

Hardness
(Barbara Butler)

MEDIUM : Water

MEASURMENT OBJECTIVES: Water hardness is defined as the sum of the polyvalent cations dissolved in the water. Calcium and magnesium are the most common cations that contribute to hardness; however, iron, strontium and manganese also may contribute (APHA et al., 1998). Hardness usually is reported in mg/l (or part per million, ppm) equivalents of calcium carbonate ($CaCO_3$). Another unit used is grains per gallon, where a grain/gal of hardness is equal to 17.1 mg/l of hardness as $CaCO_3$.

The hardness of a water body is primarily a function of the underlying geology of the area and results from dissolution of minerals by rainwater, which is naturally acidic from dissolved carbon dioxide (CO_2). Water in areas where the underlying geology is high in limestone rocks typically has a high hardness (e.g., > 100 mg/l as $CaCO_3$).

Waters are generally classified according to their degree of hardness. The general guidelines for hardness classification are presented in Table A3.4 (e.g., see http://water.epa.gov/scitech/swguidance/standards/criteria/current/index.cfm, accessed 9/16/13).

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TABLE A3.4 Classification of degree of hardness.

Classification	mg/l or ppm as $CaCO_3$
Soft	0-60
Moderately hard	61-120
Hard	121-180
Very hard	> 180

ANALYTICAL TECHNIQUES: Total hardness may be measured by a colorimetric titration method. This method involves diluting about 25 ml of sample to 50 ml with DI water and adding 1-2 ml of ammonia buffer, followed by addition of an indicator dye (clamagite or Eriochrome Black T). This is titrated, using a 0.01 M EDTA solution, until the color is a pale blue [sky blue]. The hardness is then determined by multiplying the milliliters of EDTA used by 20 and the results are reported as mg/l $CaCO_3$ (APHA et al., 1992).

Hardness may also be calculated using the following equation:

Total hardness in ppm $CaCO_3$ = 2.497 x Ca (ppm) + 4.155 x Mg (ppm) + 1.792 x Fe (ppm) + 1.822 x Mn (ppm).

In water where iron and manganese are minimal, those typically are excluded from this equation and the hardness based solely on calcium and magnesium.

***Aquatic Life Considerations*—**Several toxicity tests have shown that ambient water quality conditions (e.g., water hardness, pH, dissolved organic carbon, and dissolved oxygen) affect the toxicity of metals to aquatic life (e.g., Mount, 1966; Carroll et al., 1979; Davies et al., 1993). Water hardness is one of the more common parameters measured in toxicity tests that has shown a consistent ameliorating effect on metal toxicity. As water hardness increases, toxicity of a metal typically decreases, due to competition of the hardness ions with the metal ions for binding sites on respiratory membranes (Pagenkopf, 1983; Markick and Jeffree 1994).

To account for the effect of water hardness on metals toxicity, several of the national water quality criteria recommendations for the protection of aquatic life are expressed as hardness-dependent equations (See http://water.epa.gov/scitech/swguidance/standards/criteria/current/index.cfm).The USEPA criteria maximum concentration (CMC or acute criterion) and criteria continuous concentration (CCC or chronic criterion) recommendations for cadmium, chromium III, copper, lead, nickel, and zinc in the form of hardness-dependent equations (in micrograms per liter). For example, the equations for cadmium are as follows:

$$CMC = \{1.136672 - [\ln(hardness) \times 0.041838]\} \times e^{[1.0166 \times \ln(hardness) - 3.924]}$$

$$CCC = \{1.101672 - [\ln(hardness) \times 0.041838]\} \times e^{[0.7409 \times \ln(hardness) - 4.719]}$$

BIBLIOGRAPHY:

American Public Health Association (APHA), American Water Works Association (AWWA), and the Water Environmental Federation (WEF). 1992. Standard Methods for Examinations of Water and Wastewater, 18th ed. United Book Press, Inc. Baltimore, Maryland.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

American Public Health Association (APHA), American Water Works Association (AWWA), and the Water Environmental Federation (WEF). 1998. Standard Methods for Examinations of Water and Wastewater, 20th ed. United Book Press, Inc. Baltimore, Maryland.

Carroll, J.J., S.J. Ellis and W.S. Oliver. 1979. Influences of hardness constituents on the acute toxicity of cadmium to brook trout (*Salvelinus fontinalis*). Bull. Environ. Contam. Toxicol. 22:575-581.

Davies, P.H., W.C. Gorman, C.A. Carlson and S.F. Brinkman. 1993. Effect of hardness on bioavailability and toxicity of cadmium to rainbow trout. Chem. Spec. Bioavail. 5(2): 67-77.

Markich, S.J. and R.A. Jeffree. 1994. Absorption of divalent trace metals as analogues of calcium by Australian freshwater bivalves: an explanation of how water hardness reduces metal toxicity. Aquatic Toxicology 29: 257-290.

Mount, DI. 1966. The effect of total hardness and pH on acute toxicity of zinc to fish. International Journal Air Water Pollution 10:49-56.

Pagenkopf, GK. 1983. Gill surface interaction model for trace metal toxicity to fisher: Role of complexation, pH, and water hardness. *Environmental Science Technology.* 17(6):342-347.

Hydraulic Conductivity and Infiltration

(Virginia T. McLemore)

DEFINITIONS: Hydraulic conductivity is measure of the rate of water flowing through pore spaces or fractures in a rock or sediment and is dependent upon the permeability and the degree of saturation. It is represented by K and K_{sat} is the saturated hydraulic conductivity. The soil water characteristic curve (SWCC) describes the relationship between matric suction and water content or degree of saturation. The term infiltration generally refers to the portion of precipitation that penetrates the surface and excludes the precipitation used by vegetation, that collected on the surface (ponded water), and surface runoff. Net infiltration typically refers to that portion of the infiltration that is not lost by evapotranspiration. Fluid transport occurs by uniform unsaturated flow, preferential channeling, saturated flow below the water table, and vapor transport through the vadose zone.

MEDIA: water in porous media

PRINCIPLE: Hydraulic conductivity is defined by Darcy's Law:

$$V = -K\, dh/dz$$

Where V is the average velocity of water through the soil, K is hydraulic conductivity, dh/dz is the difference of the hydraulic head over distance.

Common units are explained in Table A3.5 and ranges of hydraulic conductivities for various materials are in Table A3.6.

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TABLE A3.5. Common used units for hydraulic conductivity (K) (http://www.connectedwater.gov.au/framework/hydrometric_k.php#table1, accessed 3/23/11).

Description	Meters/day (SI unit)	Meters/second	Millimeters/day	Millimeters/hour
Extremely slow	0.000001	1.5741×10^{-11}	0.001	0.000041667
Very slow	0.0001	1.5741×10^{-9}	0.1	0.0041667
Slow	0.01	1.5741×10^{-7}	10	0.41667
Moderate	1	1.5741×10^{-5}	1000	41.667
Fast	10	1.5741×10^{-4}	10000	416.667
Very fast	100	1.5741×10^{-3}	100000	4166.667

TABLE A3.6. Hydraulic conductivities of some common rock types and soils (Brassington, 1988)

Rock Type	Grain size (mm)	Hydraulic conductivity K (m/d)
clay	0.0005-0.002	$10^{-8} – 10^{-2}$
silt	0.002-0.06	10^{-2} - 1
Fine sand	0.06-0.25	1-5
Medium sand	0.25-0.5	5-20
Coarse sand	0.5-2	20-100
Gravel	2-64	100-1000
shale	small	$5 \times 10^{-8} – 5 \times 10^{-6}$
sandstone	medium	10^{-3} - 1
limestone	variable	10^{-5}- 1
basalt	small	0.0003-3
granite	large	0.0003-0.03

PRIMARY USES/APPLICATIONS: Calculate hydraulic conductivity measure infiltration, and determine the soil water characteristic curve (SWCC).

MEASUREMENT METHODS:

The measurement methods of hydraulic conductivity fall into three groups: (1) in situ field measurements, (2) laboratory methods, and (3) correlation methods. In situ field methods include large scale tests (at drains, pumping tests at wells) and small scale measurements (infiltrometer, inversed augerhole, augerhole, piezometer, guleph permeameter, double tube, pumped borehole). Infiltration can be directly measured by tensiometers, infiltrometers, guelph permeameters, and lysimeters. Laboratory tests include constant head and falling head methods. Correlation methods using an empirical approach include measuring and correlating to soil properties such as pore size distribution, grain size distribution, soil texture, and describing soil mapping units.

In situ field measurements

Pumping tests may be conducted to estimate extent of capture of extraction wells, assess influences of faults on groundwater flow, assess communication between upper and lower aquifer units, and estimate aquifer transmissivity, hydraulic conductivity, and specific yield and/or storativity.

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Pumping tests would consist of a 10-hour step-discharge test to estimate the optimum pumping rate for the well, a minimum 24-hour to maximum 72-hour constant-rate test (conducted at the rate selected from the step-discharge test), and a minimum 12-hour recovery test.

Following testing, water level data will be analyzed using standard aquifer test analysis methods. The methods used will be carefully selected to best fit aquifer conditions and meet the method assumptions.

Infiltrometers are devices for measuring the rate of water influx into a soil or other porous medium to estimate one or more hydraulic properties of the material, including hydraulic conductivity and sorptivity (Fig. A3.5). Tension infiltrometers allow measurement of the hydraulic conductivity (or sorptivity) of the porous medium under a narrow range of unsaturated conditions, typically where the matric potential varies from near 0 to ~-30 cm (Clothier and White, 1981; Ankeny, et al., 1988; Reynolds and Elrick, 1991). Applications of the tension infiltrometer include measurement of macropore and preferential flow, estimation of soil structure, and characterization of the soil hydraulic conductivity/matric potential relationship. The SMS Tension Infiltrometer can also be used under zero tension in order to estimate the mine soil saturated hydraulic conductivity as described below. These properties are then used to calculate fluxes through the vadose zone.

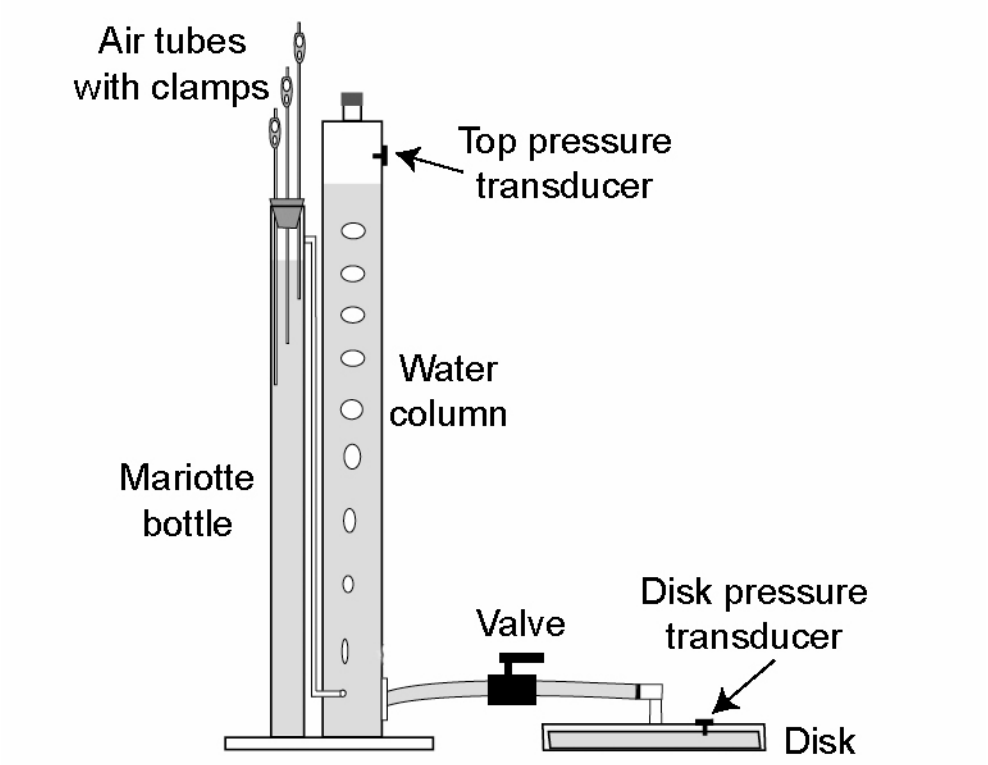


FIGURE A3.5. Tension infiltrometer design (adapted from Holt et al., 2002).

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The guelph permeameter measures saturated hydraulic conductivity, whereas the tension infiltrometer can be used to measure the saturated and unsaturated hydraulic conductivity. A Guelph permeameter measures saturated hydraulic conductivity by applying a constant pressure within a borehole or shallow hole. Like the tension infiltrometer (tensiometer), it applies a constant head using a Mariotte bottle, infiltrates water into an unsaturated material, and requires steady state flow conditions before the measurements can be used to calculate hydraulic conductivity. It differs from the tension infiltrometer in that it applies a positive pressure, instead of a tension. The Guelph Permeameter is an in-hole Mariotte bottle device for measuring the rate of water influx into most types of soil or other porous medium. It can be used to estimate one or more hydraulic properties of the material, including the saturated hydraulic conductivity, soil sorptivity, and matrix flux potential. The Guelph Permeameter method measures the steady-state rate, Q (m^3/s), necessary to maintain a constant depth of water, H (m), in an uncased cylindrical well of radius, a (m), above the water table. The field saturated hydraulic conductivity, Kfs (m/s), and matric flux, fm (m^2/s), are calculated from Q and H using an approximate analytical solution.

Laboratory methods

Determination of the drying soil water characteristic curve (SWCC) by means of the 'Hanging Column Method'. This method is suitable for making determinations of suctions in the range of 0 to 100 kPa. For this test, saturated soil specimens are placed in contact with a water-saturated porous plate. The matric suction is applied and causes the water to flow from the specimen until the equilibrium water content corresponding to the applied suction is reached. Several equilibria are established at successive matric suctions to build a soil water characteristic curve.

Correlation methods

Hydraulic conductivity and infiltration can be determined by the SWCC or by using other soil properties. The problem is that soil properties, including hydraulic conductivity and infiltration are site specific and correlation methods are dependent upon correlating the soil properties to other sites.

LIMITATIONS/INTERFERENCES: Most numerical models can account for uniform unsaturated flow but the other types of infiltration within a rock pile can be difficult to quantify and model numerically.

DOCUMENTATION/DATA HANDLING: Varies for each method.

BIBLIOGRAPHY:

Alayamani MS, Sen Z, 1993. Determination of hydraulic conductivity from complete grain-size distribution curves. Ground Water 31 (4) 551-555.

Ankeny, M.D., T.C. Kaspar, and R. Horton. 1988. Design for an automated tension infiltrometer. Soil Sci. Soc. Am. J. 52:893-896.

Ankeny, M.D., T.C. Kaspar, and R. Horton. 1990. Characterization of tillage and traffic effects on unconfined infiltration measurements. Soil Sci. Soc. Am. J. 54:837-840.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Ankeny, M.D., M. Ahmed, T.C. Kaspar, and R. Horton. 1991. Simple field method for determining unsaturated hydraulic conductivity. Soil Sci. Soc. Am. J. 55:467-470.

Brassington R, 1988. Field Hydrogeology. Geological Society of London Handbook Series. Open University Press.

Clothier, B.E. and I. White. 1981. Measurement of sorptivity and soil water diffusivity in the field. Soil Sci. Soc. Am. J. 45:241-245.

Elrick, D.E., and W.D. Reynolds, 1992, Infiltration from constant-head well permeameters and infiltrometers, p. 1–24, *In* C.G. Topp et al. (ed.), Advances in measurement of soil physical properties: Bringing theory into practice, SSSA Spec. Publ. 30, SSSA, Madison, WI.

Fredlund, D. G. and Rahardjo H. 1993. Soil Mechanics for Unsaturated Soils. John Wiley and Sons, New York, NY, USA.

Fredlund, D. G. and Xing, A. 1994. Equation for the Soil-Water Characteristic Curve, Canadian Geotechnical Journal, Vol. 31, No. 3, p. 521-532.

Fredlund, D. G., Xing, A., and Huang, S. 1994. Predicting the Permeability Function for Unsaturated Soil using the Soil-Water Characteristic Curve, Canadian Geotechnical Journal, Vol. 31, No. 3, p. 533-546.

Fredlund , M. D., Wilson, G. W., and Fredlund, D. G. 1997. Prediction of the Soil-Water Characteristic Curve from the Grain-Size Distribution Curve. Proceedings of the 3rd Symposium on Unsaturated Soils, Rio de Janeiro, Brazil, April 20-22, pp. 13-23.

Holt, R.M., J.L. Wilson and R.J. Glass. 2002. Spatial bias in field-estimated unsaturated hydraulic properties, Water Resour. Res. 38, doi:10.1029/2002WR001336.

McLemore, V.T., Dickens, A., Boakye, K., Campbell, A., Donahue, K., Dunbar, N., Gutierrez, L., Heizler, L., Lynn, R., Lueth, V., Osantowski, E., Phillips, E., Shannon, H., Smith, M., Tachie-Menson, S., van Dam, R., Viterbo, V.C., Walsh, P., and Wilson, G.W., 2008a, Characterization of Goathill North Rock Pile: New Mexico Bureau of Geology and Mineral Resources, Open-file report 523, http://geoinfo.nmt.edu/publications/openfile/details.cfml?Volume=523 , accessed 8/23/13.

Shepherd, Russell G. (1989). “Correlations of permeability and grain-size.” *Ground Water* **27** (5): 633–638.

Van Bavel, C.H.M. and D. Kirkham, 1948. Field measurement of soil permeability using auger holes. Soil. Sci. Soc. Am. Proc. 13:90-96.

Reynolds, W.D. and D.E. Elrick. 1991. Determination of hydraulic conductivity using a tension infiltrometer. Soil Sci. Soc. Am. J. 55:633-639.

Van Genuchten, M.Th. 1980. A Closed Form Equation for Predicting the Hydraulic Conductivity of Unsaturated Soils. Soil. Sci. Am. J. 44:892-898.

Vukovic M, Soro A, 1992. Determination of hydraulic conductivity of porous media from grain-size composition. Water Resources Publications, Littleton, Colorado http://enterprise.astm.org/filtrexx40.cgi?+REDLINE_PAGES/D7664.htm, accessed 8/23/13.

http://enterprise.astm.org/filtrexx40.cgi?+REDLINE_PAGES/D3385.htm, accessed 8/23/13.

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pH

(Richard T. Wilkin)

MEDIA: Water

MEASUREMENT OBJECTIVES: The pH of a solution is the negative logarithm of the hydrogen ion (H^+) activity and is expressed as:

$pH = -\log [H^+]$

where values in brackets express activity, not concentration. The pH is expressed using logarithmic units, normally with a scale from 0 to 14, but rare natural waters may have pH values outside of this range. Neutral pH (pH=7.00) is the point where $[H^+]=[OH^-]$ and should not be assumed to equal the background pH of natural waters. The general terms acidic and basic (or alkaline) waters refer to pH below (acidic) or above (basic) neutral pH. However, pure water, such as rainwater, in equilibrium with atmospheric CO_2, has a pH of 5.66 at 25ºC (Drever, 1988). Therefore, common usage of acidic waters refers to waters with a pH below 5. Most natural waters have a pH between 5 and 9; acid rain may have pH lower than 5, and some wetlands in arid climates may have pH greater than 9. Typically analysis of pH is required in the field. This is because a variety of physical, chemical, and biological processes can occur to significantly change pH values soon after sample collection. Measurements of pH are ideally made in a flow-through cell.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): The measurement of pH is normally accomplished with a digital pH meter, a plastic or glass liquid-filled or gel-filled combination Ag/Cl electrode, and a temperature compensation device. A wide variety of pH meters and electrodes are available. Operators should be thoroughly familiar with the recommendations for calibration and operation provided by the manufacturer. The standard method for pH measurements is to record values with time until a stable pH value is obtained.

ANALYTICAL TECHNIQUE(S): Electrodes need to be clean and in good order to produce reliable results. Electrodes should generally be checked in the laboratory before field activities begin. The liquid junction should be clear and the electrolyte solution in the electrode must be at the recommended level. Calibration of meters and electrodes is normally accomplished with pH 4, 7, and 10 buffers that are available from standard suppliers. Calibration routines may utilize two-point or three-point checks. It is also possible to obtain other pH buffers at lower or higher pH values. Choices of calibration buffers will ideally bracket expected pH values in water samples. Manufacturer's recommendations should be closely followed for procedures of calibration and sample measurement. Document all measurements in an instrument notebook. Because pH measurements are affected by temperature, sample temperature should always be recorded during pH measurement. When a flow-through cell is not used, proper measurement technique involves gentle stirring with a magnetic, Teflon-coated stir bar or equivalent stirring device to establish a stable reading. Stirring rates should not be too great in order to minimize the entrainment of carbon dioxide.

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LIMITATIONS/INTERFERENCES: Measurements of pH in very low ionic strength solutions can be challenging and generally long equilibration times are required. Extremely acidic solutions (<1.5), which are not typical, also require special considerations (see Nordstrom et al., 2000). The pH electrode functions by diffusing hydrogen ions through a glass membrane. Thus, the electrode has a very high electrical resistance. Consequently, any disruption of the meter or electrode can cause malfunction. This includes excessive moisture, wind, and large temperature fluctuations. Pampering the meter and electrode is necessary.

DOCUMENTATION/DATA HANDLING: All calibration notes and sample measurements should be recorded in a dedicated notebook.

BIBLIOGRAPHY:

American Public Health Association, American Water Works Association, Water Environment Federation (APHA). 1999. *Standard Methods for the Examination of Water and Wastewater, 20th Edition*. Method 423 – pH value. Washington, D.C.: American Public Health Association.

Bates, R.G. 1973. *Determination of pH, Theory and Practice*, 2nd Edition. New York: John Wiley and Sons.

Drever, J.I. 1988. *The Geochemistry of Natural Waters*, 2nd Edition. Englewood Cliffs, New Jersey: Prentice Hall.

Langmuir, D. 1971. Eh-pH determination. In *Procedures in Sedimentary Petrology,* Chapter 26, ed. R. E. Carver. New York: Wiley-Interscience, p. 597-635.

Nordstrom, D.K., Alpers, C.N., Ptacek, C.J., and Blowes, D.W. 2000. Negative pH and extremely acidic mine waters from Iron Mountain, California. *Environmental Science and Technology* 34:254-258.

Conductivity and Specific Conductance

(Kathleen S. Smith and Barbara A. Butler)

MEDIA: Water

MEASUREMENT OBJECTIVES: Conductivity and specific conductance are measures of the ability of water to conduct an electrical current, and are a general measure of stream-water quality. Conductivity is affected by temperature, with warmer water having a greater conductivity. Specific conductance is the term for conductivity values that have been compensated to 25°C. Conductivity increases with increasing inorganic dissolved solids and ions in water and commonly is positively correlated with sulfate concentration in mining influenced waters. Specific conductance generally is reported as microSiemens per centimeter (µS/cm); 1 µS/cm is equal to 1 µmhos/cm, which is an outdated unit of measurement.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): Conductivity is measured with a sensor and meter. Voltage is applied between two electrodes in the sensor when it is immersed in water. The electrical resistivity, which is the reciprocal of conductivity, is measured and is used to calculate the conductivity. Three types of sensors are available

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including a dip cell that can be suspended in the sample, a cup cell that contains the sample, and a flow cell that is connected to a fluid line. Different cell constants are available for sensors, and the choice of cell constant depends on the range of conductivity being measured. Conductivity sensors must be kept clean and dry when not in use. It is important to use calibration conductivity standard solutions that include the range of values of the samples being measured, and to calibrate with at least two standards. Field measurement of conductivity is preferable, and groundwater conductivity measurements should be made down hole or by using a flowthrough chamber. Radtke et al. (2005) provide detailed protocols for conductivity measurements in surface water and groundwater.

ANALYTICAL TECHNIQUE(S): Some conductivity meters can be used to measure or estimate total dissolved solids and salinity, based on an internal calibration. An estimate of total dissolved solids concentration also can be calculated by multiplying conductivity by an empirically determined factor between 0.5 and 0.9. The exact conversion factor depends upon the composition of the solution; however, a value of 0.67 commonly is used as an approximation when the actual factor is not known (http://www.stevenswater.com/water_quality_sensors/conductivity_info.html, accessed 8/23/13).

LIMITATIONS/INTERFERENCES: It is preferable to use a conductivity sensor that also measures temperature paired with a meter that automatically compensates for temperature in the reading. If the instrument does not automatically compensate for temperature, it is important to record both the uncompensated conductivity measurement and the temperature, and to use correction factors supplied by the instrument manufacturer. If the conductivity of the sample exceeds the range of the instrument, the sample may be diluted. However, it is important to perform dilutions according to the manufacturer's instructions because the dilution may not be linear with respect to specific conductance. Alternatively, a conductivity sensor that is designed for saline solutions can be used. Conductivity of water can change over time as a result of precipitation, sorption, and redox reactions. Therefore, it is preferable to measure conductivity in the field or as soon as possible afterwards. It is important to avoid contamination from the pH electrode filling solution when making conductivity measurements; either measure conductivity prior to measuring pH, or measure conductivity on a separate sample from that used for pH measurement. In a flowthrough chamber, position the conductivity sensor upstream of the pH electrode.

Conductivity is nonspecific (Down and Lehr, 2005); thus, it is dependent upon what ions are present, the concentrations of those ions, and any interactions between the ions. Nevertheless, it is a simple and reliable field measurement that will provide a semi-quantitative measure of the concentration of ions in solution. Conductivity can be estimated from measurements of common ions found in water samples using values listed in Table A3.7 and the equation provided below.

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TABLE A3.7 Conductivity factors for ions commonly found in water. Copied with permission from UNEP (2004).

Ion	Conductivity Factor µS/cm per mg/L
Cations	
Ca^{2+}	2.60
Mg^{2+}	3.82
K^{+}	1.84
Na^{+}	2.13
Anions	
HCO_3^{-}	0.715
Cl^{-}	2.14
SO_4^{2-}	1.54
NO_3^{-}	1.15

$Conductivity\ (\mu S/cm) = \sum C_i xf_i$, where C_i = concentration of the ion (mg/l) and f_i = conductivity factor from Table A3.7.

BIBLIOGRAPHY:

ASTM D1125. 2009. *Standard Test Methods for Electrical Conductivity and Resistivity of Water*. West Conshohocken, PA: ASTM International. Available from www.astm.org, accessed 8/23/13.

Conductivity/TDS—Stevens Water Monitoring Systems, Inc. http://www.stevenswater.com/water_quality_sensors/conductivity_info.html. Accessed 8/23/13.

Down, R.D., and Lehr, J.H. 2005. *Environmental Instrumentation and Analysis Handbook*, Chapter 23. Hoboken, NJ: John Wiley & Sons, Inc.

Eaton, A.D., Clesceri , L.S., Rice, E.W., Greenberg, A.E., and Franson, M.A.H. (eds.). 2005. *Standard Methods for the Examination of Water and Wastewater*, 21st ed. Washington, DC: American Public Health Association, American Water Works Association, and Water Environment Federation.

Radtke, D.B., Davis, J.V., and Wilde, F.D. 2005. *Specific Electrical Conductance* (version 1.2). Techniques of Water-Resources Investigations, book 9, chap. A6, section 6.3. Reston, VA: U.S. Geological Survey. http://water.usgs.gov/owq/FieldManual/Chapter6/Final508Chapter6.3.pdf. Accessed 8/23/13.

UNEP (United Nations Environment Programme). 2004. Electrical conductivity, chap. 12. In *Manual for National Training Programme*. Regional Resource Center for Asia and the Pacific. http://www.rrcap.unep.org/male/manual/national/12Chapter12.pdf. Accessed 8/23/13.

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Temperature
(Kathleen S. Smith)

MEDIA: Water and air

MEASUREMENT OBJECTIVES: Water and air temperature measurements are important because the rates of biological and chemical processes depend on temperature. Aquatic organisms require certain temperature ranges for optimal health. Of potential importance to organisms are the maximum and minimum temperatures for short periods of time, and daily and weekly average temperatures. Reproductive stages of organisms generally are the most sensitive biological stages to temperature. Temperature affects the amount of dissolved oxygen in water, with dissolved oxygen concentration decreasing as temperature increases. Most chemical/geochemical processes depend on temperature, including solubility and sorption processes, as do the rates of the processes. Temperature is measured in degrees Fahrenheit (F) or degrees Celsius (C). Compilations of national temperature data are provided by the National Climatic Data Center (see website), which is part of the National Oceanic and Atmospheric Administration of the U.S. Department of Commerce.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): Both air and water temperature should be measured in the field whenever water-quality samples are collected. Measurement devices should be routinely calibrated against a precision thermometer certified by the National Institute of Standards and Technology (NIST). Temperature can be measured with a liquid-in-glass thermometer, thermistor thermometer, or meter probe. Total-immersion liquid-in-glass thermometers are recommended for use by the U.S. Geological Survey; partial-immersion thermometers are not recommended. Thermistor thermometers are composed of a resistor made of semiconductors having resistance that varies predictably with temperature. Thermistors commonly are incorporated in instruments used for surface-water and groundwater measurements. Thermometers should have at least 0.5-degree graduations and a range of at least -5 to 45°C. Thermistors should have a digital readout to at least 0.1°C and a calibrated accuracy of at least 0.2°C. Alcohol-filled thermometers are preferable to mercury-filled thermometers for field work because they are less hazardous if broken. Armored thermometers can be purchased for field use and are much more robust than unprotected glass thermometers. Surface-water temperature measurements should always be taken in-situ, and not on subsamples. Groundwater temperature measurements should be taken either down hole or with a flow-through system. In streams, place the thermometer at least four inches below the surface or halfway to the bottom of a shallow stream. Allow enough time for the thermometer to stabilize. If possible, read the temperature while the thermometer is submerged. Wilde (2006) gives detailed instructions for temperature measurements in air, surface water, and groundwater.

LIMITATIONS/INTERFERENCES: Stream-water temperature will vary with width and depth, and temperature can be significantly different in shaded versus unshaded areas. If possible, vertical and horizontal temperature profiles should be collected in streams. Air temperature should be made with a dry thermometer; therefore, the same thermometer should not be used to collect both air- and water-temperature measurements.

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BIBLIOGRAPHY:

ASTM E2251. 2010. *Standard Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids*. West Conshohocken, PA: ASTM International. Available from www.astm.org. Accessed 8/23/13.

Eaton, A.D., Clesceri , L.S., Rice, E.W., Greenberg, A.E., and Franson, M.A.H. (eds.). 2005. *Standard Methods for the Examination of Water and Wastewater*, 21st ed. Washington, DC: American Public Health Association, American Water Works Association, and Water Environment Federation.

National Climatic Data Center—National Oceanic and Atmospheric Administration (NOAA). 2012. http://www.ncdc.noaa.gov/oa/ncdc.html. Accessed 8/23/13.

Wilde, F.D. 2006. *Temperature* (version 2.0). Techniques of Water-Resources Investigations, book 9, chap. A6., section 6.1. Reston, VA: U.S. Geological Survey. http://water.usgs.gov/owq/FieldManual/Chapter6/6.1_ver2.pdf. Accessed 8/23/13.

Turbidity

(Richard T. Wilkin)

MEDIA: Water

MEASUREMENT OBJECTIVES: Turbidity in surface water and groundwater is the result of suspended organic and inorganic matter such as clays, silts, plankton, and other microorganisms. Turbidity measurements are based on optical properties of a sample that result from the scattering and absorption of light by suspended materials. The amount of turbidity, measured in NTUs (nephelometric turbidity units), depends on the size, shape, and optical properties of suspended particles.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): Turbidity measurements are typically made in portable turbidimeters or nephelometers. A variety of such units are available. Operators should be thoroughly familiar with the recommendations for turbidity measurement and instrument calibration provided by the manufacturer.

ANALYTICAL TECHNIQUE(S): Proper measurement techniques are critical for minimizing the effects of stray light and gas bubbles in samples. The largest source of measurement error arises from unclean or scratched sample cells. Samples should be measured for turbidity immediately after collection to avoid settling of suspended particles or any alteration to sample pH or temperature. Sample dilution should be avoided because dilution can affect colloid stability, resulting in non-representative sample measurements. Formazin polymer is used as a primary standard reference suspension.

LIMITATIONS/INTERFERENCES: The presence in some samples of natural colored substances that absorb light may cause a negative interference. Condensation may occur on the outside of a sample cell, especially when cold samples are analyzed in warm and humid environments. All moisture should be wiped off the sample cell prior to placement in the instrument.

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DOCUMENTATION/DATA HANDLING: All calibration notes and sample measurements should be recorded in a dedicated notebook.

BIBLIOGRAPHY:

American Public Health Association, American Water Works Association, Water Environment Federation (APHA). 1999. *Standard Methods for the Examination of Water and Wastewater, 20th Edition*. Method 2130 – Turbidity. Washington, D.C.: American Public Health Association.

WATER-SPECIALIZED METHODS AND TECHNIQUES

Continuous Flow Analysis (Flow Injection Analyzer)

(William Lipps)

PRINCIPLE: Aqueous sample or standards are injected (usually with a valve) into a continuously flowing, non-segmented analyte-free carrier stream. The injected sample disperses and reacts with the reagents already in the carrier stream or added to it downstream from the point of sample injection. The reaction product formed is measured by a flow through detector.

The analytical cartridge may include several modules in which preparative steps such as digestion, filtration, separation by dialysis or gas diffusion, ion exchange, dilution can occur. Within the limits of pump precision, each sample undergoes the exact same processes as the ones preceding or following. The most frequently cited FIA detectors are flow-through photometers and spectrophotometers, but applications using amperometry (Milosavljevic, 1995; Solujic, 1999), voltammetry (ion selective electrode; Stulik, 1999), flame photometers, ICP-OES, ICP-MS, atomic absorption (McIntoc, 1996), and fluorescence detectors are also well documented. (Miro, 2005).

PRIMARY USES: Major ions, trace trace metals, and nutrients, in water samples and soil and plant extracts. Flow Injection Analyzers automate wet chemistry, improve repeatability, lower reagent and sample consumption, and reduce manual labor. Reducing the number manual operations performed by analysts also improves safety. Flow Injection Analyzers have been in use since the late 1970's and are the basis of several regulatory methods including OIA1677 for cyanide (USEPA, 1999).

LIMITATIONS: Samples are usually aqueous; however, systems utilizing organic liquids for solvent extraction have been described (Karlberg, 1978). Carrier solutions need to match sample matrices to minimize refractive index gradient artifacts that would otherwise compromise analytical results. Digestion methods usually require that standards be digested along with samples. Most systems use peristaltic pumps, which do not provide constant flow, and therefore limit dispensing precision; pump tube delivery rates varies with use, which necessitates frequent calibration and pump tube replacement. Although routine operation is possible with unskilled analysts, troubleshooting and developing new methods requires an analyst familiar with continuous flow analysis.

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SCOPE: Quantitation ranges can vary by method, and by instrument manufacturer, but are usually in the low ppb to high ppm range. Accuracy may be ±30% at the lower calibration levels (depending on the method), or as good as ± 1% at mid range calibration for some analytes.

DETECTION LIMITS: Detection limits vary by method and/or analyte but are usually in the low ppb to low ppm range in solution.

BIBLIOGRAPHY:

Karlberg, B.; and Thelander, S. 1978, Anal. Chim. Acta, 98

McIntosh, S.; Brindle, I., 1996, *Flow Injection Sate of the Art Applied to Atomic Spectroscopy,* Spectrochemica Acta Part B; Atomic Spectroscopy, Volume 51, Issue 14, p. 1733 – 1941

Milosavljevic, E.B; Solujic, Lj; and Hendrix, J.L. 1995,*Environmental Science and Technology,* Vol 29, No. 2, p. 426 – 430, 1995

Miro, M.; Frenzel, W. 2005, Flow Injection Analysis|Detection Techniques, Encyclopedia of Analytical Science (Second Edition), p. 48 – 56

Solujic, Lj.; Milosavljevic, E.B.; and Straka, M.R.; *Analyst,* 124, p. 1255-1260, 1999

Stulik, K. 1987, *Pure & Appl. Chem.,* Vol. 59, No 4, p. 521-530, 1987

USEPA,1999, Method OIA-1677 *Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry,* EPA – 821-R99-013

Continuous Flow Analysis (Segmented Flow Analysis)

(William Lipps)

PRINCIPLE: Aqueous sample or standards are aspirated into an analytical cartridge in alternate succession with an analyte-free wash solution that matches the matrix of the samples as closely as possible. The analytical cartridge consists of coiled, narrow bore tubing in which the continuously flowing analytical stream is segmented with inert gas (usually air) and proportioned with reagents. Air-segmented streams must wet the walls of the analytical channel for hydraulic stability and therefore surfactants are typically added to one or more of the reagents. The sample and reagent mix together by secondary flow generated within the liquid segments as they travel through the tubing. The air segments minimize mixing between liquid segments, thereby minimizing carryover between successive samples, and promote mixing within segments. Depending on the segmented flow analyzer design, a sample aspirated into its analytical cartridge for 30 seconds would be subdivided into 15 to 45 gas segments, which optimally would be added in phase with peristaltic pump pulsations.

The analytical cartridge might include several modules in which preparative steps such as such as dilution, digestion, filtration, separation by dialysis, gas diffusion, ion exchange, etc. can be affected. Within the limits of dispensing precision, which can be improved substantially by phasing gas segmentation with peristaltic pump roller lift off from the platen, each sample undergoes the exact same processes as the ones preceding or following. The analyte reacts with the reagents to form a final measurement product that is detected as a peak by a detector. Within the limits of pump precision, each sample undergoes the exact same processes as the ones preceding or following. The most

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frequently cited detectors are flow-through photometers and spectrophotometers, but detection by amperometry, voltammetry (ion selective electrodes), flame emission photometry, inductively coupled plasma optical emission spectroscopy, inductively coupled plasma mass spectroscopy, atomic absorption spectroscopy, and fluorescence are also well documented.(see the chapter on Flow Injection Analysis for a list of references)

PRIMARY USES: Minor and trace nutrients, anions, and a few metals in water samples and aqueous extracts are typical analyses. Continuous flow analyzers automate wet chemistry, improve repeatability, lower reagent and sample consumption, and reduce manual labor. The number of solution manipulations of sample and reagents by the analyst is decreased improving safety. Air segmented continuous flow analyzers have been in use since the late 1950's and are the basis of numerous regulatory methods.

LIMITATIONS: Samples are usually aqueous; however, systems utilizing organic solvents and solvent extraction have been described. Wash solutions often have to closely match the expected acid or salt concentration of the samples. Digestion methods usually require that standards be digested along with samples. Most systems use peristaltic pumps; pump tubing varies with use requiring periodic recalibration and replacement. While routine operation is possible with unskilled analysts, an analyst familiar with continuous flow needs to be available for new methods development or troubleshooting.

SCOPE: Quantitation ranges can vary by method, and by instrument manufacturer, but are usually in the low ppb to high ppm range. Accuracy may be ±30% at the lower calibration levels (depending on the method), or as good as ± 1% at mid range calibration for some analytes.

DETECTION LIMITS: Detection limits vary by method and/or analyte but are usually in the low ppb to low ppm range in solution.

BIBLIOGRAPHY:

Coakly, W.A., 1981, *Handbook of Automated Analysis, Continuous Flow Techniques*: Marcel Dekker, Inc., New York

Ewing, G.W., ed., 1997 *Analytical Instrumentation Handbook* 2nd Edition: Marcel Dekker, Inc., New York, p. 125-220

Furman, W.B., 1976, *Continuous Flow Analysis; Theory and Practice*: Dekker, New York

Snyder, L.S. and Adler, H.J., 1976, Dispersion in segmented flow through glass tubing in continuous-flow analysis: the ideal model: Analytical Chemistry, v. 48, p. 1017-1027.

Snyder, L.R., 1976, Reaction colorimeters as detectors in high-performance liquid chromatography : Extra-column band broadening with segmented flow through the reaction coil: Journal of Chromatography, v. 125, p. 287-306.

Snyder, L.R., 1980, Continuous-flow analysis: present and future: Analytica Chimica Acta, v. 114, p. 3-18.

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Automated Discrete Analysis

(William Lipps)

PRINCIPLE: A discrete analyzer is a computer-controlled, programmable, instrument that automates wet chemical analysis by dispensing samples and reagents into discrete reaction cells that often also serve as cuvettes for photometric measurement of assay chromophores. Dispensing into reaction cells is accomplished by high precision volumetric dispensers (syringe pumps, typically) that are, using interfaced to one or more robotic arms. Micro liter volumes of sample and reagent are mixed and react within individual, temperature controlled cuvettes to form analyte specific chemical reactions. Detection involves photometric measurement of the analyte within the cuvette. "Hybrid" discrete analyzers react samples within the individual cuvettes and then pump the reacted sample to a flow cell for measurement.

Methods, calibrations, sample volumes, reagent volumes, analytical wavelengths, etc, are stored in the software. An analyst loads samples and reagents into the instrument and selects which method or methods to run on each sample. Only the method selected per each sample is run and only the reagents required for the test is required, which typically results in greater reagent economy and less waste generation than can be achieved in FIA and SFA instrument formats.

Advantages of discrete analyzers include their ability to analyze a multitude of different analytes or different matrices without modifying analytical cartridges, facile, on-line preparation of calibration solutions by serial dilution, dilution and reruns of off-scale samples without operator intervention, and on demand operation, to provide analytical results within minutes of test selection or instrument startup

PRIMARY USES: Minor and trace nutrients, anions, and a few metals in water samples and aqueous extracts. Discrete Analyzers automate wet chemistry, improve repeatability, lower reagent and sample consumption, and reduce manual labor. The number of solution manipulations of sample and reagents by the analyst is decreased improving safety.

LIMITATIONS: Samples limited to aqueous solutions. The number of reagents, selectable methods, sample path-length, and number of sample positions are limited by manufacturer design and software. Discrete analyzers, while easy to use, are mechanically complex.

SCOPE: Quantitation ranges can vary by method, and by instrument manufacturer, but are usually in the low ppb to high ppm range. Accuracy may be ±30% at the lower calibration levels (depending on the method), or as good as ± 1% at mid range calibration for some analytes.

DETECTION LIMITS: Detection limits vary by method and/or analyte but are usually in the low ppb to low ppm range in solution.

BIBLIOGRAPHY:

Ruzicka, J. and Hansen, E.H., 1988, *Flow Injection Analysis* 2nd Edition: John Wiley and Sons

Stockwell, P.B., 1996, *Automatic Chemical Analysis*: Taylor and Francis Ltd., London

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Diffusive Gradients in Thin Films (DGT) for Dissolved Metals

(Laurie S. Balistrieri)

PRINCIPLE: DGT is an in-situ technique that uses a small device to accumulate dissolved, labile metals (Davison and Zhang, 1994; Zhang and Davison, 1995; Davison and others, 2000; Zhang and Davison, 2000, 2001). Labile metals are the sum of free ions, metal-inorganic ligand complexes, and some portion of metal-organic ligand complexes depending on their size, diffusion coefficients, stability, and kinetics of association and dissociation. The DGT device consists of a layer of hydrogel impregnated with a cation exchange resin (Chelex), a second diffusive hydrogel, and a filter in a 4.0 cm plastic housing. Once the device is deployed in water, a constant concentration gradient develops within the diffusive hydrogel. Labile metal species diffuse through the filter and hydrogel and accumulate on the resin. Upon retrieval, the metals are eluted with acid from the resin layer and measured using a suitable analytical technique (e.g., AA, ICP-MS). The mass of accumulated metal depends on the dissolved concentration of metals in solution, the length of deployment, temperature-dependent diffusion coefficients of metals through the hydrogel, the thickness, pore size, and exposure area of the hydrogel, and the chemical speciation of the dissolved metal. Differentiation between inorganic and organic metal species is possible by simultaneously deploying DGT units with open and restrictive pore diffusive hydrogels. Detailed technical information is available at the DGT Research Ltd. website (www.dgtresearch.com, accessed 9/16/13).

PRIMARY USES: DGT is designed to measure (1) in-situ dissolved metal concentrations, (2) time-averaged dissolved metal concentrations for monitoring purposes, (3) speciation of dissolved metals (labile inorganic and/or organic complexes), and, potentially, (4) bioavailability (effective dissolved metal concentrations).

LIMITATIONS: Possible problems include making measurements in very dilute solutions, very low flow, and at low pH (pH ~ 2-4.5) (Alfaro-De la Torre and others, 2000; Gimpel and others, 2001). The lower pH limit depends on competition between metals and hydrogen ions for Chelex. Weaker binding ions, such as Cd and Zn, only can be measured at pH values greater than the higher end of the low pH range, whereas stronger binding ions, such as Cu, can be measured at pH greater than the lower pH range.

INTERFERENCES: Not applicable.

SCOPE: DGT units have been deployed in the water columns of rivers, lakes, and oceans and in porewaters of soils and sediment. They can be used to measure a wide range of dissolved metal concentrations.

DETECTION LIMITS: Detection limits depend on the metal of interest and the length of deployment. The following example is for Zn. A typical DGT device (0.8 mm thick diffusive gel) deployed for 24 hours in a solution at 10°C containing 0.5 µg/L of fully labile Zn would accumulate 5.5 ng, which eluted and diluted to 2 mL for ICP-MS

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analysis yields 2.75 µg/L (more than 5 x the detection limit for ICP-MS). The longer the deployment time, the larger the mass of accumulated metal and the lower the detection limit. The maximum solution concentration that can be measured depends on the resin capacity, which for a 24 hour deployment is 0.5 mM (equivalent to 33 mg/L Zn).

DATA FORMAT: Information is reported as concentration of DGT available dissolved metal.

COST: DGT devices are available from DGT Research Ltd (www.dgtresearch.com, accessed 8/23/13) for a cost of 11.50 GPB ($17.53 on 5/14/13) each (minimum order 10, discounts available for larger volumes) plus associated analytical costs to measure dissolved metal concentrations.

BIBLIOGRAPHY:

Alfaro-De la Torre, M.C., Beaulieu, P.-Y., and Tessier, A., 2000, In situ measurement of trace metals in lakewater using dialysis and DGT techniques: Analytica Chimica Acta, v. 418, p. 53-68.

Davison, W., Fones, G., Harper, M., Teasdale, P., and Zhang, H., 2000, Dialysis, DET and DGT: In situ diffusional techniques for studying water, sediments and soils, *in* Buffle, J., and Horvai, G., eds., In Situ Monitoring of Aquatic Systems: Chemical Analysis and Speciation: Chichester, John Wiley & Sons, Ltd.

Davison, W., and Zhang, H., 1994, In situ speciation measurements of trace components in natural waters using thin-film gels: Nature, v. 367, p. 546-548.

Gimpel, J., Zhang, H., Hutchinson, W., and Davison, W., 2001, Effect of solution composition, flow and deployment time on the measurement of trace metals by the diffusive gradient in thin films technique: Analytica Chimica Acta, v. 448, p. 93-103.

Zhang, H., and Davison, W., 1995, Performance characteristics of diffusion gradients in thin films for the in situ measurement of trace metals in aqueous solution: Analytical Chemistry, v. 67, no. 19, p. 3391-3400.

Zhang, H., and Davison, W., 2000, Direct in situ measurements of liable inorganic and organically bound metal species in synthetic solutions and natural waters using diffusive gradients in thin films: Analytical Chemistry, v. 72, no. 18, p. 4447-4457.

Zhang, H., and Davison, W., 2001, In situ speciation measurements using diffusive gradients in thin films (DGT) to determine inorganically and organically complexed metals: Pure and Applied Chemistry, v. 73, no. 1, p. 9-15.

Stream Discharge By Tracer Injection

(Briant A. Kimball)

METHOD: A method of salt-tracer injection over a period of hours to days can be used to estimate discharge of mountain streams at many locations along a stream reach of interest. This method provides the hydrologic data needed for studies of groundwater-surface water interaction; mass loading of metals, nutrients or other constituents; and mass transfer of biogeochemical processes.

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PRINCIPLE: When a stream is uniformly mixed with an injected salt tracer that can be injected over a period of hours to days, depending on the characteristics of a particular stream and the length of the stream to be studied, discharge at multiple locations downstream from the injection can be determined by the amount of dilution of the tracer. The calculation is based on the conservation of mass. The rate and concentration of the injected tracer are known, thus the mass-flow (mass per unit time) of tracer into the stream must equal the mass flow of tracer in the stream. Because we can measure the concentration of the tracer (mass per volume) at any point of interest downstream from the tracer, we can calculate the rate of flow (volume per unit time), the discharge, of the stream at that point.

PRIMARY USES/APPLICATIONS: This method has two advantages for measuring discharge in mountain streams, which often have steep gradients, pools and riffles, and mostly cobble streambeds (Kimball et al., 2002; Kimball and Runkel, 2009). The first advantage is that the method accounts for both in-channel and exchangeable subsurface flows (called hyporheic flow). This is because the tracer travels with the water as it exchanges with the hyporheic zone connected to the stream. The second advantage is that the method allows for determination of discharge at many sampling locations, and when combined with concentration data from chemical samples, allow for the calculation of load. This provides detailed data that can be used for a Total Maximum Daily Load (TMDL) study.

SPECIFIC EQUIPMENT/TECHNIQUE(S): The use of tracers for measuring discharge is reviewed in Fitzpatrick and Cobb (1985). Application to mass-loading studies, particularly in streams affected by mine drainage is reviewed in Kimball et al. (2002) and Kimball et al. (2007). Important details of the method are given here.

Salt: Inorganic salts provide many advantages over dyes in mountain streams because dyes tend to adsorb to streambed materials and degrade in acidic streams, which lead to inaccurate calculations of discharge. A variety of salts can be used for different conditions. Sodium Chloride (NaCl) is readily available and inexpensive. A NaCl solution often must be prepared near the limit of chemical saturation of halite to overcome the presence of significant background concentrations of Cl in the stream. This has often led to pump failure with temperature variations and halite precipitates in the system. Magnesium Chloride ($MgCl_2$) is slightly more expensive, but much more soluble if chloride is the desired tracer. Sodium Bromide (NaBr) is an excellent tracer for near neutral and alkaline pH streams. It is more expensive, but Br generally has very low natural concentrations, so not as much salt is required. NaBr is much more soluble than NaCl, and does not cause problems with pumps. However, with the end of a photo-processing industry, NaBr has become more difficult to obtain, but can be purchased because it is used in oil and gas drilling fluids. Lithium Chloride (LiCl) and lithium bromide (LiBr) are good tracers in streams with pH less than about 5 because Li does not participate in chemical reactions with streambed materials at low pH; that is, it is transported conservatively. Both salts are very soluble, but are more expensive, especially LiBr.

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Mixing tank / bilge pump / injection tank: The tracer salt is mixed with stream water using a vessel that is open to allow stirring is needed for complete mixing. A plastic 30 gallon garbage can works well, using a canoe paddle for stirring. When the salt is completely dissolved the solution can be pumped from the vessel to an agricultural tank using a bilge pump (that is rinsed completely afterwards). This step is to prevent spilling the tracer that will occur by pouring from buckets. Agricultural tanks are available at farm stores and can be purchased with 120 gallon, 220 gallon, or greater capacities. An appropriate spigot must be placed at the outlet of the tank before the salt solution is added. Usually more than one "batch" of solution must be put into the tank for a given study, so the same proportion of salt to stream water (weight to volume) must be used for each batch to prevent density gradients in the agricultural tank.

Injection pumps / controller: The injection rate of the tracer must be constant over the whole period of injection to be able to attribute downstream tracer concentration variations to the hydrologic changes of the stream. If an injection rate slows as a result of decreasing battery power, the discharge calculation will be affected substantially. A system of positive displacement pumps is best, operated by a controller that counts and adjusts the revolutions of the pump to maintain a very constant injection rate. The injection generally proceeds with each minute divided into 10 pulses. Each pulse of solution lasts for some portion of 6 seconds, and as revolutions are counted for each pulse, the voltage to the pump is increased or decreased by the controller to achieve the desired rate for the next pulse. With a second pump added, the controller can start a backup pump if the first pump fails. The whole setup of pumps, data logger, and Darlington (for adjusting voltage to the pumps) can be fit into a large portable cooler (with holes drilled for tubing connections). If the pumps are higher in elevation than the outlet of the tank, the decreasing level of solution in the tank will not affect the rate.

Injection rate, salt volume needed: The injection rate is determined in a few steps. One must choose a target concentration for the tracer at the downstream end of a study reach that will be easily analytically measureable for the estimated discharge. For example, a target of 1 mg/L Br is often chosen as a sufficiently high concentration to be sure that Br concentrations could be measured all along the study reach. This concentration is comfortably greater than a typical lower limit of Br detection of 0.1 mg/L by ion chromatography. If the discharge at the end of the study reach was estimated to be 142 L/s (5 ft^3/s), that 1 mg/L Br would result in a mass flow of 142 mg/s. The injection rate from the pump must match that mass flow. If the injection solution had been mixed to be about 257,000 mg/L Br (each "batch" using a 55 lb., 25 kg, bag of NaBr in 20 gallons of stream water), then the pump rate would need to be 33 mL/min. For a 72 hour injection, that would require 38 gallons of injectate solution. Because it would be more practical to mix 40 gallons by the "batch recipe" above, the injection rate could be adjusted to 35 mL/min and the Br concentration at the end of the study reach would be slightly higher than 1 mg/L. The length of time for the injection must allow for the Br concentration at the end of the study reach to reach a constant value, indicating the desired uniform mixing along the study reach. Depending on the distance of stream to be studied, this typically could take 24 to 72 hours of injection, which must precede the synoptic chemical sampling.

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Quantification of injection rate and salt concentration: During the course of an injection, timed samples of the injection solution are obtained at the delivery point in the stream, usually by collecting 10 or fewer pulses of injectate solution. Later, in the laboratory, the collection bottle is first weighed with the salt solution for a total weight. The solution is then transferred to another bottle, and the first bottle is cleaned, dried, and weighed again without the salt solution. That gives the mass of injectate per time of the injection. When the specific gravity of the saline solution is determined the volume per time can be calculated. The specific gravity also gives the salt concentration from standard salt tables. Thus, both the rate (volume per time) and the tracer concentration (mass per volume) of the injection solution are determined.

LIMITATIONS: The method provides discharge at sampled locations along a stream study reach, but can only indicate increases in discharge. To quantify decreases, the method must be combined with independent discharge measurements. Such independent determinations of flow at appropriate locations along the study reach could be made by traditional metered area-velocity measurements or by "slug" tracer injections (Kilpatrick and Cobb, 1985). As standard practice, an independent discharge measurement should be made at the end of the study reach to constrain the discharge determined by tracer dilution. Discharge from inflows, both groundwater and surface water, is calculated as the difference between two stream sampling sites on either side of an inflow or along a given distance of the stream. The inflow discharge includes both surface and groundwater contributions to the main stream. The quantity of a surface inflow cannot be calculated separately without an independent discharge measurement.

ADDITIONAL CONSIDERATIONS: The arrival of tracer at selected downstream sites can be used to determine solute residence time and to fit parameters for a solute-transport model (Runkel, 1998). Detailed temporal sampling for the arrival is best done by an individual who can observe the arrival by tracking specific conductance or millivolts (using an ion sensitive electrode). An auto-sampler is very useful to track the tracer concentration with samples at regular intervals throughout the injection. This will indicate if the tracer has been uniformly mixed along the study reach.

DATA FORMAT: Because these data generally are part of a longitudinal study of a stream, the longitudinal distance along the study reach of each sampling site provides the reference for a calculated downstream discharge profile. Multiplying discharge values by the chemical concentration of a selected constituent provides mass-loading profiles that can be the basis of TMDL and biogeochemical-process studies.

COST: Salts can range from a few cents per pound (NaCl) to over $13 per pound (LiBr). NaBr is usually about $2 per pound. Pumps and pump heads (Fluid Metering Inc., QB 12 V DC with a Q pump head module, modified at the factory with a rotational sensor to count pump rotations) can be purchased for about $1,500 each. A data logger/controller (Campbell Scientific, CR-1000 or newer) may cost about $2,500. Auto-samplers (ISCO Teledyne 3700) might cost up to $2,500. A mixing can (greater than 20 gallon capacity) and paddle are less than $50. An agricultural tank can be less than $250. A bilge pump

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may cost $100. Tubing, fittings, values (for the tank), and incidental parts should all cost less than $250.

BIBLIOGRAPHY:

Kilpatrick, F.A., and Cobb, E.D. 1985. *Measurement of Discharge Using Tracers*. Techniques of Water-Resources Investigations book 3, chap. A16. Reston, VA: U.S. Geological Survey.

Kimball, B.A., and Runkel, R.L. 2009. Spatially detailed quantification of metal loading for decision making: Metal mass loading to American Fork and Mary Ellen Gulch, Utah. *Mine Water and the Environment* 28:274-290.

Kimball, B.A., Runkel, R.L., Walton-Day, K., and Bencala, K.E. 2002. Assessment of metal loads in watersheds affected by acid mine drainage by using tracer injection and synoptic sampling: Cement Creek, Colorado, USA. *Applied Geochemistry* 17:1183-1207.

Kimball, B.A., Walton-Day, K., and Runkel, R.L. 2007. Quantification of metal loading by tracer injection and synoptic sampling, 1996-2000. Professional Paper 1651. Reston, VA: U.S. Geological Survey. p. 417-495

Runkel, R.L. 1998. *One-dimensional Transport with Inflow and Storage (OTIS): A Solute Transport Model for Streams and Rivers*. Water-Resources Investigations Report 98-4018. Reston, VA: U.S. Geological Survey.

Groundwater Tracer Techniques

(Mike Wireman)

MEDIA: Water

MEASUREMENT OBJECTIVES: In recent years ground-water tracing techniques have been used in a variety of hydrogeologic settings as a tool for aiding in characterization of ground-water flow systems and have proven to be useful for identifying and characterizing contaminant transport pathways and transport velocities. Tracer tests can be used to obtain empirical data related to ground-water recharge, ground-water flow direction, flow rates, groundwater age, groundwater discharge to streams and flow system boundaries. Tracer recovery data, when combined with ground-water discharge data, can also provide quantitative data that can be useful for assessing the relative importance of various flow paths and fate of contaminants in the subsurface. Tracer test data can also be useful for refining hydrologic models and for designing and testing groundwater monitoring systems. Several tracers can be used together allowing several potential pathways to be evaluated simultaneously.

SPECIFIC SAMPLING TECHNIQUE(S): In general tracing can be can be divided into two categories: label-tracing and pulse-tracing. Using tracers as labels allows for identification of specific waters or plumes. Pulse-tracing involves sending an identifiable signal through part of a ground-water flow system at concentrations significantly above background. Ground-water tracers can be divided into two types - natural and artificial. Natural tracers are more applicable for labeling waters whereas artificial tracers are more suitable for pulse-tracing. Important natural tracers include; stable and radioactive isotopes, selected ions in solution, selected field parameters (specific conductance and

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temperature) and selected microorganisms. Commonly used artificial tracers include organic dyes and dye-intermediates, fluorocarbons, gases and salts (chloride, bromide, etc.).

Natural Tracers

Stable Isotopes - Stable isotopes have proven to be versatile natural tracers. For a given element, isotopic composition can vary due to partitioning or fractionation related to differences in reaction rates isotopic mass. This allows the ratios of isotopes to become fingerprints of climatic and hydrological conditions. Variations in annual rainfall, snow melt , latitude and elevation strongly affect the isotopic composition of melt waters. Isotopic fractionation can be more pronounced in low temperature settings than in warmer settings. Stable isotopes of oxygen and hydrogen are ideal tracers of water sources and movement because they are integral constituents of the water molecule. Oxygen isotopes include ^{16}O, ^{17}O and ^{18} O. Hydrogen isotopes include protium, (^{1}H) and deuterium (^{2}H). Stable isotopes of sulfur, nitrogen and carbon are also important environmental isotopes. These isotopes are constituents that are dissolved in water or carried in the gas phase. Stable isotopes of oxygen and hydrogen behave conservatively in groundwater which means interactions with organic and geologic material along the flowpath will have a negligible effect on the ratios of isotopes in the water molecule. The ratios of stable isotopes of dissolved sulfur, nitrogen and carbon can be significantly altered by reactions with organic and geologic material. Thus these solute isotopes have limited use for tracing water sources and flow paths. However data for solute isotopes can provide information on the reactions that are responsible for their presence in the water and the flow paths implied by their presence (Kendall, 1998). For detailed information on isotope geochemistry, the reader is referred to Kendall and McDonnell (1998) and Clark and Fritz (1997).

Radioactive Isotopes -Tritium, a radioactive isotope of hydrogen (half-life = 12.43 yrs), is naturally produced in the upper atmosphere by bombardment of nitrogen by neutrons. In addition, several hundred kilograms of tritium were released to the atmosphere during the period of above ground thermonuclear testing which was at a maximum during the 1950s and was discontinued in 1963. Pre-bomb concentrations of tritium in water have been determined to be close to the detection limit of 1 TU. Tritium is the only conservative tracer that can be used to determine the age of water older than one year. Since it is part of the water molecule, it is the only direct water dating method. The presence of tritium above background levels in ground water is an indication that recharge occurred during or after the bomb testing period. In addition to radioactive decay, tritium in ground water is subject to significant attenuation by matrix diffusion and dilution by mixing with younger water with less tritium. Consequently, tritium concentrations cannot be used to obtain an “absolute” age of ground water.

Artificial Tracers

Artificial tracers are those introduced into the ground-water flow system either purposely as part of a designed tracer test, or inadvertently as a spill or other human related activities which result in the introduction of a tracer into the ground water. To serve as a suitable tracer a substance must be: (1) non-toxic to people and the ecosystem, (2) either

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not present naturally in the ground-water system or present at very low, near-constant levels, (3) for chemical substances, soluble in water with the resultant solution having nearly the same density as water, (4) conservative, (5) easy to introduce into the flow system, and (6) unambiguously detectable in very low concentrations, (parts per billion)and (7) affordable and readily available. Organic fluorescent dyes are the most commonly used groundwater tracers.

Salts - Chloride and bromide solutions are commonly used for both ground-water and surface water tracing. These salts are very soluble, relatively inexpensive, conservative and non-toxic at concentrations typically used for tracing. They are also easily detectable at low concentrations. Chloride occurs naturally in some ground waters often in the tens to hundreds of parts per million. Natural concentrations of bromide are much lower. Chloride has commonly been used to trace contaminant plumes that originate at landfills or other industrial facilities. Stream tracing techniques which include the continuous injection of a constant concentration of a tracer (bromide) provide very accurate stream discharge measurements based on dilution of the tracer. The application of surface water tracing techniques in combination with ground-water tracing provides detailed information on ground-water inflow zones to streams.

Organic Fluorescent Dyes - Fluorescent dyes constitute some of the most analytically sensitive, versatile, non-toxic and inexpensive artificial water tracers available. Commonly used fluorescent dyes used in ground-water investigations include: uranine, rhodamine, eosin and phloxine, and sulpho-rhodamine B. A single dye may have more than one commercial name and several different dyes may have the same commercial name. The most complete reference for fluorescent organic dyes is The Colour Index (The Society of Dyers and Colourists, 1971, 1999). Dyes are listed and described by color and by chemistry using both the Color Index (C.I.) constitution number and the C.I. generic name.
Most fluorescent dyes are detectable at low concentrations and will work well in waters where pH is near neutral. In acidic conditions the fluorescence of some dyes is minimized. However these dyes will fluoresce again if the pH of the sample is adjusted to more alkaline conditions (Davies personal, communication). Commercial grade, organic, fluorescent dyes can be purchased as a liquid compound or as a powder. Uranine, eosin and phloxine are FDA approved. Many organic dye and salt tracers have been approved for use in aquifers and streams which are used to obtain drinking water supplies. However some States, such as Wisconsin, have restricted the use of all artificial tracers in ground water.

FIELD/ANALYTICAL TECHNIQUE(S): The usefulness and appropriateness of a ground-water tracer test depend on the questions to be answered by the hydrogeologic investigation. Tracer tests are appropriate when ground-water flow velocities are such that results will be obtained within a reasonable period of time -usually less than a year. The usefulness of tracer test results are highly dependent on proper test design (particularly determination of sampling locations) and execution, the nature of the tracer, the ability to detect the tracer at low concentrations, and correct interpretation of recovery data. Prior to conducting a tracer test it is very important to use other geologic and

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hydrologic data and information to develop a fundamental understanding of the hydrogeologic setting and the ground-water flow system to be traced. This understanding can then be used to: determine the appropriate type of tracer; determine the tracer injection location and method; determine appropriate sample collection locations; and / or determine which stable or radioactive isotopes should be included in the sampling plan. It is always advisable to sample more locations rather than fewer locations. It is also important to know precisely how much tracer mass is injected. This will allow for a determination of the percent of tracer mass recovered at a given sampling location if the flow can be measured at the sampling location. This quantitative aspect of tracing can be important in helping to evaluate the significance of any given ground-water flow path.

Isotopes - Chapter 10 of Clark and Fritz (1997) includes an excellent discussion and comparison of sampling and analytical protocols and procedures for collection of water samples for isotopic analysis. Sample size, filtering, preservation, container type, holding times and method of analysis vary quite a bit between different stable and radioactive isotopes. In general isotopes of water (oxygen 18, deuterium, tritium), have simpler sampling protocols than isotopes of dissolved inorganic and organic carbon (carbon 13 and carbon 14), dissolved sulfate and sulfide (sulfur 34) , dissolved gases(helium, argon 39, krypton 85) and dissolved uranium (uranium 234 and uranium 238). Water samples for isotopic analyses must be collected and stored in well-sealed bottles. This is necessary to prevent any additional fractionation.

Organic Dyes - The use of organic dyes as hydrologic tracers requires specific field sampling and analysis procedures. Careful thought should be given to the selection of the proper dye and the method used to introduce the dye into the ground-water. Dye selection is based on the chemical and toxicity characteristics of the dye. For example, recent research from Germany indicates that rhodamine is somewhat toxic. Also, fluorescence is reduced in some dyes when dissolved in low pH waters and some dyes will fluoresce better in cooler waters. All of these factors should be considered when choosing an organic dye for use as a tracer.

Tracer injection - It is important to carefully consider the best way to introduce the dye tracer into the ground-water system. There are five common methods:

- Inject into a previously constructed or a new well - making sure the well will take water prior to introducing a dye tracer, which can be ascertained by conducting simple aquifer tests using the proposed injection well.
- Inject into a stream - make sure the stream gradient is low and the reach of stream below the injection point is a losing reach.
- Inject into a constructed trench - making sure the trench takes water
- Inject into a sinkhole - in karst terrain
- Inject into a mine shaft

Sample collection protocols for water samples that may contain the dye tracer are relatively simple. Sample containers and storage should minimize all exposure to light to prevent degradation of the dye. Samples do not require filtering or preservation, but water

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samples that may contain dye should be kept cool until analysis is complete. They should be analyzed within 2 weeks to minimize bacterial degradation of the dye.
Collection of water samples can be done using an autosampler (useful for collecting many samples in a short time and from locations with difficult access) or by grab sampling. Once the water samples are collected, they should be analyzed on a spectrofluorometer to confirm the nature of the fluorescence and then analyzed with wet chemistry methods for dye concentration. It is important to conduct both types of analysis to better confirm the presence of the dye that was injected. Sophisticated sampling techniques can be achieved by using flow - through flourometers which measure the fluorescence of the dye in water on a real time basis. This type of sampling requires a power source and data loggers. Portable, dye specific, submersible fluorometers are also available for installation in wells.
Small bags of activated charcoal can also be used to detect dyes. Organic dyes will sorb onto charcoal if water that contains dye comes into contact with the charcoal. Charcoal bags are placed in water at sampling locations and then retrieved for analysis at selected time intervals. It is important to note that determining the travel time from an injection location to a given charcoal bag is constrained by the time interval between retrieval of the bags.
The most important rule of thumb for sampling is to collect samples often at many places. Collecting samples for organic dye analysis is relatively easy and inexpensive. Since it is not always possible to predict all locations where dye may be recovered, it is best to have more rather than fewer sampling locations. By collecting samples frequently, at important locations, the data can be used to construct breakthrough curves of recovery versus time. Detailed breakthrough curves can be used to do rigorous analyses of the recovery data.

LIMITATIONS/INTERFERENCES: Tracing techniques have been demonstrated to be especially useful in fractured rock and karst hydrogeologic settings. Ground-water tracing techniques make fewer assumptions about hydrogeological conditions than do hypothetical (e.g. Darcy) or numerical simulations and are therefore often more reliable.

DOCUMENTATION/DATA HANDLING:
Chain-of-custody. All samples are subject to normal USEPA guidelines on chain-of custody. USEPA chain-of-custody tags are placed on each sample by USEPA field personnel.
Preservation methods. All water samples are refrigerated at 5°C prior to sample analysis. No additional preservation is needed.
Maximum holding time. Samples are shipped to analytical labs within one-week of collection. Sample analysis for isotope samples is generally initiated within four weeks of being received by the analytical laboratory. Stable water isotopes and tritium samples have a maximum storage time of several years when refrigerated.

BIBLIOGRAPHY:
ASTM, 1996, Volume 4.09 Soil and Rock, D 5717-95, Guide for the Design of Ground-Water Monitoring Systems in Karst and Fractured Rock Aquifers
Clark, I. D., Fritz, P., 1997, Environmental Isotopes in Hydrogeology, CRC Press LLC, Boca Raton, Florida, USA, 328 p.

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Davies, Gareth, 2001, Cambrian Groundwater, personal communication
Kendall, C., McDonnell, J.J., 1998, Isotope Tracers in Catchment Hydrology, Elsevier, Amsterdam, the Netherlands, 839 p.
Society of Dyers and Colorists & American Association of Textile Chemists & Colorists, 2009 "The Colour Index," 4th Edition
Tremblay, G.A. and Hogan, C.M., eds. 2001, MEND manual, volume2—sampling and analysis: MEND document 5.4.2b, 160 p.

SOLIDS-GENERAL INFORMATION, PREPARATION, MEASUREMENT, AND COLLECTION TECHNIQUES

Country Rocks, Overburden, Ore, Sub-ore, Subsurface, Soils Samples

(Virginia T. McLemore)

MEDIA: Country rocks, overburden, rock outcrops, altered areas, drill cuttings, drill core, subsurface samples, soils

SAMPLING OBJECTIVES: The science of sampling rocks, subsurface samples, soils etc. is complicated. Each solid sample type, may have multiple sampling strategies depending on the particular goals of the study/QAPP. Obtain a representative sample of each zone or lithology to define and understand the geological, geochemical, and hydrological processes involved in the project area. Required for petrographic analysis (including thin section and microprobe analysis). Determine lithology, texture, mineralogy, and general solid chemistry. Samples can be used in leaching tests. Determine extent of alteration, weathering, and mineralization.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUES: Plastic bags with twist ties or zip lock plastic bags are preferred for sample containers to prevent contamination in most cases. Many cloth bags contain additives to enhance their strength, therefore, cloth bags that could contaminate the sample shall not be used for sample containers. In cases where plastic bags are not appropriate, other containers will be used as specified in the project SOPs and on the field sample forms (for example, paper or cloth for moist samples, buckets for large samples). The sampling will focus on selected outcrops which represent specific rock types, mineralization, and alteration assemblages. Clean the rock surface with a hammer to avoid superficial alteration materials and collect 3-5 kg of rock using hammer and chisel. Take GPS survey reading and record it along with coordinate system used. Take a photograph of the outcrop using a bar scale. Record the photo number with the sample number and the GPS location on the sample form and in your field logbook. Fill out field sample form and chain of custody form, if required.

SPATIAL OR TEMPORAL CONSIDERATIONS: Determine the lithology (rock type) and visible mineralogy in the field with a hand lens and sample a spectrum of the lithologies, different mineralogy, alteration, and other characteristics. Many ore deposits are surrounded by zones of different alteration assemblages and each zone should be sampled and characterized. Many of these alteration assemblages could vary in the degree of alteration and each degree should be sampled.

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ANALYTICAL TECHNIQUES: Specific types of samples can be collected and designated as *grab* (single rock piece or a sample from a confined area), *composite* (small chips of uniform material collected over a specific defined area), *chip or channel* (small chips of rocks collected over a specified interval), *high grade* (small chips or a piece of the mineralized material and separated from the low grade or nonmineralized material).

CONTAINERS: plastic bags, plastic jars w/lids, 5-gallon buckets w/lids, 55-gallon drums, other bags

CONTAINER PRECLEANING: Containers should be dry, free of grease or visible contaminants; no special precleaning is required.

PRESERVATION: In some projects, care should be taken to protect them from oxidation by taking measures such as enclosing samples in sealed containers (under nitrogen if possible) or, better yet, collecting them under nitrogen followed by freezing. Wet samples should be immediately air-dried or dried at temperatures <40°C.

HANDLING AND PREPARATION: none generally required except for specific analyses

ADDITIONAL CONSIDERATIONS: Prior to sampling all safety precautions for using hammers and chisels will be reviewed. In addition, care should be taken at sample locations to minimize the risk of rock falls while sampling. Samples should be taken from areas with good footing to prevent personnel falls. Water in some areas could be very acidic and extra care should be exercised near natural seeps. Archiving a portion of the sample is recommended. If the sample is wet, homogenize the sample in separate containers and take representative portions from each container to make approximately 1000 grams for bagging (or whatever size the project requires). Use water and paper towels to decontaminate the equipment after each wet sample.

BIBLIOGRAPHY:

ASTM Method D5956, 2006, Standard guide for sampling strategies for heterogeneous wastes: West Conshohocken, PA, ASTM International, DOI: 10.1520/D5956-96R06, www.astm.org. accessed 8/23/13.

ASTM Method D6044, 2009, Standard guide for representative sampling for management of waste and contaminated media: West Conshohocken, PA, ASTM International, DOI: 10.1520/D6044-96R09, www.astm.org. accessed 8/23/13.

ASTM Method D6051, 2006, Standard guide for composite sampling and field subsampling for environmental waste management activities: West Conshohocken, PA, ASTM International, DOI: 10.1520/D6051-96R06, www.astm.org. accessed 8/23/13.

McLemore, V.T. and Frey, B.A., 2009, Appendix 8. Quality control and quality assurance report (Task B1): New Mexico Bureau of Geology and Mineral Resources, Open-file Report OF-523, 65 p.,

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http://geoinfo.nmt.edu/publications/openfile/downloads/OFR500-599/500-525/523/Appendix8_QAQC.pdf, accessed 8/23/13

Sediment

(Virginia T. McLemore)

MEDIUM: lake and stream sediments, bottom sediments

SAMPLING OBJECTIVES: Sediments collected for mineralogy, general sediment chemistry and for physical characteristics such as particle size distribution and particle shape. The essential tasks in sediment sampling are to collect representative, undisturbed samples that meet the requirements of the program, and to prevent deterioration and contamination of the samples before analyses.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUES: Three types employed are (1) hand trowel or shovel, (2) grab samplers for collecting surface sediments, which provides material for the determination of horizontal distribution of variables; and (3) core samplers or augers for collecting a depth profile of sediments, which provides material for determination of vertical distribution of variables. Continuous core sediment samples are collected using hand auger at a frequency of a maximum of one sample per site. Discrete sediment samples are collected from desired depths using hand auger.

SPATIAL OR TEMPORAL CONSIDERATIONS: The active stream sediment represents the fine- to medium-grained bed load material (silty-clayey-sandy), which is transported by running water. The active stream sediment is collected from the project-specified stream order in a defined drainage basin. A drainage basin is an area of water drainage with a network of streams like the branches of a tree; smaller streams join together leading into larger and larger streams. The sample depth is project specific.

Mechanical erosion of the soil and rock material in the drainage basin results in their breakdown. Small particles, minerals, and other material form the suspended load of the stream, which is kept in suspension by turbulence of the water. Turbulence is greatest in steeper areas where the stream water flows faster. Downstream where the topography is gentler, the stream water move slower and turbulence decreases, which causes the suspended load to settle out, resulting in deposition of the material in the stream sediments. Heavy minerals, like ore minerals, tend to drop out first because less turbulence is needed to keep them in suspension. Some projects could designate specific portions of the stream to be sampled, such as overbank, sand bars, etc.

ANALYTICAL TECHNIQUES: Some project could require dry or wet sieving/screening the stream sediment sample to a specified size-fraction. Stainless steel or nylon sieves are generally recommended to avoid contamination. Other projects could require collection of heavy-mineral concentrates or coarse material only, generally using a pan or other gravimetric methods. Metal-free polyethylene or wooded collectors and funnels should be used to avoid contamination of the sample.

CONTAINER: plastic bags, plastic jars or glass w/lids, 5-gallon buckets w/lids, 55-gallon drums, other bags

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CONTAINER PRECLEANING: Containers should be dry, free of grease or visible contaminants; no special precleaning is required.

PRESERVATION: Keeping the samples cool is desired. Some specific chemical analyses could require additional preservation techniques. In some projects, care should be taken to protect them from oxidation by taking measures such as enclosing samples in sealed containers (under nitrogen if possible) or, better yet, collecting them under nitrogen followed by freezing. Wet samples should be immediately air-dried or dried at temperatures <40°C. Sediment samples also need to be dried immediately at ambient or low heat, and loosely packed into the sample container.

HANDLING AND PREPARATION: No jewelry or other potential material should be worn that could contaminate the sample.

ADDITIONAL CONSIDERATIONS: Sampling from a boat or during winter could require additional considerations.

BIBLIOGRAPHY:

http://ilmbwww.gov.bc.ca/risc/pubs/aquatic/lake-stream/index.htm, accessed 8/23/13.

Fischenich, C. and Little, C., 2007, Sediment sampling and analysis for stream restoration projects: U.S. Army Corps of Engineers report ERDC TN-EMRRP-SR-39, 23 p., http://el.erdc.usace.army.mil/elpubs/pdf/sr39.pdf, accessed 8/23/13.

http://www.clu-in.org/download/contaminantfocus/sediments/methods-for-collection-epa-manual.pdf, accessed 8/23/13.

Suspended Sediment

(Kathleen S. Smith)

MEDIUM: Water (streams, rivers, lakes)

SAMPLING OBJECTIVES: Suspended-sediment measurement involves sampling a water-sediment mixture to determine the suspended-sediment concentration (and sometimes the particle-size distribution) at a given cross section of a stream. The suspended-sediment concentration can be combined with water-discharge measurements to determine the suspended-sediment load of a stream. Suspended-sediment load can be used to assess the effect of land-use changes and engineering practices, to monitor the movement of contaminants sorbed to sediments and rates of erosion, or to meet regulatory requirements (e.g., Total Maximum Daily Loads (TMDLs)).

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUES: The choice of sediment-sampling methods and equipment depends on the objectives and budget of the study. The U.S. Federal Interagency Sedimentation Project (FISP; see http://water.usgs.gov/fisp/ , accessed 8/23/13.) provides oversight and guidelines on sediment sampling equipment and collection techniques. Wren et al. (2000) and Edwards and Glysson (1999) provide

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detailed discussions of a variety of suspended-sediment methods and tools. The most commonly used suspended-sediment methods that provide a physical sample include:

(1) **Bottle-sampling methods** currently are the most widely used methods to obtain suspended sediment samples. These methods require that personnel be present to collect the samples, which limits the amount of temporal variation data that can be obtained. Bottle-sampling methods have good spatial resolution but only sample to within about 9 cm above the streambed. Suspended-sediment bottle samplers should be used with specialized nozzles designed to match the speed of the water in the stream with the speed of the water entering the nozzle. There are two main types of bottle-sampling devices, including:
    (a) depth-integrating samplers, which are lowered and raised through the water column at a uniform rate, and
    (b) point-integrating samplers, which are designed to open and sample at a particular point or portions of a given vertical in the water column.
(2) **Single-stage samplers** are designed to fill a sample container when the stream stage reaches a particular height in remote locations.
(3) **Pump-sampling methods** generally are automated methods that are able to address temporal variations. A sediment/water sample is collected by applying a vacuum to tubing within a stream channel, which pulls the sample into a collection device (often similar to the bottle-sampling devices).

Common suspended-sediment methods that do not provide a physical sample include:

(1) **Acoustic methods** are relatively new methods that can be remotely deployed and provide good spatial and temporal resolution. These methods do not work well at high particle concentrations. Acoustic methods represent a non-intrusive approach that are good for determining suspended-sediment concentration and particle size distribution in the water column for situations when it is not necessary to obtain physical samples.
(2) **Focused laser beam reflectance methods** provide a one-point measurement. This method does not work well at low particle concentrations, high particulate organic matter concentrations, or for particles that greatly deviate from a spherical shape.
(3) **Optical methods**, which can be remotely deployed for temporal resolution, are relatively inexpensive but provide a one-point measurement.

Handheld samplers can be used in wadable streams or from a low bridge or cableway. Samplers also are designed for use with cables and reels when necessary. Bedload is defined as sediment that moves by sliding, rolling, or bouncing on or near the streambed, and bed material is defined as the sediment mixture of which the bed is composed. There are samplers available that specifically sample the bedload or bed material. Bedload samplers are designed to sample particulates carried by a stream on or immediately above the bed, whereas bed-material samplers are designed to collect sediment samples from bed material of a stream, lake, or reservoir with varying depths of penetration depending upon the design of the sampler. There also are core samplers for collecting streambed material, runoff samplers, and various types of erosion monitors.

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SPATIAL OR TEMPORAL CONSIDERATIONS: Suspended-sediment concentration in a stream can vary from the water surface to the streambed and laterally across the stream. Suspended fine sediment tends to be fairly well mixed across a stream cross section; however, sand-size particles (and larger) tend to have large fluctuations both temporally and spatially. Concentration generally increases from a minimum at the water surface to a maximum at or near the streambed. Particles larger than sand size will move as bed load in most streams.

Temporal variation in suspended-sediment concentrations can fluctuate rapidly, and most sediment is carried during episodic events. It is difficult to obtain suspended-sediment concentrations unless some type of automated system is used. Water discharge has been used as a surrogate for suspended-sediment concentration. However, suspended-sediment concentration commonly is out of phase with changes in discharge (e.g., Rieger and Olive, 1984). Lewis and Eads (2009) describe a relatively new approach using turbidity as a surrogate for suspended-sediment concentration.

ANALYTICAL TECHNIQUES: Laboratory analysis of water samples for suspended-sediment concentration generally involves filtration or evaporation methods. For filtration, the sample is passed through a pre-weighed glass fiber filter in a Gooch crucible. The sample on the filter is dried at about 103 to 110°C for a specified time until loss of all visible water, cooled in a desiccator at room temperature, and weighed.

ADDITIONAL CONSIDERATIONS: There is a lack of uniformity in names of sediment-size fractions. The standard and nomenclature commonly used is:
Clay, which is less than 1/256 mm,
Silt, which is less than 1/16 mm and greater than 1/256 mm,
Sand, which is less than 2 mm and greater than 1/16 mm, and
Gravel, which generally refers to all sediment sizes larger than coarse sand.

Possible sampling errors can be caused by using too small a nozzle for the bottle-sampling methods. These errors usually are minor when dealing with fine material, but tend to increase in importance with increasing particle size.

BIBLIOGRAPHY:

ASTM D5907. 2010. *10 Standard Test Methods for Filterable Matter (Total Dissolved Solids) and Nonfilterable Matter (Total Suspended Solids) in Water*. West Conshohocken, PA: ASTM International. Available from www.astm.org. Accessed 8/23/13.

Caux, P.Y., Moore, D.R.J., and MacDonald, D. 1997. *Sampling Strategy for Turbidity, Suspended and Benthic Sediments*. Technical Appendix Addendum prepared for BC Ministry of Water, Land and Air Protection by Cadmus Group Inc. and MacDonald Environmental Sciences Ltd. www.env.gov.bc.ca/wat/wq/BCguidelines/samp_strat/sampstrat.html. Accessed 8/23/13.

Danielsson, L.G. 1982. On the use of filters for distinguishing between dissolved and particulate fractions in natural waters. *Water Res*. 16:179–182.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Eaton, A.D., Clesceri , L.S., Rice, E.W., Greenberg, A.E., and Franson, M.A.H. (eds.). 2005. *Standard Methods for the Examination of Water and Wastewater*, 21st ed. Washington, DC: American Public Health Association, American Water Works Association, and Water Environment Federation.

Edwards, T.K., and Glysson, G.D. 1999. *Field Methods for Measurement of Fluvial Sediment*. Techniques of Water-Resources Investigations, book 3, chap. C2. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/twri/twri3-c2/. Accessed 8/23/13.

Federal Interagency Sedimentation Project (FISP)—U.S. Geological Survey. 2012. http://water.usgs.gov/fisp/. Accessed 8/23/13.

Guy, H.P. 1977. *Laboratory Theory and Methods for Sediment Analysis*. Techniques of Water-Resources Investigations, book 5, chap. C1. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/twri/twri5c1/. Accessed 8/23/13.

Lewis, J., and Eads, R. 2009. *Implementation Guide for Turbidity Threshold Sampling: Principles, Procedures, and Analysis*, Gen. Tech. Rep. PSW-GTR-212. Albany, CA: U.S. Department of Agriculture, Forest Service, Pacific Southwest Research Station. www.fs.fed.us/psw/publications/documents/psw_gtr212/psw_gtr212.pdf. Accessed 8/23/13.

Nolan, K.M, Gray, J.R., and Glysson, G.D. 2005. *Introduction to Suspended-Sediment Sampling*. Scientific Investigations Report 2005-5077 (CD-ROM). Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/sir/2005/5077/. Accessed 8/23/13.

Rieger, W.A., and Olive, L.J. 1984. The behavior of suspended sediment concentrations during storm events. In R. Loughran (ed.), *Drainage Basin Erosion and Sedimentation*. New South Wales, Australia: Univ. Newcastle, p. 121–126.

Wren, D.G., Bennett, S.J., Barkdoll, B.D., and Kuhnle, R.A. 2000. *Studies in Suspended Sediment and Turbulence in Open Channel Flows*. Research Report No. 18. Oxford, MS: U.S. Department of Agriculture, National Sedimentation Laboratory. www.ars.usda.gov/SP2UserFiles/Place/64080510/TechnicalReports/NSLTechnicalReport18.pdf. Accessed 8/23/13.

USEPA (U.S. Environmental Protection Agency). 1971. *Method No. 160.2—Residue, Non-Filterable (Gravimetric, dried at 103-105 C)*. U.S. Environmental Protection Agency Methods for the Chemical Analysis of Water and Wastes (MCAWW), EPA/600/4-79/020.

Crushing/Grinding

(Virginia T. McLemore)

METHOD: Describes crushing and grinding techniques of solid samples using standard jaw crushers and disc grinders.

FUNCTION: Solid samples must be crushed for chemical and other analyses.

SPECIFIC EQUIPMENT/TECHNIQUE(S):

- Jaw crushers and catch bins
- Tungsten carbide disc grinders
- Paper
- Spatula

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- Envelopes for crushed sample
- Sample preparation forms
- Indelible marker pens to label samples and fill out forms
- Deionized water
- Acetone in squirt bottle
- Shallow tin trays
- Compressed air and hose assembly
- Wire brush
- Silica sand (laboratory grade, clean and pure)
- Appropriate sample splitting equipment

PROCEDURE: Prior to crushing, air-dry samples in shallow tin trays if they are damp. Run silica sand in disc grinders in between samples to prevent cross contamination of samples. Rinse components of grinder with acetone. If applicable, homogenize fine-grained material and split out 50 grams using cone and quarter method to be used for DI leach. Crush rock samples using large jaw crusher (if necessary), then use small jaw crusher until particles are pea sized. Homogenize. Crush pea-size sample in tungsten carbide disc grinder (hockey puck) to approx. 100 mesh (feels like talcum powder between fingers). Homogenize sample using cone and quartering techniques. Split sample into three—one to chemical laboratory for analysis, one to X-ray laboratory, and one to Bureau archive. Archive remaining pea sized material. Clean the jaw crushers and disc grinders with compressed air. Use a clean wire brush if necessary. Run silica sand in disc grinders in between samples to prevent cross contamination of samples. Rinse with acetone. NOTE: Wash hands periodically. Follow normal procedures to prevent contamination. Maintain field number throughout sample preparation.

LIMITATIONS: none

ADDITIONAL CONSIDERATIONS: Cleaning the equipment with silica sand and acetone is necessary to prevent contamination.

BIBLIOGRAPHY:

Walsh, M.E., Ramsey, C.A., and Jenkins, T.F., 2002, The effect of particle size reduction by grinding on subsample variance for explosive residues in soil: *Chemosphere*, v. 49, p. 1267-1273.

Sizing/sieving

(Virginia T. McLemore)

METHOD: Weathering of minerals involves mostly surface reactions and the rates of these reactions depend upon the available reactive surface area of the mineral. Mineral surface area is dependent on the mineralogy, the extent to which the mineral is liberated from the rock matrix, particle size (especially mineral grain size), and the surface morphology (i.e. roughness of the mineral surface; Lapakko et al., 1998; Lapakko, 2003; McLemore et al., 2009). Furthermore, when mineral surfaces are covered with coatings, such as iron oxyhydroxides, oxidation and dissolution of the mineral can be inhibited.

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Thus, particles of different sizes expose different proportions of the surfaces for chemical reaction, which plays an important role in weathering.

Many studies have examined the variations in composition between different particle size fractions. Strömberg and Banwart (1999) showed large differences in weathering rates between fine waste rock material and larger particles (diameters >0.25 mm) and found that waste rock with diameters less than 0.25 mm (27% of the total mass) accounts for 80% of both the sulfide and silicate weathering. Taboada et al. (2006) showed that Zr and Ti behave differently in residual weathering soil profiles; Zr is enriched in the silt fraction, whereas Ti is enriched in the silt and clay fractions and depleted in the sand fraction. Dultz (2002) cautions that normally feldspars are found in the coarser fractions of soils, but breakdown of feldspar grains by physical weathering could increase the surface area and therefore, increase the reactivity of the grains to weathering. Dultz (2002) also cautions that the parent material affects the weathering of feldspars. Brandvold and McLemore (1998) and McLemore et al. (1995) found that the smallest size fractions (<63 μm) of stream sediments contained the largest concentrations of metals (Cu, Pb, Zn) in the Pecos and La Bajada Rivers in northern New Mexico. Dinelli et al. (2007) found that the effects of grain-size distribution on mineralogy and geochemistry are greater in sediments that have not undergone diagenesis, compaction, or lithification. Some chemical indices are not affected by grain size distribution, including Ni/Al, Cr/Al, and Mg/Al and other indices are affected only by the coarse fraction, including SiO2/Al2O3, Na/Al, Zr/Rb, Zr/V, Y/Rb, Y/V, and Ti/V (Dinelli et al., 2007).

Graf (2008) found an increase in FeO, CaO, and SO_4 between the coarse- and fine-size fractions in samples from the Hansen alteration scar in the Questa area, New Mexico. He attributed the increase in CaO and SO_4 to the precipitation of gypsum. Silicate dissolution can be recognized from coarse- to fine-size particles, as indicated by the decrease in Na_2O and K_2O, and the large change in total feldspar from coarse- to fine-size fractions. The clay mineralogy was consistent between the coarse- and fine-size fractions in these samples.

MEDIUM: solid samples (soils, stream sediments, rocks, etc.)

MEASUREMENT OBJECTIVES: To determine differences in mineral and chemical composition between different grain sizes

SPECIFIC SAMPLE EQUIPMENT/TECHNIQUES: Use standard screens to size the sample. An example of a particle size analysis is in Table A3.8.

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TABLE A3.8. Example of a particle size analysis.

Maximum Particle Size	Minimum Weight of Sample (g)
3 inches	6000
2 inches	4000
1 inch	2000
½ inch	1000
Finer than No. 4 sieve	200
Finer than No. 10 sieve	100

ANALYTICAL TECHNIQUES: Solid samples are collected in the field and analyzed in the laboratory, similar to other solid samples. Samples can be sieved in the field if desired. Samples are selected based upon the project data quality objectives.

LIMITATIONS/INTERFENCES: Not all samples will have enough fines or clay-size material, which could require large amounts collected.

BIBLIOGRAPHY:

Brandvold, L.A. and McLemore, V.T., 1998, A study of the analytical variation of sampling and analysis of stream-sediments from areas contaminated by mining and milling: Journal of Geochemical Exploration, v. 64, p. 185-196.

Dinelli, E., Tateo, F., and Summa, V., 2007, Geochemical and mineralogical proxies for grain size in mudstones and siltstones from the Pleistocene and Holocene of the Po alluvial plain, Italy; in Aiibas, J., Critelli, S., and Johnson, M.J., eds., Sedimentary province and petrogenesis: Perspectives from petrography and geochemistry: Geological Society of America; Special Paper 420, p. 25-36.

Dultz, S., 2002, Effects of parent material and weathering on feldspar content in different particle size fractions from forest soils in NW Germany: Geoderma, v. 106, p. 63-81.

Graf, G.J., 2008, Mineralogical and geochemical changes associated with sulfide and silicate weathering, natural alteration scars, Taos County, New Mexico: M. S. thesis, New Mexico Institute of Mining and Technology, Socorro, 193 p., http://www.ees.nmt.edu/outside/alumni/papers/2008t_graf_gj.pdf, accessed 8/23/13.

Lapakko, K., 2003, Solid phase characterization for metal mine waste drainage quality prediction: Preprint 03-093, 8 p.

Lapakko, K.A., Haub, J., and Antonson, D.A., 1998, Effect of dissolution time and particle size on kinetic test results: Society for Mining, Metallurgy, and Exploration, SME, Littleton, CO, preprint 98-114.

McLemore, V.T., 2012, Geologic Processes Affecting the Chemistry, Mineralogy, and Acid Potential on Particle Size Fractions: Examples from Waste Rock Piles in New Mexico, USA: Proceedings 9th ICARD, Ottawa, Canada, May 20-26, 2012, 12 p., CD-ROM,

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http://geoinfo.nmt.edu/staff/mclemore/projects/environment/documents/0115-McLemore.pdf

McLemore, V.T., Ayakwah, G., Boakye, K., Campbell, A., Donahue, K., Dunbar, N., Gutierrez, L. Heizler, L., Lynn, R., Lueth, V., Osantowski, E., Phillips, E., Shannon, H., Smith, M. Tachie-Menson, S., van Dam, R., Viterbo, V.C., Walsh, P., and Wilson, G.W., 2009, Characterization of Goathill North Rock Pile: New Mexico Bureau of Geology and Mineral Resources, Open-file Report OF-353, 203 p., http://geoinfo.nmt.edu/publications/openfile/downloads/OFR500-599/500-525/523/ofr_523.pdf, accessed 9/16/13

McLemore, V.T., Brandvold, L.A., Hossain, A.M., Pease, T.C., 1995, The effect of particle size distribution on the geochemistry of stream sediments from the upper Pecos River, San Miguel County, New Mexico: New Mexico Geological Society, Guidebook 46, p. 323–329.

Morkeh, J. and McLemore, V.T., 2012, The effect of particle size fractions on chemistry, mineralogy, and acid potential of the Questa rock piles, Taos County, New Mexico: New Mexico Bureau of Geology and Mineral Resources, Open-file Report 545, 96 p., http://geoinfo.nmt.edu/publications/openfile/downloads/OFR500-599/526-550/545/ofr_545.pdf, accessed 9/17/13.

Strömberg, B. and Banwart, S.A., 1999, Experimental study of acidity-consuming processes in mining waste rock: some influences of mineralogy and particle size: Applied Geochemistry, v. 14, p. 1-16.

Taboada, T., Cortizas, A.M., García, C., García-Rodeja, E., 2006, Particle-size fractionation of titanium and zirconium during weathering and pedogenesis of granitic rocks in NW Spain: Geoderma, v. 131, p. 218-236.

http://www.advantechmfg.com/pdf/principles_procedures_manual_with_tables-2.pdf

Splitting/Subsampling

(Kathleen S. Smith)

METHOD: Describes splitting and subsampling particulate earth materials for chemical analysis.

FUNCTION: A subsample is a sample of a sample. Generally only a small portion (subsample) of a sample is needed for analysis. It is essential to obtain a representative subsample of a sample in order to adequately represent the population from which the original sample was obtained. Subsampling is a very important step in the process from field sampling to analysis, and improper subsampling can lead to incorrect interpretations and decisions. Gerlach and Nocerino (2003) state, "Laboratory subsampling errors (e.g., incorrectly taking an aliquot from a sample bottle for analysis) could potentially overwhelm other errors, including other sampling steps and the analytical error, associated with the analyses of samples."

The Gy theory of sampling can be used to minimize subsampling error (see Pitard, 1993). Subsampling error results from heterogeneity (both compositional and distributional) of the sample. There are two practical issues for obtaining a representative subsample: obtaining adequate subsample mass and proper removal of the subsample

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from the sample. The sample mass required for a particular fundamental error can be calculated as:

$$FE^2 = \frac{(22.5)(D^3)}{\text{mass}}$$

Where FE is the fundamental error (a relative variance), D is the diameter of the largest particle (cm), and mass is the mass of the analyzed portion of the sample (g). For a given subsample mass to be analyzed, it is necessary to reduce the particle size of the bulk sample to reduce the fundamental error.

SPECIFIC EQUIPMENT/TECHNIQUE(S): Common methods used for subsampling include sampling with a spoon, coning and quartering, chute splitting, and rotary subsampling. In the spoon method the sample is poured onto a flat surface and several subsamples are collected from different parts of the pile using a spoon. In the coning and quartering method, the sample is poured out in the shape of a cone, flattened, and the cone divided into four equal quarters. One of the quarters is scraped onto another flat surface and the procedure is repeated until the desired subsample mass is obtained. In the chute-splitting method, a special apparatus is used that consists of a series of chutes that feed into two containers on either side of the apparatus. The sample is poured at a steady rate into the chutes, and one of the containers is chosen for further splitting. In rotary sampling (aka spinning riffler or rotary riffle splitter) a special apparatus is used that spins and distributes the sample into a series of containers. Mullins and Hutchison (1982) and Gerlach et al. (2002) found that rotary subsampling and chute splitting are more reliable methods of subsampling than cone and quartering or random spoon sampling. Variability between subsamples depends on subsampler design, the details of the subsampling procedure, and the physical properties of the sample. Gerlach and Nocerino (2003) discuss various subsampling methods and their recommendations for use are listed in Table A.3.9.

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TABLE A3.9 Relative rankings (from best to worst) for subsampling methods according to Gerlach and Nocerino (2003) [N is the number of increments; GE is the grouping and segregation error; N.R. means not recommended; N.A. means not applicable]

Method	Typical Increment Size	Sensitivity to Grouping and Segregation	Moisture Content	Correct Sampling Possible?	Comments
Sectorial. splitter	Very small	Low	Dry	Yes	Method of choice
Paper cone sectorial splitter	Small	Low	Dry	Yes	Performs well
Incremental sampling	Small to medium	Moderate; low with many (N ≥30) correct increments	Dry to moist	Yes, with 1-d pile	Bias still possible, decreases with increasing N and correct sampling
Riffle splitting	Small to medium	Low to moderate	Dry	Yes, takes skill	Possible loss of fines
Alternate shoveling	Medium	Low to moderate	Dry to moist	Yes, if careful	Takes time to prepare from a large lot
Fractional shoveling	Medium to large	Moderate	Dry to moist	Yes, if careful	Performance tied to lot mass
Table sampler	Medium	Moderate to high	Dry	Very difficult; depends on design	High variability; N.R.
Degenerate fractional shoveling	Medium to large	Moderate to high	Dry to moist	Yes, if careful	Performance tied to lot mass; subject to bias; N.R.
Rolling and quartering	Large	High	Dry	Yes, if careful	Highly variable; N.R.
Coning and quartering	Large	High	Dry	Yes, if careful	Usually biased; N.R.
V-Blender	N.A.	High	Dry	N.A.	Problems with GE; N.R.
Vibratory spatula	Small	Very high	Dry	No	Problems with GE; NR
Grab sampler	Variable	Very High	Dry to moist	No	Biased and variable; N.R.

Source: modified from Gerlach and Nocerino (2003; table 8).

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Design of sampling devices also is an important consideration. It is important to note that finer, denser particles tend to settle to the bottom of the sample container or sample pile when poured onto a surface. The best implement for subsampling is a spatula, scoop, or shovel that is square in shape with vertical sides. This design allows all material to have the same chance to be collected. When the implement is rounded, material at the bottom of a poured-out sample is excluded from the subsample (see Figure A3.6).

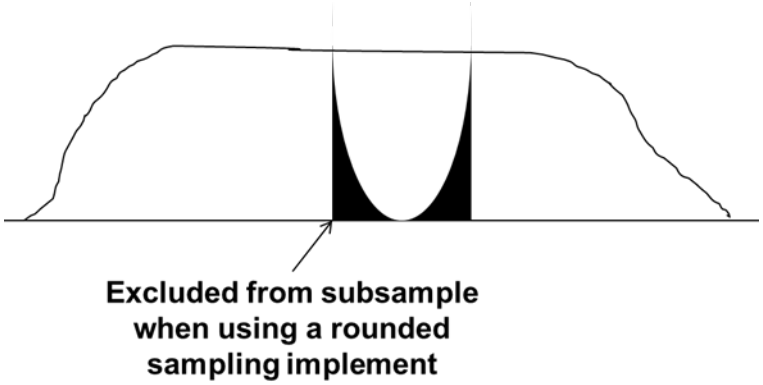


FIGURE A3.6 Cross-section of a sample collected using a rounded implement (modified from Pitard 1993)

BIBLIOGRAPHY:

ASTM D5956. 2006. *Standard Guide for Sampling Strategies for Heterogeneous Wastes*. West Conshohocken, PA: ASTM International. Available from www.astm.org. accessed 8/23/13.

ASTM D6044. 2009. *Standard Guide for Representative Sampling for Management of Waste and Contaminated Media*. West Conshohocken, PA: ASTM International. Available from www.astm.org. accessed 8/23/13.

ASTM D6051. 2006. *Standard Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities*. West Conshohocken, PA: ASTM International. Available from www.astm.org. accessed 8/23/13.

Gerlach, R.W., Dobb, D.E., Raab, G.A., and Nocerino, J.M. 2002. Gy sampling theory in environmental studies. 1. Assessing soil splitting protocols. *J. Chemometrics* 16:321–328.

Gerlach, R.W., and Nocerino, J.M. 2003. Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples. EPA/600/R-03/027. Washington, D.C.: U.S. Environmental Protection Agency.

Gerlach, R.W., Nocerino, J.M., Ramsey, C.A., and Venner, B.C. 2003. Gy sampling theory in environmental studies. 2. Subsampling error estimates. *Anal. Chim. Acta* 490:159–168.

Mullins, C.E., and Hutchison, B.J. 1982. The variability introduced by various subsampling techniques. *J. Soil Sci*. 33:547–561.

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Pitard, F.F. 1993. *Pierre Gy's Sampling Theory and Sampling Practice: Heterogeneity, Sampling Correctness, and Statistical Process Control*, 2nd ed. Boca Raton, FL: CRC Press.

Ramsey, C.A., and Suggs, J. 2001. Improving laboratory performance through scientific subsampling techniques. *Environ. Testing Anal.* 10:12–16.

Walsh, M.E., Ramsey, C.A., and Jenkins, T.F. 2002. The effect of particle size reduction by grinding on subsampling variance for explosives residues in soil. *Chemosphere*. 49:1267–1273.

Dissolution

(Kathleen S. Smith)

MEDIA: Solids (dissolution, digestion)

MEASUREMENT OBJECTIVES: Dissolution of solid samples often is a necessary step to determine trace-element concentrations in earth materials. Solid digestion procedures involve destruction of inorganic and organic fractions using various digestion and oxidation procedures to liberate the elements into solution. The amount of trace elements liberated by digestion methods depends on the properties of each element, element concentration, mode of occurrence, properties of the solid matrix, and the digestion procedure.

SPECIFIC TECHNIQUE(S): Most digestion procedures involve heating a solid sample (usually with grain size reduced to a particle size of <250 μm to increase surface area and reduce variability in subsamples) in contact with a strong acid or a combination of acids in either open or closed vessels. The main heating procedures include boiling with a reflux condenser, heating using a hot plate, heating using a heating or digestion block, and microwave assisted digestion. Since the 1980s microwave-assisted digestion procedures have become very popular. Variables in microwave digestions include power, time, and pressure. General digestion variables include the volume, concentration, and combination of acids, and the reaction conditions (e.g., acid-to-sample ratio, time, temperature, and pressure). In open systems there are risks of atmospheric contamination and loss of volatile compounds. Use of a Teflon bomb offers rapid digestion and is not susceptible to losses of volatile metals.

Some digestion procedures result in complete dissolution of the sample whereas others are less thorough. Common acids used in digestions are HNO_3 and aqua regia (3:1 v/v HCl to HNO_3), both of which usually do not result in complete dissolution of the sample. Nitric acid digestion results in dissolution of many common metals and oxidizes organic matter. Aqua regia digestion procedures are widely used in Europe. Mixture of aqua regia with HF results in more complete dissolution because HF is necessary to dissolve most silica matrices. One reason why relatively incomplete digestion procedures have been used is related to safety concerns involved with using hydrofluoric acid (HF).

USEPA digestion methods include:

- Method 3050B (HNO_3 and H_2O_2),
- Method 3051 (microwave; HNO_3),
- Method 3051A (microwave; HNO_3 and HCl), and
- Method 3052 (microwave; HNO_3 and HF, optional HCl and H_2O_2).

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It is recognized that Methods 3050B, 3051, and 3051A do not recover all metals in samples. Method 3052 uses microwave-assisted digestion for total sample digestion (USEPA, 1996).

The U.S. Geological Survey uses a complete digestion procedure consisting of a mixture of HCl, HNO_3, $HClO_4$, and HF (Crock et al., 1983).

Some additional combinations of acids used for digestions include:

$HCl + HNO_3 + HClO_4$
$HNO_3 + HClO_4 + HF$
$HNO_3 + H_2O_2 + HF$
$HNO_3 + H_2O_2 + HClO_4 + HF$
$HNO_3 + HBF_4$
$H_2SO_4 + HClO_4 + HNO_3 + HF$

Note that digestion procedures using $HClO_4$ require a special fume hood designed for $HClO_4$. Also, $HClO_4$ should not be used in a microwave oven unless that application is approved by the manufacturer.

ANALYTICAL TECHNIQUE(S): The digestion method used should be appropriate for the analytical techniques in accordance with project objectives. Common methods used to analyze digests include atomic absorption spectrometry (FLAAS, HGAAS, CVAAS, GFAAS), inductively coupled plasma emission spectrometry (ICP-OES), and inductively coupled plasma mass spectrometry (ICP-MS).

LIMITATIONS/INTERFERENCES: The use of many digestion procedures may leave portions of the sample undissolved, which can result in poor reproducibility and/or poor accuracy. For determination of elements forming volatile compounds (e.g., Hg, As, Pb) special care must be taken during sample pretreatment, digestion, and analysis. High acid and solute concentrations in the digest may cause interferences with analytical techniques.

BIBLIOGRAPHY:

ASTM D4698. 2007. *Standard Practice for Total Digestion of Sediment Samples for Chemical Analysis of Various Metals*. West Conshohocken, PA: ASTM International. Available from www.astm.org. accessed 8/23/13.

ASTM D5198. 2009. *Standard Practice for Nitric Acid Digestion of Solid Waste*. West Conshohocken, PA: ASTM International. Available from www.astm.org. accessed 8/23/13.

ASTM D5258. 2007. *Standard Practice for Acid-Extraction of Elements from Sediments using Closed Vessel Microwave Heating*. West Conshohocken, PA: ASTM International. Available from www.astm.org. accessed 8/23/13.

Chen, M., and Ma, L.Q. 1998. Comparison of four USEPA digestion methods for metal analysis using certified and Florida soils. *J. Environ. Qual*. 27:1294–1300.

Crock, J.G., Lichte, F.E., and Briggs, P.H. 1983. Determination of elements in National Bureau of Standards geological reference materials SRM 278 obsidian and SRM 688 basalt by inductively coupled plasma-atomic emission spectroscopy. *Geostandards Newsletter* 7:335–340.

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Lamble, K.J., and Hill, S.J. 1998. Microwave digestion procedures for environmental matrices. *Analyst* 123:103R–133R.

Lamothe, P.J., Fries, T.L., and Consul, J.J. 1986. Evaluation of a microwave oven system for the dissolution of geologic samples. *Anal. Chem*. 58:1881–1886.

McQuaker, N.R., Brown, D.F., and Kluckner, P.D. 1979. Digestion of environmental materials for analysis by inductively coupled plasma-atomic emission spectrometry. *Anal. Chem*. 51:1082–1084.

Novozamsky, I., van der Lee, H.J., and Houba, V.J.G. 1995. Sample digestion procedures for trace element determination. *Microchimica Acta* 119:183–189.

USEPA (U.S. Environmental Protection Agency). Variously dated. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846)*: U.S. Environmental Protection Agency. www.epa.gov/wastes/hazard/testmethods/sw846/. accessed 8/23/13.

Method 3050B, 1996, Acid digestion of sediments, sludges, and soils

Method 3051, 1994, Microwave assisted acid digestion of sediments, sludges, soils, and oils

Method 3051A, 2007, Microwave assisted acid digestion of sediments, sludges, soils, and oils

Method 3052, 1996, Microwave assisted acid digestion of siliceous and organically based matrices

Sample preservation

(B. Frey)

MEDIA: Water

OBJECTIVES: To use physical and chemical preservation techniques that slow reactions of inorganic analytes in aqueous samples, thus stabilizing them for transport and storage. To increase the holding times required in standard operating procedures.

PRINCIPLE: Preservation of water samples is necessary because chemical changes that begin as soon as the sample is taken and can affect the analyst's ability to produce quality data. The best procedure to address these changes is to complete analyses immediately; however, the reality of transporting samples from remote sites to the laboratory make this goal difficult, even impossible. For this reason, procedures and chemicals that slow chemical changes are critical to quality analyses.

Common considerations for inorganic sample preservation are cooling and adjusting the sample pH, but there are numerous other procedures for preservation varying from the physical to the chemical. Śliwka-Kaszyńska et al. (2003) call physical techniques "passive preservation" because they retain the sample's original composition. They range from choosing the optimal bottle material, proper cooling (usually to 4°C), filtration with 0.45 µm filters, and blockage of UV rays (with amber bottles, for example).

Chemical additives retard biological and chemical reactions and reduce volatility of some components. A key consideration is whether the chosen preservative can affect analyses adversely. As a fail-safe, contact the laboratory that will be receiving the

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samples and ask for the laboratory's recommendation for preservation. Additionally laboratories often provide sample bottles and materials to preserve the samples.

HANDLING AND PREPARATION: Sample cooling is used to slow reactions for nearly all parameters in water samples. The recommended temperature for transport and storage is about 4°C. If chemical means are used to preserve the samples, the chemicals should be added when the sample is collected. It is desirable, especially with volatile parameters, to overfill the sample bottle and cap with no headspace (no air at the top when closed). Pack samples in ice or with ice substitutes before transporting. Avoid dry ice, which may affect the pH of samples.

Some constituents, such as nitrogen species, can be affected by microbial activities and have very short holding times (hours). Keeping samples cool and in the dark can retard microbial activities. Using chloroform can prevent microbial growth, but it can also adversely affect analyses. Sulfuric acid is a recommended preservative to prevent changes among nitrogen species, but can also affect the analyses on some instruments. Contact the laboratory to determine the preservatives that can work with its methods.

When acidifying samples for trace metal analysis, consider whether total suspended solids or total dissolved solids analysis is required. If dissolved solids are the target, the acid should only be added after filtration to 0.45 μm. For suspended solid analysis, the sample should not be filtered before acidification; in this case, the samples should be held for 16 hours before analysis (see below).

Eaton et al (2005) and Sliwka-Kaszyńska et al. (2003) give more complete discussions of water sample preservation. Both have compiled thorough tables that list recommended preservation, holding times and information sources.

SAFETY CONSIDERATIONS: Concentrated acids can cause severe burns, and many chemicals can present inhalation hazards. Workers who will use chemicals in the field should review the material safety data sheets (MSDS) for them before heading to the field and should obtain the proper chemical handling gear, such as gloves and safety glasses.

MATERIALS: Coolers, ice or ice packs, appropriate sample bottles, dropper bottles with ultrapure nitric acid, sulfuric acid or other chemicals.

LIMITATIONS: Because of the dangers presented by acids, some field conditions may make chemical handling undesirable. Constituents susceptible to biochemical alteration to other species, such as changes among nitrogen species, are most successfully preserved in the field. Other preservation, such as acidification for trace metal analyses, can be performed on samples after arrival in the laboratory. In such cases, USEPA method 200.8 (see bibliography) recommends adding nitric acid 16 hours before trace metals analysis, particularly for samples requiring total suspended solid analyses. Alteration to trace metal concentrations can include adsorption to the bottle or precipitation within the bottle, which can be reversed with pH adjustment before total analysis. See Śliwka-Kaszyńska et al. (2003) for a discussion of metals speciation considerations.

Some users find ice works better than ice substitutes to cool samples to near freezing; however, water samples should be packaged in such a way that they are not

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immersed or floating in the water resulting from melted ice. Melt from ice melts can infiltrate poorly sealed containers.

Certain parameters can change within minutes and should be analyzed in the field: these include temperature, pH and dissolved gases.

Chemical additives can introduce contaminants to the samples. Purity must be considered when choosing a chemical for preservation.

BIBLIOGRAPHY:

Eaton, A.D., Clesceri, L.S., Rice, E.W., Greenberg, A.E., (eds.), 2005, Standard Methods for the Examination of Water & Wastewater, 21th Edition: American Public Health Association, 1100 p.

Sliwka-Kaszyńska, M., Kot-Wasik, A., Namieśnik, J., 2003, Preservation and Storage of Water Samples: Critical Reviews in Environmental Science and Technology, v. 33, issue 1, p. 31-44.

U.S. Environmental Protection Agency, 1994, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometry. Method 200.8, Revision 5.5. Methods for the Determination of Metals in Environmental Samples, Supplement I, EPA/600/R-94-111, Environmental Monitoring Systems Laboratory Office of Research and Development.

Sample drying

(B. Frey)

MEDIA: Soil, plants, sediments and rock

OBJECTIVES: To use drying techniques to optimize subsequent analyses of solids without compromising critical tests. To remove the water from solids that is not part of the molecular or crystal structure of the sample material.

PRINCIPLE: When preparing samples for analytical measurements, it is critical to know the effect drying will have on the analysis. Each matrix type requires different considerations.

For rocks and soil, one of the primary purposes of drying is to allow easier handling during preparation techniques such as crushing, mixing, sieving or grinding. Drying considerations for rocks samples are minimal; most authors find that simple air drying of rock samples before crushing is sufficient, and oven drying is deterred.

The hygroscopic nature of some soils might require oven drying if sample grinding is required before analyses, as high clay samples might cake during grinding. As mentioned earlier, it is critical to understand what effect oven temperatures will have on subsequent analyses, including analyses of volatile elements such as mercury (see mercury analysis section for details). Sample splits may be required for samples that will undergo multiple analyses requiring very different handling. Bulk density, for example, requires in situ volume measurements followed by later oven drying in the lab; a separate field split is recommended. In another case, soil moisture measurements usually use an oven temperature of 105°C, but the crystal water of gypsum is driven off at this temperature; separate splits should be made. (See Nelson et al. 1978) for details of gypsum determination.)

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For plant samples, oven drying prevents chemical and biological decomposition that can result when plants are stored with moisture.

HANDLING AND PREPARATION:
Rocks: Air drying is usually sufficient for all rock preparation and analytical procedures.

Soils: The ideal result when drying soils is to allow hand crushing to a uniform particle size. Air drying may achieve this. Although several authors recommend oven drying soil samples at 100 to 110°C before total elemental analysis of soils (Shugar et al., 1973; Huat Lim and Jackson, 1982), James and Wells (1990) recommend much lower temperatures (35-50°C) to prevent the loss of crystalline waters .

Plants: Before drying plant samples, soil and other possible contaminants should be washed from the plant matter. Jones and Case (1990) recommend drying plant samples as quickly as possible after collection in a force-draft electrical oven. Further drying is recommended after grinding the samples to a constant weight between 65 and 90°C. See Jones and Case for a more complete discussion.

See the appendix section on mercury analysis for a discussion of drying samples before mercury analysis.

MATERIALS: Balance, desiccator with fresh desiccant, drying oven, drying tins or crucibles.

LIMITATIONS: Gypsum can be dehydrated at temperatures as low as 105°C. Dehydration of gypsum and other hydrates has been observed as a result of heating during sample grinding as well.

pH of soils is found to drop with air drying, particularly in soils high in sulfides.

If plant samples are not dried at high enough temperatures, further chemical and biological changes can continue to occur during storage. If plant material is dried at too high a temperature, samples can undergo thermal decomposition that can affect the final dry weight.

BIBLIOGRAPHY:
Huat Lim, C., and Jackson, M.L., 1982, Dissolution for Total Elemental Analysis *in* Methods of Soil Analysis, Part 2 – Chemical and Microbiological Properties, 2nd Edition. Page, A.L., Miller, R.H., Keeney, D.R. (eds.), American Society of Agronomy and Soil Science Society of America, pp. 1-10.

James, D.W., and Wells, K.L., 1990, Soil Sample Collection and Handling: Technique Based on Source and Degree of Field Variability, *in* Soil Testing and Plant Analysis, 3rd Edition, Westerman, R.L., ed., Soil Science Society of America Book Series, No 3. pp. 389-427.

Jones, B.J., and Case, V.W., 1990, Sampling, Handling, and Analyzing Plant Tissues Samples *in* Soil Testing and Plant Analysis, 3rd Edition, Westerman, R.L., ed., Soil Science Society of America Book Series, No 3. pp. 389-427.

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Liu, C., and Evett, J.B., 1984. Soil Properties: Testing, Measurement, and Evaluation, Prentice-Hall, Englewood Cliffs, New Jersey, 303 p.
Nelson, R.E., Klameth, L.C., Nettleton, W.D., 1978, Determining soil gypsum content and expressing properties of gypsiferous soils: Soil Science Soc. Am. Journal, v. 42: p 659-661.
Shugar, G.J., Shugar, R.A., Bauman, L., 1973. Chemical Technicians' Ready Reference Handbook. McGraw-Hill Book Company, New York, 463 p.
Sobek, A.A., Schuller, W.A., Freeman, J.R., Smith, R.M., 1978. Field and Laboratory Methods Applicable to Overburdens and Minesoils, EPA-600/2-78-054, Environmental Protection Technology Series, Industrial Environmental Research Laboratory, Office of Research and Development, USEPA.

Mineral Separation Techniques (Heavy Liquids, Gravity, Magnetic)

(Virginia T. McLemore)

MEDIA: solid

METHOD: Mineral separation techniques

PRINCIPLE: Mineral separation techniques generally involve the density separation of minerals of different specific gravity using a medium, such as water or heavy liquids, to separate the different minerals. The heavier minerals settle first from a mixture suspended in water or other liquid.

PRIMARY USES: To separate minerals of different densities or magnetic properties for more detailed analyses, such as mineral abundance, chemistry, and textures and dating of minerals.

LIMITATIONS: Most heavy liquids are toxic and special care need to be employed when using them. Sodium metatungstate is an ideal liquid used for mineral separation because it is not toxic and can be prepared at different densities (Krukowski, 1988).

INTERFERENCES: Some media could introduce contaminants to the separated minerals or oxidize some minerals.

SCOPE: The most common method is hand or gold panning to obtain a heavy mineral concentrate using a pan and water and was used by prospectors for decades to locate placer gold deposits (Theobald, 1957). Generally the sample is crushed and sieved. The most common medium is water, although other liquids can be used (i.e. methylene iodide, density of 3.325; sodium metatungstate, density of 1-3.1). Large volumes of samples can be processed using a Wilfley table (a specialized table that shakes and uses water). Smaller samples can be processed using a hand pan (i.e. gold pan). Magnetic minerals can be separated using a magnet or a Frantz isodynamic magnetic separator or other type of separator. Acids, such as hydrofluoric acid, can be used to dissolve the major minerals and leave the more acid-resistant minerals such as graphite, rutile, topaz, zircon, and others (Neuerburg, 1961). A centrifuge also can be used to separate minerals (Rittenhouse and Bertholf, 1942).

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BIBLIOGRAPHY:
Krukowski, S.T., 1988, Sodium metatungstate: a new heavy-mineral separation medium for the extraction of conodonts from insoluble residues: Journal of the Paleontological Society, v. 62, p. 314-316.
Neuerburg, G.J., 1961, A method of mineral separation using hydrofluoric acid: American Mineralogist, v. 46, p. 1498-1501, http://www.minsocam.org/ammin/AM46/AM46_1498.pdf, accessed 8/23/13.
Rittenhouse, G. and Bertholf, W.E., 1942, Gravity versus centrifuge separation of heavy minerals from sand: Journal of Sedimentary Research, v. 12, p. 85 - 89.
Schoen, R. and Lee, D.E., 1964, Successful separation of silt-size minerals in heavy liquids: U.S. Geological Survey, Professional Paper, B154-B157.
Theobald, P.K., Jr., The gold pan as a quantitative geologic tool: U.S. Geological Survey, Bulletin 1071-A, 54 p.

Geotechnical Sampling

(Virginia T. McLemore)

MEDIA: Broken rock, soil, tailings

SAMPLING OBJECTIVES: Determine the geotechnical characteristics of mine wastes or in-situ soils that might be used to construct waste management or process facilities such as compacted fills, embankments, heap leach pads, dams and rock dumps. If geotechnical samples will be used for geochemical analyses, the sampling considerations that follow may be inadequate.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUES: Because the geotechnical properties of mine wastes and soils vary significantly over time only to a limited extent, sampling is usually grouped around a single investigation or program of investigations that can be summarized once and used over an extended period. An exception to this is a change in water content of the soil or waste material. See the section titled spatial or temporal considerations below. Each investigation usually includes periods of planning, field investigation and sample collection, laboratory analysis, and reporting.

Sampling equipment selection will be a function of the money and the time available for investigation. It is also a function of the grain size, moisture content, location, depth of the waste sampled. Samples from the surface to a foot and a half deep can be collected with hand tools such as a hand auger. Samples deeper than this generally require a back hoe, track hoe or drill rig. Track hoes or back hoes can commonly sample to a depth of about 15 feet. Beyond this depth a drill rig is required. The type of drill rigs used for geotechnical investigations can vary from a small truck mounted rigs meant for 6 inch diameter borings up to 50 feet deep to track mounted rigs that can use 8 foot diameter augers or rock coring equipment that can go 500 to 1000 feet deep.

Fine grained wastes such as tailings can be sampled from a drill rig with a thin wall (Shelby) tube or split spoon sampler in conjunction with a hollow stem auger provided that a stable drilling platform can be established on the surface of the mine waste. Thin wall tube samples are relatively undisturbed; other samples are typically

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considered disturbed. Test pits dug with trackhoe or backhoe excavators will yield bulk samples of fine or coarse grained materials such as waste rock. Moderately saturated or liquefiable fine grained materials such as tailing offer significant sampling challenges that cannot be addressed in the context of a simple guideline. Some sampling procedures are outlined by the American Society for Testing Materials (ASTM), which provides updated manuals that specify sampling protocols.

SPATIAL OR TEMPORAL CONSIDERATIONS: Mine wastes can be heterogeneous with regard to geotechnical characteristics due to material segregation effects controlled by differences in grain size, shape and specific gravity. For example, a tailing sample collected close to the tailings discharge pipe will likely have a different (coarser) grain size distribution than a sample collected further away. Also, coarser materials typically tend to collect at the toes of waste rock dumps. Samples can vary significantly over time periods of greater than a few days. Settlement and consolidation of tailing under a load and drying of heap leach material during drain down are examples of this. Generally, geotechnical samples, once they are isolated from loads and potential changes in moisture content, will not have their properties change significantly.

ANALYTICAL TECHNIQUES: Numerous laboratory testing procedures have been developed to determine the geotechnical properties of soils. At a minimum, samples are tested to verify a classification performed in the field. Samples may be tested for grain size distribution, moisture content, specific gravity, compacted (Proctor) density, saturated hydraulic conductivity, direct shear strength, triaxial shear strength, consolidation, Atterberg (moisture) Limits and other tests that may be required to characterize a sample or material. Many analytical procedures are outlined by ASTM, which provides updated manuals that specify testing protocols.

CONTAINERS: Thin-wall sampling tubes, resealable plastic bags, plastic jars w/lids, 5-gallon buckets w/lids, 55-gallon drums.

CONTAINER PRECLEANING: Containers should be dry, free of grease or visible contaminants; no special precleaning is required.

PRESERVATION: Sample moisture preservation is paramount. The ends of thin wall sample tubes may be sealed with hot paraffin or plastic caps that are secured with heavy-duty tape. Double- or triple-bagging samples is recommended to prevent moisture loss or gain. Minimizing the air space around the sample is important but is not crucial to sample preservation.

HANDLING AND PREPARATION: Samples should be kept from freezing; sample temperature should be controlled in special cases to preserve in-situ conditions that may be important to subsequent testing (e.g., permafrost).

ADDITIONAL CONSIDERATIONS: Geotechnical sampling should be conducted by an individual trained as a civil or geotechnical engineer or a trained engineering

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technician. Sampling plans should be developed and supervised by a registered professional engineer.

BIBLIOGRAPHY:

The classic text for geotechnical sampling is Hvorslev's volume from 1949, reprinted in 1962 and 1965:

Hvorslev, M. Juul; Subsurface Exploration and Sampling of Soils for Civil Engineering Purposes; 1965; Engineering Foundation.

The Army Corps of Engineers has a volume that covers most aspects of geotechnical sampling, including a chapter on underwater sampling of soils.

U.S. Army Corps of Engineers; Soil Sampling; Document No EM 1110-1-1906; 30 September, 1996.

Another classic publication is the Earth Manual, now in its third edition, by the Bureau of Reclamation:

U.S. Bureau of Reclamation; Earth Manual, A guide to the use of soils as foundations and as construction materials for hydraulic structures; 1974.

If a more brief treatment is required, Chapter 3 in Bowles is a good resource:

Bowles, Joseph E., P.E., S.E.; Foundation Analysis and Design; Fourth Edition; 1988; McGraw-Hill, Inc.

ASTM provides individual procedures for a number of the techniques developed in the above volumes. Most of them are contained in two volumes from ASTM:

American Society for Testing and Materials; Soil and Rock (I), D420 to D5779; April 2000; 1622 p.

American Society for Testing and Materials; Soil and Rock (II), D5780 to Latest; Geosynthetics; May 2000; 804 p.

Paste pH

(Virginia T. McLemore)

MEDIA: Soil or fine-grained solid material (also known as soil pH)

MEASUREMENT OBJECTIVES: To estimate the pH and conductivity of the porewater resulting from dissolution of secondary mineral phases on the surfaces of oxidized rock particles. The paste pH of a solid is the pH of a slurry or paste of a solid or other fine-grained solid material. The paste pH is the negative logarithm of the hydrogen ion (H^+) activity and is expressed as:

$$pH = -\log [H+]$$

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where values in brackets express activity, not concentration. The pH is expressed using logarithmic units, normally with a scale from 0 to 14, but rare natural waters may have pH values outside of this range. Neutral pH (pH=7.00) is the point where $[H^+]=[OH^-]$ and should not be assumed to equal the background pH of natural waters. The general terms acidic and basic (or alkaline) waters refer to pH below (acidic) or above (basic) neutral pH. However, pure water, such as rainwater, in equilibrium with atmospheric CO_2 has a pH of 5.66 at 25ºC. Therefore, common usage of acidic waters, refer to waters with a pH below 5. Most natural waters have a pH between 5 and 9; acid rain may have pH lower than 5, and some wetlands in arid climates may have pH greater than 9. Typically analysis of pH is required in the field. This is because a variety of physical, chemical, and biological processes can occur to significantly change pH values soon after sample collection. Measurements of pH are ideally made in a flow-through cell.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): The measurement of paste pH is normally accomplished with a digital pH meter, a plastic or glass liquid-filled or gel-filled combination Ag/Cl electrode, and a temperature compensation device. A wide variety of pH meters and electrodes are available. Operators should be thoroughly familiar with the recommendations for calibration and operation provided by the manufacturer. The standard method for pH measurements is to record values with time until a stable pH value is obtained.

ANALYTICAL TECHNIQUE(S): Electrodes need to be clean and in good order to produce reliable results. Electrodes should generally be checked in the laboratory before field activities begin. The liquid junction should be clear and the electrolyte solution in the electrode must be at the recommended level. Calibration of meters and electrodes is normally accomplished with pH 4, 7, and 10 buffers that are available from standard suppliers. Calibration routines may utilize two-point or three-point checks. It is also possible to obtain other pH buffers at lower or higher pH values. Choices of calibration buffers will ideally bracket expected pH values in water samples. Manufacturer's recommendations should be closely followed for procedures of calibration and sample measurement. Document all measurements in an instrument notebook. Because pH measurements are affected by temperature, sample temperature should always be recorded during pH measurement.

To obtain the slurry or paste, obtain approximately 25 grams of fines (particles smaller than 1 mm if possible) from the soil or rock sample to be tested, and place in a fresh or decontaminated beaker. Reseal the bag from which the paste test sample was obtained. Add approximately 25 mL of distilled water to sample. (More water may be required if the sample is very dry or extremely fine). Stir sample with fresh or cleaned spatula to form a paste or slurry. Paste should slide off spatula easily. Let stand for 10 minutes. Tip the testing container to one side to allow a pool of water or slurry to collect in the corner. Dip each of the probes into the slurry, and allow the meter reading to stabilize. The conductivity reading should however be done first, as electrolyte from the combination pH probe may affect the conductivity of the solution. Decontaminate probes and containers by rinsing with distilled water.

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LIMITATIONS/INTERFERENCES: Readings taken on uncrushed samples in the field or lab usually provide a much better indication of the extent of oxidation than crushed samples do. It is strongly recommended to use a more robust field pH electrode for this measurement.

DOCUMENTATION/DATA HANDLING: All calibration notes and sample measurements should be recorded in a dedicated notebook.

BIBLIOGRAPHY:

British Columbia AMDT Task Force (1989), Draft Acid Rock Drainage Technical Guide, Vol I, Crown Publication, Victoria, B.C.

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, 203 p.

Atterberg Limits

(S. Nunoo and Virginia T. McLemore)

METHOD: Describes determination of Atterberg Limits test. Atterberg Limits tests consist of two tests called Plastic Limit, PL, and Liquid Limit, LL.

PRINCIPLE: Atterberg Limits are a measure of the nature of fine-grained soil and is an approximate measure of the clay material in the soil. The soil can appear as a solid, semi-solid, plastic and liquid state depending on its water content. In current engineering usage, Atterberg Limits refer only to liquid limit and plastic limit, rarely including the shrinkage limit. The liquid limit is the percent water content of the soil at the arbitrarily defined boundary between the liquid and plastic states. The plastic limit is the percent water content of the soil at the boundary between plastic and brittle states.

PRIMARY USES: This test is used to determine the consistency (relative ease with which a soil can be deformed) of clay, and characterize the fine-grained fraction of soils. The liquid limit (LL), plastic limit (PL), and plasticity index (PI) of soils also are used with other soil parameters to correlate with engineering behavior such as compressibility, permeability, compactibility, shrink-swell, and shear strength.

LIMITATIONS: must be a soil material, not suitable for rocks

INTERFERENCES: none

SCOPE: Obtain a 150 to 200 gram representative portion of material that passed the number 40 sieve. Put the material in a ceramic bowl and add distilled water to achieve a putty-like uniform consistency that can achieve 15-35 blows of the liquid limit device to close the groove. Put in a beaker and cover with Para film for 16 hours. Remix sample thoroughly before starting test.

***Liquid Limit Test*—**Adjust the cup on the liquid limit device so it drops 10 mm from the base (plus or minus 0.2 mm). Use the end of the grooving tool handle to measure this since it is 10 mm. Take approximately 20 grams of material from the beaker and place

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on the glass plate. Set this aside for the plastic limit test. Take material and pack it into the cup of the liquid limit device, smoothing with the spatula. The material should fill the cup to the same level that water would stand in the cup without overflowing. Use the grooving tool to cut a groove in the center of the material starting from the back and scraping forward. Clean crumbs from under and around the cup. Start to turn the crank on the device to raise and drop the cup. Crank to approximately 2 drops per second until the two sides of the material in the cup touch for a distance of 13 mm (or ½ inch). Record the number of blows it took for the material to touch. Remove the area of the material that touched using a spatula, and record weight. Put in oven overnight and record weight again to obtain the corresponding water content. Clean cup completely between tests, returning the material to the beaker each time and remixing. Repeat test until each blow count range is achieved. One trial shall require 25 to 30 blows, another requires 20 to 30 blows, and another requires 15-25 blows. Additional distilled water may be added to obtain each range.

Plastic Limit Test—Take the 20 grams of material that was set aside and roll it on the glass plate to reduce the water content. Make a thread that is a uniform thickness of 3.2 mm (1/8 inch) and a length of 10 cm (4 inches) within two minutes. Keep taking material away to get these dimensions. Once the thread has been made to the proper dimensions in two minutes, roll the material into a ball, and start to make a new thread. Repeat this process until a thread of the desired dimensions cannot be made or a ball cannot be made in hand. Once this happens, weigh the sample and record. Put the sample in an oven over night and record the weight next day. Do this test at least twice to get an average plastic limit for the sample.

DETECTION LIMITS: If either the liquid limit or plastic limit cannot be determined, the plasticity index cannot be computed and should be reported as "NP" (indicating sample is nonplastic). The plasticity index should also be reported as "NP" if the plastic limit turns out to be greater than or equal to the liquid limit. For very sandy soil, the plastic limit test should be performed before the liquid limit test; if it cannot be determined, both the liquid and the plastic limit should be reported as "NP."

DATA FORMAT:

Liquid Limit, LL—Plot the relationship between the water content and the corresponding number of drops of the cup on a semi-logarithmic graph with the water content ordinates on the arithmetical scale, and the number of drops as abscissas on the logarithmic scale. Draw the best straight line through the three or more plotted points. Obtain the water content corresponding to the intersection of the line with 25 drops on the abscissa as the liquid limit, LL, of the soil.

Plastic Limit, PL—Compute the average of the water contents obtained from the two plastic limit tests. The plastic limit, PL, is the average of the two water contents.

Plasticity Index, PI—The plasticity index is determined by subtracting the plastic limit from the liquid limit. In equation form, PI = LL – PL.

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TABLE A3.10. Atterberg Limits Test Sheet.

Project:

Analyzed By: _____ Initials: _____ Date: _____

Sample Field ID: _____

	Liquid Limits			Plastic Limits	
	1	2	3	1	2
Tare No.					
Tare Mass (g)					
Tare + Wet Soil (g)					
Tare + Dry Soil (g)					
Mass Dry Soil (g)					
Water Content (%)					
Number of Blows					

Liquid Limit (LL)	
Plastic Limit (PL)	
Plasticity Index (PI)	
Symbol from Plasticity Chart	

BIBLIOGRAPHY:

ASTM D4318-84: Standard Test Method for Liquid Limit, Plastic Limit, and Plasticity Index of Soils.

Bowles, Joseph E., 1992, Engineering properties of soils and their measurement, 4th edition, New York, NY.

Liu, C. and Evett, Jack B., 2003, Soil Properties: testing, measurements, and evaluation, 5th edition, Upper Saddle River, NJ.

Clay Mineralogy

(Virginia T. McLemore)

MEDIUM: solid

SAMPLING OBJECTIVES: The objectives are to determine the type and measure the amount of clay minerals. The texture of clay minerals is characterized.

THEORY: Clay minerals are hydrous-aluminum silicates and are classified as phyllosilicates, or layered silicates. Clay minerals are too small in size to identify using normal petrographic techniques and the clay minerals are so similar in composition and

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structure, that it also is difficult to determine individual minerals. Yet clay minerals can play important roles in AMD, especially in mine wastes (Donahue et al., 2007, 2009). Most clay minerals have in common a plate-like morphology and perfect (001) cleavage (Moore and Reynolds, 1997). The (00l) cleavage is one of the (hkl) or Miller indices that defines the orientation of an internal crystal plane (Fig. A3.7). In this case, the (001) represents the plane perpendicular to the c-axis in which clay-mineral layers often stack (Fig. A3.7). There are four clay mineral groups based upon their layered morphology: kaolin, illite, smectite, mixed layered clays, and chlorite. For a further explanation of this system, see Moore and Reynolds (1997) or any introductory mineralogy text.

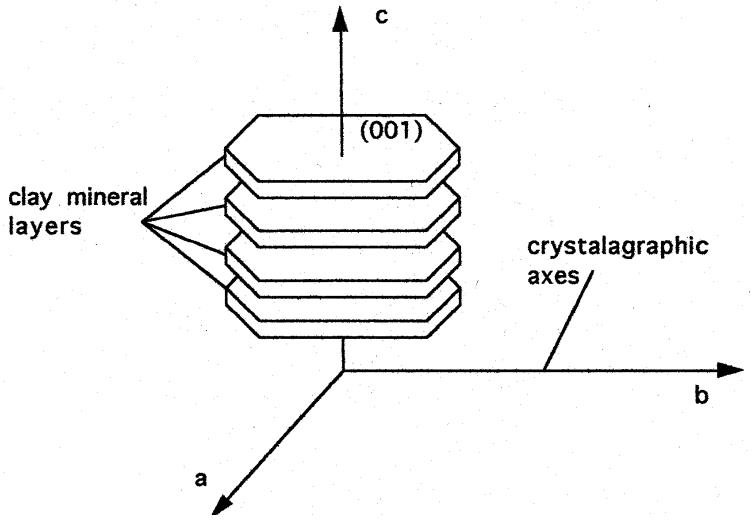


FIGURE A3.7. Clay mineral layers on crystallographic axes.

SPATIAL OR TEMPORAL CONSIDERATIONS: Record the locations and note if the sample was a subset of a balk sample.

ANALYTICAL TECHNIQUES: X-ray diffraction (XRD), electron microprobe

CONTAINER: Plastic or paper can be used to store the sample.

PRESERVATION: Keep dry.

HANDLING AND PREPARATION: None needed.

METHOD OF SAMPLE PREPARATION: Samples must be crushed (not pulverized), split, and dried for preparing oriented clay slides for XRD analysis. Soil samples generally do not require crushing. See section on electron microprobe for procedures.

PROCEDURE: Procedure for preparing oriented glass slides for XRD analysis is by Hall (2004) and Moore and Reynolds 1997).

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LIMITATIONS: The sample fraction used in the analysis must be representative of the entire sample. Additionally, the sample could be subjected to a gravimetric-analysis procedure in the cases of particle-size analysis and leaching. Check for flocculation, which can destroy the preferred orientation of the clay particles. Samples with high sulfur could be problematic to prepare (Hall, 2004).

ADDITIONAL CONSIDERATIONS: See Hall (2004) and Moore and Reynolds (1997) for other problems in preparing oriented glass slides and alternative methods for clay mineral analysis.

BIBLIOGRAPHY:

Donahue, K., Dunbar, D., and McLemore. V., 2007. Origins of clay minerals in the Molycorp Goathill North rock pile, Questa, NM, SME preprint 07-100, 8 p.

Donahue, K.M., Dunbar, N.W., and McLemore, V.T., 2009, Clay mineralogy of the Goathill North rock pile, Questa mine, Taos County, NM: Origins and indications of in-situ weathering: Society of Mining, Metallurgy and Exploration Preprint 09-98. 7 p.

Hall, J.S., 2004, New Mexico Bureau of Mines and Mineral Resource's Clay Laboratory Manual: Unpublished New Mexico Bureau of Geology and Mineral Resources report.

Moore, O.M. and Reynolds, R.O., Jr., 1997, X-Ray Diffraction and the Identification and Analysis of Clay Minerals, Oxford University Press, New York, 378 p.

Gravimetric Moisture Content

(Virginia T. McLemore)

SAMPLING PURPOSE: To estimate the moisture content of the material at a specific time (Gardner, 1986; Jury et al., 1991; Hillel, 1998). Moisture content is the ratio of the mass of the water contained in a soil sample to the mass of the solid fraction of the soil sample. Observations of moisture content values are critical to modeling the seepage and stability of the rock piles (Fredlund, D. G., 2002; Fredlund, D. G. et al., 1994; Fredlund, M. D. et al., 1996, 1997).

SPECIFIC SAMPLING TECHNIQUES: Samples are collected from split spoon samples from drill holes, test pits, and trenches immediately upon exposure to the atmosphere to avoid drying. Exposure of samples to ambient surface conditions can cause significant changes in moisture content through evaporation, precipitation, spills, etc. if the exposure is longer than a few minutes. The rate of change in moisture content is a function of the differences in temperature and relative humidity between the sample's original environment and the surface environment. Two samples are collected from each split spoon sample, one at the top and one at the bottom of the split spoon sample. Collect sample and place into sealed moisture content jars.

The sample mass collected should be proportional to the range of grain and clast sizes contained in the material of interest. Larger sample masses are required for representative samples of stony or rocky soils compared to sandy or silty sediments or soils. However, collecting smaller masses of finer-grained material from stony or rocky soils does not necessarily cause a bias in a particular study because variably saturated

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flow will generally be confined to the finer-grained fraction, not the cobbles or larger clast sizes.

SPATIAL OR TEMPORAL CONSIDERATIONS: Measurement precision should be selected as a function of the sample mass to be measured. For sample masses less than 500 g, the weight precision should be within 0.01 g. Precisions for larger sample masses should be scaled up similarly: e.g., sample masses between 0.5 and 2 kg should have measurement precision on the order of 0.1 g.

ANALYTICAL TECHNIQUES: Remove the lid and oven dry jar + lid at 105 degrees C for 24 hr, cool, then weigh dry jar + lid as above to within the appropriate precision. Remove sample from jar and archive in plastic bag. Weigh empty jar + lid before cleaning. Note mass of sample on the plastic bag (sample dry weight – weight of clean empty jar + lid). Gravimetric water content (g/g) = (sample wet weight – sample dry weight) / sample dry weight.

CONTAINER/ PRESERVATION AND HANDLING AND PREPARATION: Keep the collected sample sealed and cool so that the sample does not prematurely dry out.

ADDITIONAL CONSIDERATIONS: None.

BIBLIOGRAPHY:

Fredlund, D. G. 2002. Use of soil-water characteristic curves in the implementation of unsaturated soil mechanics: Keynote address presented at the 3rd International Conference on unsaturated soils, Recife, Brazil, March 10-13.

Fredlund, D. G., Xing, A., and Huang, S., 1994, Predicting the permeability function for unsaturated soils using the soil-water characteristic curve: Canadian Geotechnical Journal, v. 31, p. 533-546.

Fredlund, M. D., Sillers, W. S., Fredlund, D. G., and Wilson, G. W., 1996, Design of a knowledge based system for unsaturated soil properties: 3rd Canadian Conference on Computing in Civil and Building Engineering, 19 p.

Fredlund, M. D., Wilson, G. W., and Fredlund, D. G., 1997, Indirect procedures to determine unsaturated soil property functions: Proceedings of the 50th Canadian Geotechnical Conference, Ottawa, Ontario, 9 p.

Gardner, W.H., 1986, Water Content, in *Methods of Soil Analysis Part 1. Physical and Mineralogical Methods*, Agronomy Monograph no. 9, Soil Science Society of America, Madison, WI, USA, p. 493-544.

Hillel, D., 1998, *Environmental Soil Physics*, Academic Press, New York, p.129-142.

Jury, W.A., W.R. Gardner, and W.H. Gardner, 1991, *Soil Physics*, John Wiley and Sons, New York, p.45-47.

Porosity

(S. Nunoo)

METHOD: To measure the amount of water stored in the pore space of a soil sample.

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PRIMARY USES: Soils are made of particles of different types and sizes. The space between particles is called pore space. Pore space determines the amount of water that a given volume of soil can hold. Porosity is the percentage of the total volume of soil that consists of pore space. This is an important measurement in areas where drinking water is provided by groundwater reserves.

LIMITATIONS: must be soil and rock material

INTERFERENCES: none

PROCEDURE: Pour 100mL of water into your cup and draw a line where the water comes up to. Write 100mL in the total volume column on your data sheet. Dump out the water. Fill the cup with the first soil sample up to the line you drew. Using your graduated cylinder, slowly and carefully pour water into the cup until the water reaches the top of your sample. Write the volume of water remaining in the graduated cylinder on your data sheet. Subtract the volume remaining from the total volume. This is the amount of water you added to your sample. Write the volume of water added to the sample on your data sheet – this is the pore space. To determine the porosity of the sample, divide the pore space volume by the total volume and multiply the result by 100. Write the porosity on your data sheet. (Note: % pore space = pore space / total volume x 100).

TABLE A3.11. Porosity Data Sheet

Sample Number	Description of Sample	Total Volume (mL)	Volume remaining in cylinder (mL)	Pore Space	Porosity (%pore space)	Time of water to pass through (sec)
Example	Sandy gravel	100	80	100-80=20	(20/100)x100= 20	10

BIBLIOGRAPHY:

ASTM F1815 - 06 Standard Test Methods for Saturated Hydraulic Conductivity, Water Retention, Porosity, and Bulk Density of Putting Green and Sports Turf Root Zones

Particle Size Analysis

(S. Nunoo and Virginia T. McLemore)

METHOD: This test is performed to determine the percentage of different grain sizes contained within a soil. The mechanical or sieve analysis is performed to determine the distribution of the coarser, larger-sized particles, and the hydrometer method is used to determine the distribution of the finer particles.

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PRINCIPLE: Two general methods are considered for particle size analysis for air-dried samples. One condition is that the fine particles in the sample do not clog the sieve openings. In this case, the distribution of particle sizes larger than 75 μm (retained on the No. 200 sieve) is determined by sieving, whereas the distribution of particle sizes smaller than 75 μm is determined by a sedimentation process using a hydrometer to secure the necessary data. Another condition is that the fine particles clog the sieve openings.

PRIMARY USES: The distribution of different grain sizes affects the engineering properties of soil. Grain size analysis provides the grain size distribution, and it is required in classifying the soil.

LIMITATIONS: must be a soil material, not suitable for rocks

INTERFERENCES: none

SCOPE: Two general methods are considered for particle size analysis for air-dried samples. One condition is that the fine particles in the sample do not clog the sieve openings. In this case, the distribution of particle sizes larger than 75 μm (retained on the No. 200 sieve) is determined by sieving, whereas the distribution of particle sizes smaller than 75 μm is determined by a sedimentation process using a hydrometer to secure the necessary data. Another condition is that the fine particles clog the sieve openings. In this case, the sample is divided into two portions. One portion contains only particles retained on the No. 10 sieve that its distribution is determined by sieving, whereas the other portion contains only particles passing the No. 10 sieve that its distribution is determined by sedimentation process (Hydrometer) and sieving. For this purpose, sedimentation process is performed for 100 g particles passing No. 10 in order to obtain the distribution of fine particles (finer than No. 200 sieve), and then, after taking final hydrometer reading, the suspension is transferred to a No. 200 sieve and washed with tap water until the wash water is clear. Then the material retained on the No. 200 sieve is transferred to a container and dried in an oven. Finally, in order to obtain the distribution of particles passing No. 10 sieve and retained on No. 200 sieve, sieve analysis of the oven-dried material remained on No. 200 sieve is made.

Mechanical Particle Size Analyses

The air-dried sample is split using the sample splitter to obtain the representative sample for particle size analysis. The size of the sample shall depend on the diameter of the largest particles in the sample. The total sample weight is recorded, and the top sieve is selected based on the weight of the sample as the one with openings that are slightly larger than the diameter of the largest particle in the sample. The series of sieves are arranged so that they have decreasing opening size from the top to the bottom of the stack. The sieve stack is placed in the shaking machine for at least 10 minutes or until additional shaking does not produce appreciable changes in the amounts of material on each sieve screen. Finally, the sieve stack is removed from the machine, and the weight of retained soil particles on each sieve is recorded on the data sheet.

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Hydrometer Analyses

The sample is placed in a 250 ml beaker, and distilled or demineralized water is added until the sample is submerged, then 15 ml of the dispersing agent (deflocculant) is added at this time. The sample is allowed to soak overnight or until all soil lumps have disintegrated. At the end of the soaking period, the sample is dispersed further by transferring the complete sample to the dispersion cup. Any residue from the beaker is washed with distilled or demineralized water. Distilled water is added if the cup is less than half full. The cup is placed in dispersing machine to disperse the suspension for 1 to 10 minutes. The solution is transferred to the sedimentation cylinder and distilled water is added to the 1000 ml mark. The suspension is shaken vigorously for a few seconds. At the end of the 1 minute shaking period, the cylinder is set on a stable surface and the time is noted. The hydrometer is immersed slowly into the liquid for each reading. Hydrometer readings are taken after 1 and 2 minutes have elapsed from the time the cylinder was placed on the table. As soon as each reading is taken, the hydrometer is removed from the suspension carefully. Since during the first two readings, errors are likely to happen, these first reading steps are repeated to make sure that the same values are obtained. Then the sample is shaken again to proceed to the next steps. At the end of the second 2 minutes and after each subsequent hydrometer reading, a thermometer is placed in the suspension and the temperature is recorded. After reading at 1 minute and 2 minutes, hydrometer readings are recorded at the following time intervals: 4, 15, 30, 60, 120, 240, 960, 1440 minutes.

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TABLE A3.12. Grain Size Analysis Sheet.

Sieve Analysis Data Sheet

Sieving performed by (initials): _____ Date: _____

Project: Molycorp Sample ID: _____

Note: Dry sieve following ASTM-Separation made on No. 10 sieve.

Mass of total air-dried sample (g): _____

Portion coarser than No. 10 (retained on No. 10).

Sieve Opening		U.S. Standard		
Inches	Millimeters	Sieve No.	Bag Label	Weight Retained
3.00	75	3 in.	xxx-xxx-0000-53	
2.00	50	2 in.	xxx-xxx-0000-54	
1.50	37.5	1 ½ in.	xxx-xxx-0000-55	
1.00	25	1 in.	xxx-xxx-0000-56	
.75	19	¾ in.	xxx-xxx-0000-57	
.375	9.5	3/8 in.	xxx-xxx-0000-58	
.187	4.75	4	xxx-xxx-0000-61	
.132	2	10	xxx-xxx-0000-82	
Pan			xxx-xxx-0000 Passing No. 10	

Calculation for the portion finer than a No. 10 sieve, after hydrometer test.

Hydrometer performed by (initials): _____ Date: _____
Amount and type of dispersing agent used: 125ml of sodium hexametaphosphate solution (160g/l).

Calculation for the Hygroscopic Moisture Correction Factor

Mass of container + air-dried soil (g): _____

Mass of container + oven-dried soil (g): _____

Mass of container: _____

Mass of air-dried soil sample for hydrometer analysis (g): _____

Sieve Opening		U.S. Standard	
Inches	Millimeters	Sieve No.	Weight Retained
.047	1.18	16	
.023	.6	30	
.0165	.425	40	
.0177	.3	50	
.0083	.212	70	
.0059	.15	100	
.0029	.075	200	

	Time	Hydrometer Reading
Trial #1	1 minute	
	2 minutes	
Trial #2	1 minute	
	2 minutes	
Trial #3	1 minute	
	2 minutes	

Hydrometer Type: **152H** Hydrometer Number: A C D E

Date	Time	Elapsed Time (hour)	Elapsed Time, T (minutes)	Temperature (°C)	Actual Hydrometer Reading
			1		
			2		
			4		
			8		
			15		
			30		
		1	60		
		2	120		
		4	240		
		16	960		
		24	1440		

COST: $65 - $70 in terms of unit cost for both Particle Size and Hydrometer Test.

BIBLIOGRAPHY:

ASTM (1988b) D422-63: Standard Method for Particle-Size Analysis of Soils.

SOLIDS-SPECIATION TECHNIQUES

AVS/SEM solid phase analysis

(Richard T. Wilkin)

MEDIA: Sediment and Soil

MEASUREMENT OBJECTIVES: The acid-volatile sulfide/simultaneously extracted metals (AVS/SEM) method is an approach to assess the bioavailability and toxicity of metals in sediments. Acid-volatile sulfides (AVS) are a group of metal sulfides that dissolve in dilute hydrochloric acid. These minerals are thought to sequester toxic metals

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within anoxic sediments and thereby prevent them from entering the aqueous, bioavailable phase. The metal concentration that is extracted from a sediment sample as it reacts with hydrochloric acid is referred to as simultaneously extracted metals (SEM). No acute toxicity is indicated if the molar concentration of AVS is greater than the molar concentration of SEM. The approach assumes that the bioavailable fraction of total metals in sediments is the difference between the SEM and AVS.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): Determination of AVS and SEM in sediment is conducted by treatment of the sample with dilute hydrochloric acid, typically 1 M HCl, for one hour in an oxygen-free environment. Hydrogen sulfide that is liberated during this procedure is purged with an inert gas (nitrogen), trapped in NaOH or as a zinc sulfide precipitate, and measured. The concentration of sulfide is referenced back to the mass of sediment extracted to determine the concentration of AVS on a dry weight basis. To determine SEM, the sediment plus acid mixture is filtered and the liquid extract is analyzed for selected metals. Extraction is typically conducted in a flat-bottom reaction flask. Sediment samples to be analyzed for AVS/SEM must be carefully collected and preserved to prevent the oxidative loss of metal sulfides. Sediment samples should be frozen after collection until analysis.

ANALYTICAL TECHNIQUE(S): Sulfide measurements may be made using colorimetric methods (methylene blue), coulometric methods, gravimetric methods, or by using a sulfide-specific electrode. Measurements of selected metals may be made using AA, ICP-AES, or ICP-MS.

LIMITATIONS/INTERFERENCES: Target metals typically include Cd, Ni, Pb, Zn, and Ag. There may be complicating factors for interpreting SEM data for metals that form highly insoluble sulfides such as Hg and Cu (Mikac et al., 2000; Cooper and Morse, 1998). The standard extractant solution (1N HCl) may not be strong enough to dissolve these metal sulfides. The method is not appropriate for metalloids such as As (Wilkin and Ford, 2002).

BIBLIOGRAPHY:

Boothman, W.S., Hansen, D.J., Berry, W.J., Robson, D.L., Helmstetter, A., Corbin, J., and Pratt, S. 2001. Biological response to variation of acid volatile sulfides and metals in field exposed spiked sediments. *Environ. Tox. Chem.* 20:264–272.

Cooper, D. C. and Morse, J. 1998. Extractability of metal sulfide minerals in acid solutions: Application to environmental studies of trace metal contamination within anoxic sediments. *Environ. Sci. Technol.* 32:1076–1078.

Di Toro, D.M., Mahony, J.D., Hansen, D.J., Scott, K.J., Hicks, M.B., Mayer, S.M., and Redmond, M.S. 1990. Toxicity of cadmium in sediments: The role of acid volatile sulfide. *Environ. Tox. Chem*. 9:1487–1502.

Mikac, N., Niessen, S., Ouddane, B., and Fischer, J. 2000. Effects of acid volatile sulfides on the use of hydrochloric acid for determining solid phase associations of mercury in sediments. *Environ. Sci. Technol.* 34:1871–1876.

Wilkin, R. T. and Ford, R. G. 2002. Use of hydrochloric acid for determining solid-phase arsenic partitioning in sulfidic sediments. *Environ. Sci. Technol.* 36:4921–4927.

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Equilibrium Partitioning Sediment Benchmark (ESB)

(Kathleen S. Smith)

MEDIUM: Sediment and interstitial-water partitioning

PRINCIPLE: Equilibrium partitioning sediment benchmarks (ESBs) for metals are sediment-quality guidelines (SQGs) developed by the U.S. Environmental Protection Agency to relate chemical concentrations in sediment to potential biological effects to benthic organisms (USEPA, 2005). A benchmark is a chemical concentration above which there is the possibility of harm or risk. Benchmarks are for screening purposes only and are not regulatory or clean-up standards. Metal concentrations commonly are elevated in aquatic sediments, yet total metal concentrations in the sediment generally do not correlate well with biological effects (e.g., Salomons et al., 1987). ESBs are mechanistic SQGs based on equilibrium partitioning theory (EqP) (Di Toro et al., 1991), which predicts metal concentrations that are potentially bioavailable to benthic organisms from sediment interstitial water. For example, in anoxic sediments sulfide can sequester many cationic metals (e.g., cadmium, copper, lead, nickel, silver, and zinc). Resulting metal sulfides are relatively insoluble and tend to exhibit low toxicity. Consequently, for sediments that contain more sulfide than metal, most cationic metals are present as insoluble sulfides and are relatively nontoxic. The sulfide content in aquatic sediments is measured as acid-volatile sulfide (AVS), and the metals associated with the AVS are measured as simultaneously extracted metals (SEM). Details of the AVS/SEM solid-phase analysis are discussed under a separate method. When AVS exceeds SEM, then interstitial water metal concentrations remain low and metal toxicity is not anticipated. All of the metals need to be considered together because they have different binding constants with sulfide. Currently only cadmium, copper, lead, nickel, silver, and zinc are considered. However, if another sulfide-forming metal is present, it should also be considered in the ESB.

APPLICATION: An ESB index is calculated from SEM, AVS, and total organic carbon (TOC) in the aquatic sediment. The first step in calculating an ESB index is to determine the concentration of each SEM in moles by dividing the measured SEM concentration by the molar mass for each metal. The next step is to sum the moles of SEM as:

$$\sum SEM = SEM_{Cd} + SEM_{Cu} + SEM_{Pb} + SEM_{Ni} + SEM_{Zn} + 0.5SEM_{Ag}$$

The SEM for silver is halved because it is a monovalent cation, whereas the other metals are divalent cations. The ∑SEM - AVS fraction is divided by the organic carbon fraction of sediments (f_{OC}) to obtain the equation:

$$(\sum SEM - AVS) / f_{OC},$$

which is the basis for the ESB index (USEPA, 2005). The ESB index is reported as µmol/g organic carbon (OC). Sediments with an ESB index value less than 130 µmol/g OC are predicted to be nontoxic and those with values greater than 3,000 µmol/g OC are

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predicted to be toxic. Intermediate values indicate uncertain toxicity and require additional testing.

An alternative approach is to assess the aggregate toxicity of metals in the interstitial water. This is done by dividing the dissolved concentration of each of the metals in the interstitial water by their respective final chronic value (FCV) from the appropriate USEPA aquatic life water-quality criteria. If the sum of the ratios is ≤ 1.0, then the sediment should not cause direct toxicity (USEPA, 2005).

LIMITATIONS: EqP assumes equilibrium between the sediment and interstitial water, which may not be the case in locations near a point source of contamination shortly after release of the contaminants. Also, metal bioaccumulation may occur even in situations when AVS > SEM. The parameters needed to apply ESBs (e.g., AVS, SEM, TOC) are not routinely collected during most monitoring programs. Aggregate toxicity of metals in interstitial water assumes additivity of metal toxicity.

BIBLIOGRAPHY:

Ahlf, W., Drost, W., and Heise, S. 2009. Incorporation of metal bioavailability into regulatory frameworks—metal exposure in water and sediment. *J. Soils Sediments* 9:411–419.

Ankley, G.T., Di Toro, D.M., Hansen, D.J., and Berry, W.J. 1996. Technical basis and proposal for deriving sediment quality criteria for metals. *Environ. Toxicol. Chem*. 15:2056–2066.

Bufflap, W.E., and Allen, H. E. 1995. Sediment interstitial water collection methods: A review. *Water Resources* 29:65–177.

Di Toro, D.M., Zarba, C.S, Hansen, D.J., Berry, W.J., Swartz, R.C., Cowan, C.E., Pavlou, S.P., Allen, H.E., Thomas, N.A., and Paquin, P.R. 1991. Technical basis for establishing sediment quality criteria for non-ionic organic chemicals using equilibrium partitioning. *Environ. Toxicol. Chem.* 10:1541–1583.

MacDonald, D.D., Ingersoll, C.G., and Berger, T.A. 2000. Development and evaluation of consensus-based sediment quality guidelines for freshwater ecosystems. *Arch. Environ. Contam. Toxicol*. 39:20–31.

Salomons, W., de Rooij, N.M., Kerdijk, H., and Bril, J. 1987. Sediments as a source for contaminants? *Hydrobiol*. 149:13–30.

Simpson, S.L., and Batley, G.E. 2007. Predicting metal toxicity in sediments: A critique of current approaches. *Integrated Environmental Assessment and Management* 3:18–31.

USEPA (U.S. Environmental Protection Agency). 2005. *Procedures for the Derivation of Equilibrium Partitioning Sediment Benchmarks (ESBs) for the Protection of Benthic Organisms: Metal Mixtures (Cadmium, Copper, Lead, Nickel, Silver and Zinc)*. U.S. Environmental Protection Agency Office of Research and Development, EPA-600-R-02-011.

Iron Speciation

(Richard T. Wilkin)

MEDIA: Sediment, Soils, and Aquifer Solids

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MEASUREMENT OBJECTIVES: The objective of chemical extraction methods for iron is to evaluate the solid-phase partitioning of iron and to determine the association of various trace metals with particular mineral fractions. Nondestructive analytical methods, such as X-ray diffraction, are often not effective in quantitative identification of iron minerals because of their low sensitivity. Selective sequential or single-step extractions are common procedures that can provide useful information on element partitioning within sediments and soils.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): Basic procedures for determining iron speciation are described by Kostka and Luther (1994). In their procedure the following chemical extractants are used: ascorbate (pH 8), oxalate (pH 2.5), dithionite (pH 4.8), and HCl (0.5 M). Ascorbate is intended to remove amorphous Fe(III) oxides (ferrihydrite) from a solid material. Oxalate is intended to remove amorphous Fe(III) oxides, crystalline Fe(III) oxides, and acid-volatile sulfides (ferrihydrite, magnetite, mackinawite, but not goethite). Dithionite is intended to selectively dissolve amorphous Fe(III) oxides and crystalline Fe(III) oxides (ferrihydrite, hematite, magnetite, and goethite). Dilute hydrochloric acid is intended to selectively dissolve amorphous Fe(III) oxides and acid-volatile sulfides (ferrihydrite and mackinawite).

ANALYTICAL TECHNIQUE(S): Iron measurements are made on the solution extracts using AA or ICP methods.

LIMITATIONS/INTERFERENCES: It is important to note that, although chemical extractions attempt to isolate and dissolve particular mineral fractions, the overall efficiency of this procedure depends on the affinity of the extractant solution for the targeted mineral. Chemical extractions are at best "operationally defined" and should be referenced as a method of extraction rather than as a target phase. Note the method described by Kostka and Luther does not specifically target pyrite (FeS_2). The chromium reduction method can be used to determine the amount of pyritic sulfur in sediment and soil materials (Canfield et al., 1986). In all selective extraction procedures, the amount of iron extracted with each extractant solution should be calculated based on the weight percentage and compared to the total amount of iron in the sample. To measure total iron in a sample, total digestion using multiple acids or analysis by X-ray fluorescence is required.

BIBLIOGRAPHY:

Canfield, D.E., Raiswell, R.R., Westrich, J.T., Reaves, C.M., and Berner, R.A. 1986. The use of chromium reduction in the analysis of reduced inorganic sulfur in sediments and shales. *Chemical Geology* 54:149-155.

Kostka, J.E., and Luther, G.W. 1994. Partitioning and speciation of solid phase iron in saltmarsh sediments. *Geochimica et Cosmochimica Acta* 58:1701-1710.

Trolard, F., Bourrie, G., Jeanroy, E., Herbillon, A., and Martin, H. 1995. Trace metals in natural iron oxides from laterites: A study using selective kinetic extraction. *Geochimica et Cosmochimica Acta* 59:1285-1297.

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Meteoric Water Mobility Procedure (MWMP)

(Charles H. Bucknam)

MEDIA: Solids

MEASUREMENT OBJECTIVES: The MWMP is a short-term leaching method developed by the State of Nevada and Nevada Mining Association that is intended to determine the mobility of inorganic species, including metals and wet chemistry parameters, such as cations and anions in mine rock and solid wastes. Results from overnight testing are compared with a list of parameters developed by the particular jurisdiction to determine potential impacts to surface and ground waters (i.e. Nevada Profile II).

Typical analysis includes: alkalinity, total and bicarbonate, aluminum, antimony, arsenic, barium, beryllium, bismuth, boron, cadmium, calcium, chloride, chromium, cobalt, copper, fluoride, gallium, iron, lead, lithium, magnesium, manganese, mercury, molybdenum, nickel, nitrate-nitrogen, nitrite0nitrogen, pH, phosphorus, potassium, scandium, selenium, silver, sodium, strontium, sulfate, thallium, tin, titanium, total dissolved solids, vanadium, weak and dissociable (WAD) cyanide and zinc.

The method is broadly comparable to the synthetic precipitation leaching procedure (SPLP) but different leachate solutions and volumes are used, de-ionized water. In the MWMP waste materials are treated with de-ionized water in an equal weight of the dry test sample. Nominal 5-kg aliquots of waste material are leached with nominal 5-L volumes of de-ionized water a column for 24 hours until approximately 5-L of extract is collected. The temperature is maintained at 23 ±2°C during the extraction. An ASTM version of the method has been subjected to interlaboratory testing to determine the within laboratory and between laboratory reproducibility. Reproducibility is generally poor, with the reproducibility index ranging from 9-258 percent of the mean, and eleven of the 38 parameters evaluated (28%) providing non quantitative results.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): The test field sample is screened at 2" particle size and oversize crushed to just pass the 2" screen prior to sub-sampling. Leaching is carried out in laboratory columns. Laboratory pH meters and electrodes are needed to measure the final pH of the extractant solution. After extraction, liquid and solids are separated by filtration. The liquid phase is preserved for analysis. Metals aliquots must be acidified with nitric acid to pH<2. Analyte concentrations in the MWMP extract are compared with appropriate regulatory levels in the State of Nevada. The standard filter used in the MWMP is a 0.45 μm filter.

ANALYTICAL TECHNIQUE(S): A variety of methods can be used to measure concentrations of components in the leach solution. For inorganic components, titration, gravimetric, ion chromatographic, colorimetric, combustion, flow injection amperometric, AA, ICP and ICP-MS methods may be appropriate.

LIMITATIONS/INTERFERENCES: If recovery of solution from the column apparatus is limited modified extraction procedures are used for such as SPLP or bottle roll tests. Reproducibility studies indicate that the most significant contributor to poor MWMP precision is related to solid sample heterogeneity.

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BIBLIOGRAPHY:

ASTM E2242 - 07 Standard Test Method for Column Percolation Extraction of Mine Rock by the Meteoric Water Mobility Procedure, American Society for Testing and Materials International, West Conshohocken, PA, 2010, www.astm.org, accessed 8/23/13.

Sequential, Selective, and Partial Extractions

(Kathleen S. Smith)

MEDIA: Solids (extraction)

MEASUREMENT OBJECTIVES: Sequential, selective, and partial extractions provide semi-quantitative information about element partitioning within operationally defined fractions in earth materials (e.g., soil, sediment, mine waste). Selective extractions are based on the premise that chemical reagents (extractants) can remove trace elements from specific fractions (e.g., carbonates, oxides, organic matter) of the solid phase. The effectiveness of extractions depends on the specificity of the extractant for the target fraction.

Selective extraction of specific mineralogical/geochemical fractions in earth materials relates geochemistry to ecosystem and human health. This is because the total-element content provides little information about potential availability to biota or the environment. The mobility, bioavailability, and ecotoxicity of trace metals are a function of their residence phase in solid materials. Extraction procedures can be element specific, mineralogical phase specific, or application specific. There are a number of extraction procedures that have been used, but none of these procedures serves as a universal procedure for all applications, and all of the procedures have limitations. Extraction procedures can provide useful information and insights when planned and interpreted with the aforementioned caveats in mind, and they are relatively inexpensive to perform.

Sequential extraction is a technique in which a series of extractants is applied in sequence to one solid, and the extractants are applied in order of increasing aggressiveness. The extractants are selected for their ability to react with different fractions of the solid material and to release the associated trace metals into soluble forms within the extractant solution. The extractant solution usually is separated from the solid phase by centrifugation. Many sequential extraction schemes are modifications of a procedure developed by Tessier et al. (1979), which includes the following fractions: 1) exchangeable, 2) carbonates, 3) iron and manganese oxides, 4) organic matter, and 5) residual. In the 1990s a simple three-step extraction procedure (the BCR procedure) was developed by the Commission of the European Communities, Community Bureau of Reference (now called the Standards, Measurement and Testing Program) in an attempt to provide a standard sequential-extraction procedure for trace metals (Ure et al., 1993). However, significant inter-laboratory variability was apparent upon certification and the procedure was reevaluated. A modified BCR procedure was developed by Rauret et al. (1999, 2000) and is summarized in Table A3.13. The modified BCR sequential extraction procedure has been widely applied to soil and sediment samples and standard reference materials.

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TABLE A3.13 Modified BCR sequential extraction procedure.

Step	Extractants	Phases
1	0.11 M acetic acid (CH_3COOH; shake 16 hr at 22±5°C)	Exchangeable and water/acid soluble (soluble, carbonates, adsorbed)
2	0.5 M hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$; pH 1.5; shake 16 hr at 22±5°C)	Reducible (manganese and iron oxides)
3	8.8 M hydrogen peroxide (H_2O_2; digest 1 hr at room temp; heat at 85°C for 1 hr; add more H_2O_2; heat at 85°C for 1 hr; cool) Add 1 M ammonium acetate (CH_3COONH_4; pH 2; shake 16 hr at 22±5°C)	Oxidizable (organic matter)

Source: information from Rauret et al. 1999 and 2000.

SPECIFIC TECHNIQUE(S): Reviews of sequential-extraction procedures and extractants are provided by Chao (1984), Filgueiras et al. (2002), and Hass and Fine (2010). Many selective and sequential extractions are applied according to the mineralogical phases with which the particular extractants are expected to react. Some examples of reagents used for particular fractions are listed below.

Readily soluble: H_2O
This extraction step often is omitted because of the generally low concentration of water-soluble metals. An exception is readily soluble salts that are common in mine-waste material.

Exchangeable: NH_4OAc, $MgCl_2$, $CaCl_2$, KNO_3, $Mg(NO_3)_2$, $Ca(NO_3)_2$
This extraction step involves releasing exchangeable metals by shaking the sample in a concentrated solution of an inert electrolyte. Use of acetate salts may attack other phases such as carbonates or oxyhydroxides. Both pH and ionic strength effects can result in metal release from other phases.

Adsorbed: KF, $Pb(NO_3)_2$, EDTA in NH4OAc, NH_4 or Na or H acetate
This extraction step is rarely included in sequential extraction schemes. Release of specifically adsorbed metals generally involves competition for adsorption sites or complexation with the surface to release metals.

Carbonate: HOAc, NaOAc at pH 5, EDTA at pH 6.5
This extraction step usually is the first acidic extraction in a sequence. Selectivity of this step often is problematic.

Organic matter: NaOH, H_2O_2, $NaClO_4$, DTPA, EDTA, $Na_4P_2O_7$
Mikutta et al. (2005) provide a detailed review of methods used to remove organic matter. This extraction step hydrolyzes, oxidizes, or disperses organic matter to release

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the associated metals. Release of metals associated with easily reducible oxides is problematic for this extraction. Precipitation or adsorption of released metals also is an issue for this extraction.

Oxyhydroxides:
Oxyhydroxides commonly are extracted under two or three subcategories including manganese oxides, amorphous iron oxides, and crystalline iron oxides. A wide variety of acids, reducing agents, and ligands have been used to attempt to selectively attack various oxyhydroxides. For manganese oxides, 0.1 M $NH_2OH{\cdot}HCl$ in 0.01 N HNO_3 has been used. For amorphous iron oxides a mixture of ammonium oxalate and oxalic acid at pH 3.25 in the dark (also known as Tamm's reagent) or a mixture of 0.25 M $NH_2OH{\cdot}HCl$ in 0.25 N HCl at 50°C have been used. For crystalline iron (and aluminum) oxides $Na_2S_2O_4$ in a citrate-bicarbonate buffer has been used as has Tamm's reagent under UV radiation at 80-100°C. Also, a mixture of $NH_2OH{\cdot}HCl$ with acetic acid at pH 2 and 96-100°C or a mixture of ammonium oxalate and oxalic acid and ascorbic acid at pH 3 and 97°C or strong acids (e.g., 4 N HCl at 95°C) have been used to selectively dissolve crystalline oxides.

Residual: The residual component consists of all the minerals that are resistant to the previous steps. Various acids or combinations of acids usually are used for this extraction. See the separate method on dissolution for further information.

Biological and regulatory extractions and dissolution are discussed under separate methods.

ANALYTICAL TECHNIQUE(S): A variety of analytical methods can be used to measure concentrations of constituents in the extracts. Most procedures rely on solution measurements, which may have interferences from the matrix of the extractants. Occasionally techniques such as X-ray diffraction, scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS), or isotopic analysis of the solid phase after extraction have been used.

LIMITATIONS/INTERFERENCES: There are a wide variety of extractants and extraction procedures that have been used, which makes it very difficult to compare datasets and results. Sample pretreatment, such as drying, can affect the partitioning of metals (e.g., Zhang et al., 2001) and use of fresh wet sediments is strongly recommended for extraction procedures. La Force and Fendorf (2000) suggest that selective extractions be used in conjunction with spectroscopic techniques, when possible, to fully ascertain elemental partitioning within the solid phase. Selectivity of a given extractant for a specific fraction may be limited or an extracted element may readsorb during an extraction sequence (e.g., Bunzl et al., 1999). Redox-sensitive elements may change oxidation states during an extraction sequence, resulting in erroneous conclusions regarding its partitioning (e.g., Gruebel et al., 1988).

BIBLIOGRAPHY:
Bunzl, K., Trautmannsheimer, M., and Schramel, P. 1999. Partitioning of heavy metals in a soil contaminated by slag: a redistribution study. *J. Environ. Qual*. 28:1168–1173.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Chao, T.T. 1984. Use of partial dissolution techniques in geochemical exploration. *J. Geochem. Explor.* 20:101–135.

Filgueiras, A.V., Lavilla, I., and Bendicho, C. 2002. Chemical sequential extraction for metal partitioning in environmental solid samples. *J. Environ. Monit*. 4:823–857.

Gruebel, K.A., Davis, J.A., and Leckie, J.O. 1988. The feasibility of using sequential extraction techniques for arsenic and selenium in soils and sediments. *Soil Sci. Soc. Am. J*. 52:390–397.

Hass, A., and Fine, P. 2010. Sequential selective extraction procedures for the study of heavy metals in soils, sediments, and waste materials—a critical review. *Crit. Rev. Environ. Sci. Tech.* 40:365–399.

La Force, M.J., and Fendorf, S. 2000. Solid-phase iron characterization during common selective sequential extractions. *Soil Sci. Soc. Am. J*. 64:1608–1615.

Mikutta, R., Kleber, M., Kaiser, K., and Jahn, R. 2005. Review: Organic matter removal from soils using hydrogen peroxide, sodium hypochlorite, and disodium peroxodisulfate. *Soil Sci. Soc. Am. J*. 69:120–135.

Rauret, G., Lopez-Sanchez, J.F., Sahuquillo, A., Rubio, R., Davidson, C., Ure, A., and Quevauviller, Ph. 1999. Improvement of the BCR three step sequential extraction procedure prior to the certification of new sediment and soil reference materials. *J. Environ. Monit*. 1:57–61.

Rauret, G., Lopez-Sanchez, J.-F., Sahuquillo, A., Barahona, E., Lachica, M., Ure, A.M., Davidson, C.M., Gomez, A., Luck, D., Bacon, J., Yli-Halla, M., Muntau, H., and Quevauviller, Ph. 2000. Application of a modified BCR sequential extraction (three-step) procedure for the determination of extractable trace metal contents in a sewage sludge amended soil reference material (CRM 483), complemented by a three-year stability study of acetic acid and EDTA extractable metal content. *J. Environ. Monit*. 2:228–233.

Tessier, A., Campbell, P.G.C., and Bisson, M. 1979. Sequential extraction procedure for the speciation of particulate trace metals. *Anal. Chem*. 51:844–851.

Ure, A.M., Quevauviller, P., Muntau, H., and Griepink, B. 1993. Speciation of heavy metal in soils and sediments. An account of the improvement and harmonization of extraction techniques undertaken under the auspices of the BCR of the Commission of the European Communities. *Int. J. Environ. Anal. Chem.* 51:135–151.

Zhang, S., Wang, S., and Shan, X-q. 2001. Effect of sample pretreatment upon the metal speciation in sediments by a sequential extraction procedure. *Chem. Spec. Bioavail*. 13:69–74.

Synthetic Precipitation Leaching Procedure (SPLP)

(Richard T. Wilkin and Charles H. Bucknam)

MEDIA: Liquids, solids, typically water rock and tailings

MEASUREMENT OBJECTIVES: The SPLP (Synthetic Precipitation Leaching Procedure) is a short-term leaching method developed by the USEPA that is intended to determine the mobility of both organic and inorganic species in liquid and solid wastes. The method is broadly comparable to the Toxicity Characteristic Leaching Procedure (TCLP) but different leachate (non-acetate) solutions are used, either the actual rain-

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water pH estimated for the site eastern and western US default pH values or de-ionized water if the Nevada convention for meteoric water mobility is being followed. This procedure is applicable for materials where the leaching potential due to normal rainfall is to be determined instead of landfill leachate. In the SPLP, waste materials are treated with one or two extractant solutions. The acidic solutions are prepared by diluting a sulfuric acid plus nitric acid mixture at the ratio 60% by weight sulfuric acid plus 40% nitric acid with water until the pH is the pH of the acidic rainfall at the site, eastern site 4.20 ±0.05 or western site 5.00 ±0.05. The non-acidified extraction solution is water and is used to determine the leachability of cyanide, volatiles, or inorganics if the Nevada convention is being followed. Aliquots of waste material are leached with specified volumes of these extractant solutions in a sealed vessel for 18 ±2 hours and continuously rotated at 30 ± 2 rpm. The temperature is maintained at 23 ±2°C during the extraction. An ASTM version of the method has been developed specifically for inorganic analysis of relatively dry mining and mineral processing wastes that uses nominal 100-g solids and 2-L extraction volume.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): The SPLP varies depending on the solid-liquid ratio in the waste material. The apparatus used during the extraction can be obtained from several different vendors. A zero-headspace extraction vessel (ZHE) is used if the waste is being tested for the mobility of volatile species. The vessels have an internal volume of 500-600 mL and are equipped to hold 90-110 mm diameter filters. The vessels are composed of inert materials that do not leach or adsorb waste components. Laboratory pH meters and electrodes are needed to measure the final pH of the extractant solution. After extraction, liquid and solids are separated by filtration. The liquid phase is preserved for analysis. Metals aliquots must be acidified with nitric acid to pH<2. Analyte concentrations in the SPLP extract are compared with appropriate regulatory levels. The standard filter used in the SPLP is a glass fiber 0.7µm filter.

ANALYTICAL TECHNIQUE(S): A variety of methods can be used to measure concentrations of components in the leach solution. For inorganic components, titration, gravimetric, ion chromatographic, colorimetric, combustion, flow injection amperometric, AA, ICP and ICP-MS methods may be appropriate.

LIMITATIONS/INTERFERENCES: If precipitation is observed upon acidification with nitric acid, then the liquid phase should be analyzed as soon as possible without acidification. Particle size reduction is required if the waste material fails to pass through a 9.5 mm standard sieve. Modified extraction procedures are used for wastes containing volatile compounds and for mixed solid plus liquid wastes. Reproducibility studies indicate that the most significant contributor to poor SPLP precision is related to solid sample heterogeneity.

BIBLIOGRAPHY:

ASTM D6234-98. 2007. *Standard Test Method for Shake Extraction of Mining Waste by the Synthetic Precipitation Leaching Procedure*. West Conshohocken, PA: ASTM International. Available from www.astm.org. accessed 8/23/13.

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USEPA (U.S. Environmental Protection Agency). 1992. *Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods*: 3rd edition. Office of Solid Waste, SW-846, Method 1312, U.S. Government Printing Office, Washington, D.C.

Toxicity Characteristic Leaching Procedure (TCLP)

(Richard T. Wilkin)

MEDIA: Liquids, Solids

MEASUREMENT OBJECTIVES: The TCLP is a regulatory tool that is intended to determine the mobility of both organic and inorganic species in liquid and solid wastes. The inorganic target list of elements is As, Ag, Ba, Cd, Cr, Hg, Pb, and Se. Volatile organic compounds include: benzene, carbon tetrachloride, chlorobenzene, chloroform, 1,4-dichlorobenzene, 1,1-dichloroethene, methyl ethyl ketone, tetrachloroethene, trichloroethene, and vinyl chloride. In this procedure test materials are treated with two extractant solutions. One solution is 0.1 N acetic acid plus 0.064 N NaOH (pH 4.9), and the second solution is 0.1 N acetic acid (pH 2.9). Aliquots of waste material are leached with specified volumes of these extractant solutions in a sealed vessel for 18 ±2 hours and continuously rotated at 30 rpm. The temperature is maintained at 23 ±2°C during the extraction.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): The TCLP varies depending on the solid-liquid ratio in the waste material. The apparatus used during the extraction can be obtained from several different vendors. A zero-headspace extraction vessel (ZHE) is used if the waste is being tested for the mobility of volatile species. The vessels have an internal volume of 500-600 mL and are equipped to hold 90-110 mm diameter filters. The vessels are composed of inert materials that do not leach or adsorb waste components. Laboratory pH meters and electrodes are needed to measure the final pH of the extractant solution. After extraction, liquid and solids are separated by filtration. The liquid phase is preserved for analysis. Metals aliquots must be acidified with nitric acid to pH<2. Analyte concentrations in the TCLP extract are compared with appropriate regulatory levels. The standard filter used in the TCLP is a glass fiber 0.7µm filter.

ANALYTICAL TECHNIQUE(S): A variety of methods may be used to measure concentrations of components in the leach solution. For inorganic components, AA and ICP methods may be appropriate (EPA SW-846 methods).

LIMITATIONS/INTERFERENCES: If precipitation is observed upon acidification with nitric acid, then the liquid phase should be analyzed as soon as possible without acidification. Particle size reduction is required if the waste material fails to pass through a 9.5 mm standard sieve. Modified extraction procedures are used for wastes containing volatile compounds and for mixed solid plus liquid wastes. Reproducibility studies indicate that the most significant contributor to poor TCLP precision is related to sample heterogeneity.

BIBLIOGRAPHY:

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USEPA (U.S. Environmental Protection Agency). 1992. *Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods*: 3rd edition. Office of Solid Waste, SW-846, Method 1312, U.S. Government Printing Office, Washington, D.C.

USGS Field Leach Test (USGS FLT)

(Kathleen S. Smith)

MEDIA: Solids

MEASUREMENT OBJECTIVES: The Field Leach Test (USGS FLT; Hageman, 2007) was developed by the U. S. Geological Survey (USGS) as a fast, simple, and cost-effective leach test to screen and prioritize mining wastes for their water reactivity. The FLT is a short-term batch-extraction procedure that releases readily soluble components in earth materials. It has been used to screen and prioritize mine-waste piles for acid generation and metal release (e.g., Hageman, 2004; Smith and others, 2007), and to help characterize a number of other diverse earth-material matrices (such as soil, sediment, volcanic ash, biosolids, and dusts). The FLT can be performed both in the field and in the laboratory. Although the FLT is not a regulatory test, comparison of results obtained for leaching of mine-waste material using the FLT with results obtained using the Toxicity Characteristic Leaching Procedure (TCLP) and the Synthetic Precipitation Leaching Procedure (SPLP) indicate that the FLT is a good tool for the quick estimation of leaching potential (Hageman and Briggs, 2000; Al-Abed et al., 2006).

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUE(S): The FLT uses deionized water to leach a sample at a 20:1 ratio. This extraction ratio reduces the likelihood of exceeding solubility limits of dissolved constituents while still providing constituent concentrations that can be detected using modern analytical techniques. The leaching procedure is conducted in a plastic bottle, and uses 100.0 g of sample in 2 L of water. The sample/water mixture is shaken for 5 min. and allowed to settle for 10 min. Subsamples are collected for pH and specific conductance measurements, and additional subsamples are collected, filtered (0.45 micrometer filter) using a syringe, and preserved for analysis of selected constituents. Subsamples for metal analyses must be stored in plastic or Teflon containers and be acidified with nitric acid to pH <2. For earth materials such as mining waste and soil, the FLT uses the <2 mm size fraction for leaching. This size fraction provides a worst-case scenario for short-term leaching and provides a reasonable sample mass for field collection while reducing the sampling error (Smith et al., 2000).

ANALYTICAL TECHNIQUE(S): A variety of analytical methods can be used to measure concentrations of constituents in the leachate. For use in the field, constituents can be measured using field instruments or field kits.

LIMITATIONS/INTERFERENCES: The FLT is designed to leach only the readily soluble components in an earth material. Sample heterogeneity can lead to poor reproducibility.

BIBLIOGRAPHY:

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Al-Abed, S.R., Hageman, P.L., Jegadeesan, G., Madhavan, N., and Allen, D. 2006. Comparative evaluation of short-term leach tests for heavy metal release from mineral processing waste. *Sci. Tot. Env.* 364:14–23.

Hageman, P.L. 2004. *Use of Short-Term (5-minute) and Long Term (18-hour) Leaching Tests to Characterize, Fingerprint, and Rank Mine Waste Material from Historical Mines in the Deer Creek, Snake River, and Clear Creek Watersheds in and Around the Montezuma Mining District, Colorado*. Scientific Investigations Report 2004–5104. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/sir/2004/5104/. Accessed 8/23/13.

Hageman, P.L. 2007. *U.S. Geological Survey Field Leach Test for Assessing Water Reactivity and Leaching Potential of Mine Wastes, Soils, and Other Geologic and Environmental Materials*. Techniques and Methods, book 5, chap. D3. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/tm/2007/05D03/pdf/TM5-D3_508.pdf. Accessed 8/23/13.

Hageman, P.L., and Briggs, P.H. 2000. A simple field leach for rapid screening and qualitative characterization of mine waste material on abandoned mine lands. In *Proceedings, 5th International Conference on Acid Rock Drainage (ICARD 2000)*. Littleton, CO: Society for Mining, Metallurgy, and Exploration. p. 1463–1475. http://crustal.usgs.gov/projects/minewaste/pdfs/hageman1.pdf. Accessed 8/23/13.

Smith, K.S., Hageman, P.L., Briggs, P.H., Sutley, S.J., McCleskey, R.B., Livo, K.E., Verplanck, P.L., Adams, M.G., and Gemery-Hill, P.A. 2007. *Questa Baseline and Pre-Mining Ground-Water Quality Investigation: 19. Leaching Characteristics of Composited Materials from Mine Waste-Rock Piles and Naturally Altered Areas near Questa, New Mexico*. Scientific Investigations Report 2006–5165. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/sir/2006/5165/. Accessed 8/23/13.

Smith, K.S., Ramsey, C.A., and Hageman, P.L. 2000. Sampling strategy for the rapid screening of mine-waste dumps on abandoned mine lands. In *Proceedings, 5th International Conference on Acid Rock Drainage (ICARD 2000)*. Littleton, CO: Society for Mining, Metallurgy, and Exploration. p. 1453–1461. http://crustal.usgs.gov/projects/minewaste/pdfs/kathy.pdf. Accessed 8/23/13.

X-ray Absorption Spectroscopy (XAS)

(Andrea L. Foster)

MEDIUM: Solids including plants, sediment, rock

PRINCIPLE: X-ray absorption spectroscopy (XAS) is an element-specific technique that provides information about the electronic structure (including but not limited to nominal valence) and molecular-scale coordination environment of the absorbing element (i.e., the type, number, and average distance of atoms within about 70 nanometers radius). These two pieces of information are usually characteristic of a given elemental species, therefore it is often said that XAS provides "speciation" information. The technique can be performed on all states of matter, but for environmentally focused work is usually performed on solid media (plants, sediment, rock). XAS is not typically an in-house technique, but it is primarily performed at synchrotron facilities where the high-

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energy X-rays required for the experiment can be generated. (A synchrotron is a circular device for accelerating photoelectrons to speeds approaching that of the speed of light; under these conditions, X-rays are produced in ports connected tangentially to the circular ring. A variety of electromagnets are used to control the orbit of the photoelectrons in the "storage ring" and the characteristics of the X-rays emanating from the beam ports. Synchrotron rings vary in size from about 0.1 to 1 km in diameter. More about synchrotron facilities and their uses can be found at www.lightsources.org (accessed 8/23/13). XAS spectra are typically obtained by exposing the sample to a beam of X-rays and measuring the quantity of X-ray beam absorbed by (or fluorescent X-rays emitted from) the sample as a function of beam incident energy. XAS experiments are usually performed at energies between 4 and 30 kilo-electron volts (keV), which allows for the examination of most major and trace elements.

The brevity of this review does not allow a complete description of the techniques or of their applications and limitations: the reader is referred to Koningsberger and Prins (1987), Fenter et al. (2002), and Kelly et al. (2008) for additional information and references.

PRIMARY USES: XAS has been widely used to determine elemental speciation in natural samples, which in many cases has been information vital to determining some key aspect of the element's geochemical, biological, or toxicological characteristics. XAS has been applied to many studies of trace metal fate and transport related to mining activities; in those studies XAS has helped to identify primary and secondary forms of elements, to determine mechanisms of trace element dispersal (such as ternary complex formation of Cu, Pb, Zn, and Cd with SO_4 on ferric (hydr)oxides; Swedlund et al., 2009), and to determine the stoichiometry of adsorbed species for use in modeling (Ponthieu et al., 2006). XAS has also been used to measure the efficacy of chemical and biological solid waste remediation treatments, and to determine the form and distribution of metals in plant and animal tissues. A review of publication lists available at just one of the facilities where XAS data are collected demonstrate the wide variety of systems to which XAS has been applied (see http://www-ssrl.slac.stanford.edu/publications/index.html, accessed 8/23/13). Table A3.14 below provides primary uses of some of the subtechniques available under the umbrella term "XAS."

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TABLE A3.14 Summary of limitations and interferences for selected synchrotron X-ray techniques.

Technique	Sample size (solids)	Primary use	Limitations	Interferences	Lower Detection Limit* (approximate)
Bulk XAS (4-30 keV)	< 0.75g	Weight-averaged speciation	Difficult to identify minor species	Absorption lines of other elements (As on Pb)	XANES: 1 ppm EXAFS: 200 ppm
Micro-XAS (4-30 keV)	Microgram quantities; usually as thin section	Species distribution; identification of minor species	Sample representativeness; difficult to obtain high-quality EXAFS	Trace elements in mounting media	Not generally defined; study specific. Low ppm-high ppb range
Micro-Xray Fluorescence (4-30 keV)	Microgram quantities; usually as thin section or whole specimen	Element distribution and nominal valence or nominal species distribution maps of micrometer scale	Sample representativeness; Valence and/or species differentiation must be specific and accessible with the resolution of the beamline	Trace elements in mounting media; X-ray emission line overlap among elements	Not generally defined; study specific. Low ppm-high ppb range

*The upper detection limit in XAS is defined as that concentration at which artifacts in the spectrum are discernible (self-absorption; see Kelley et al., 2008); for most elements it is about 1%, but for others can be considerably less.

INTERFERENCES:

Matrix effects: X-ray fluorescence from high-abundance elements (such as Fe in mine wastes) can exceed that of the element of interest, and produce non-linear detector performance. Unwanted fluorescence is attenuated upstream of the detector, when possible. Tables are available to estimate the magnitude of these effects if the chemistry of the sample is known or can be estimated.

Spectral interferences: X-ray fluorescence of similar or equivalent energy can arise from two different elements, if the fluorescence arises from energy transitions within two different orbitals (usually the K and L in trace metal/metalloid work). A classic example important in mine wastes is the interference of AsK_{α} on Pb $L_{\beta III}$ X-ray fluorescence. Both emission lines fall at 10.5 keV, but the X-ray absorption event related to this emission occurs at 11.867 keV for As and 13.050 keV for Pb. Therefore As interferes with Pb, but not vice versa. Tables are

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available to estimate the magnitude of these effects if the chemistry of the sample is known or can be estimated.

DETECTION LIMITS: Detection limits in XAS are element and technique-specific; general guide lines are given for each technique in Table A3.14.

MASS/VOLUME REQUIREMENTS: The amount of sample required is highly dependent on the matrix, water content, metal content, and technique, and ranges from as little as 0.01grams to about 0.5 grams (powdered dry sediment used for this estimate) for bulk XAS/XRF. Thin sections of 30-60 μm thickness are typically used for micro-XAS and XRF experiments—the amount of sample analyzed is even smaller, but approximately the same amount of powdered material would be required to prepare the section (see below).

SAMPLE PRESERVATION/PREPARATION: Sediment or other solid sample to be tested should be collected into acid-washed plastic containers, and immediately frozen (dry ice or liquid nitrogen are used in the field for this purpose) and/or stored at -20 ºC until analysis. If samples are anaerobic care should be taken to maintain in that way, by use of an anaerobic box. even in the freezer. Since the disbursement of beam time is not under user control, preservation of samples until beam time is extremely important and much thought should be given to not only the means of preservation but also to the measures that will be used to test whether the preservation techniques were effective. Sample preparation for bulk XAS/XRF can be as simple as loading wet or dry sediment into a holder, but can also be more sophisticated. Sieving and mineral separation by gravity, magnetism, or other characteristics may be required in order to effectively concentrate the element of interest or two separate mixed species. Sample preparation for micro-XAS/XRF can be minimal or zero if the sample is physically large and can be accommodated in the available sample holders. In the analysis of mine wastes, dried material is typically impregnated with epoxy, sliced into thin sections as described above, and polished. This provides a sample of defined thickness that can be analyzed with complementary techniques such as electron microprobe or micro-Raman spectroscopy. Often these techniques are used in concert with micro-XAS and it is useful to be able to analyze exactly the same area of a sample.

DATA FORMAT: The averaged, calibrated, background-subtracted, normalized XAS spectrum is separated into two parts for analysis and interpretation: the X-ray absorption near-edge (XANES) spectrum, which extends for 100-200 eV above the absorption edge, and the EXAFS spectrum, which begins approximately 100 eV above the absorption edge and extends to about 1000 eV above the edge. XANES spectra are used in their native form, or the smoothed first or second derivative may be analyzed to emphasize key spectral features in fits. The units of the EXAFS spectrum are converted from energy to momentum (in units of k, usually represented in inverse angstroms) and the spectrum is usually weighted by the cube of k for analysis (although with noisy spectra, k^2 or unweighted spectra may be analyzed). The analyzed forms of the data are simple, 2-column ASCII data files that can be imported by many different programs.

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LIMITATIONS: Interpretation of XAS spectra relies on adequate match of the sample spectrum to a physical or theoretical model of the species, and the solution may not be unique (i.e., another physical or theoretical model may provide an equally-good match to the data). Uncertainty in species assignment is more often a limitation of XANES spectra analysis than of EXAFS. There are several accepted methods of interpretation of XAS data, each with its own level of confidence (Kelly et al., 2008). The highest level of confidence is assigned to well-documented fits of EXAFS spectra that essentially reconstruct the observed EXAFS spectrum from theoretical (*ab initio*) calculations (Kelly et al., 2008; Sayers and Bunker, 1988; Rehr et al., 1992). In fits to EXAFS spectra collected from simple natural systems or from laboratory (model) systems, theoretical functions have largely supplanted those derived from physical models. However analysis of XANES spectra and EXAFS spectra of heterogeneous natural materials such as mine wastes still requires a representative library of model compound spectra collected from physical specimens. The per-sample data collection time depends on technique and sample, and can range from 20 minutes to 8 hrs per sample. In general, XAS and its affiliate techniques are not high-throughput, and are only employed after all other types of techniques have failed to provide the required speciation information.

COST: A great benefit of doing XAS is that the analytical time cost is funded by several government agencies (predominantly DOE and NIH). Users pay for travel, lodging, meals/incidentals, etc., and for minor reagents if they are using specialized equipment such as a low-temperature cryostat, which is cooled by liquid helium gas. All at-home laboratory and analytical costs are of course also borne by the user. These subsidies result in very low costs per sample relative to other techniques. Note that there is a per-hour charge for beamtime for for-profit users that is assessed.

ADDITIONAL CONSIDERATIONS: Beamtime is usually granted through one of two mechanisms: (1) collaboration with beamline scientists (and use of their allotted beamtime, usually resulting in co-authorship of resulting manuscripts) and (2) submission of a general user proposal and use of beamtime allotted to the specific proposal. Each synchrotron facility has different policies on this access, so it is best to check the websites. A one-stop link to the synchrotrons of the world can be found at http://www.lightsources.org/cms/ (accessed 8/23/13). There is also a long lead time for synchrotron work; first access to the synchrotron on a successful proposal can take up to 6 months.

BIBLIOGRAPHY:

Fenter, P.A. Rivers, M.L., Sturchio, N.C., Sutton, S.R. 2002. Volume 49: Applications of synchrotron radiation in low-temperature geochemistry and environmental science. In *Reviews in Mineralogy and Geochemistry*. Edited by P.H. Ribbe. Washington, D.C.: Mineralogical Society of America.

Kelly, S.D., Hesterberg, D. and Ravel, B. 2008. *Analysis of Soils and Minerals Using X-ray Absorption Spectroscopy*. Methods of Soil Analysis. Part 5. Mineralogical Methods. Vol. SSSA Book Series. Soil Science Society of America: Madison, WI. 387-464.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Koningsberger, D.C. and Prins, R., 1987. *X-ray Absorption: Principles, Applications, Techniques of EXAFS, SEXAFS, and XANES*. John Wiley & Sons. 688.

Ponthieu, M., Juillot, F., Hiemstra, T., van Riemsdijk, W.H. and Benedetti, M.F. 2006. Metal ion bonding to iron oxides. *Geochim. Cosmochim. Acta*, 70:2679–2698.

Rehr, J.J., Albers, R.C., and Zabinsky, S.I. 1992. High-Order Multiple-Scattering Calculations of X-ray Absorption Fine Structure. *Phys. Rev. Let.* 69:3397–3400.

Sayers, D.E. and B.A. Bunker. 1988. Data Analysis. In *X-ray Absorption: Principles, Applications, Techniques of EXAFS, SEXAFS, and XANES*. Edited by D.C. Koningsberger and R. Prins. New York: John Wiley and Sons.

Swedlund, P.J., Webster, J.G., and Miskelly, G.M. 2009. Goethite adsorption of Cu(II), Pb(II), Cd(II), and Zn(II) in the presence of sulfate: Properties of the ternary complex. *Geochim. Cosmochim. Acta*. 73:1548–1562.

Aquatic Macroinvertebrates and Habitat

(Travis S. Schmidt)

MEDIA: Biological (Aquatic Invertebrates)

MEASUREMENT OBJECTIVES: Aquatic invertebrate communities are a reliable indicator of the ecological effects ARD and mining related disturbances can impose on aquatic ecosystems. Sampling objectives could be to answer any of the following types of questions:

1) Is the diversity or proliferation of aquatic invertebrate populations impacted in areas where mining-related impacts are observed?
2) What aquatic invertebrates would we expect to be present at a given site?
3) Are aquatic invertebrates present at the expected frequency both temporally and spatially?
4) Is ARD a direct limiting factor for the aquatic invertebrate community?
5) What other factors (e.g., lack of suitable habitat) could be limiting of aquatic invertebrates at a given site?

ADVANTAGES OF INVERTEBRATES:

- Cost effective to collect in the field.
- Sensitive to changes in water and sediment quality and respond predictably to change.
- Sessile: often living their entire juvenile life stages within a small area.
- Life spans range from weeks to years- so they integrate changes in habitat over long time frames.
- Are near the base of the food chain—fish and riparian organisms depend on them as a major source of food.
- Are increasingly being used to determine if a water body is biologically impaired.
- Collecting aquatic invertebrates can require fewer permits and government permissions.

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DISADVANTAGES OF INVERTEBRATES:

- Sample processing and the conversion of data into information can be expensive and time consuming.
- Precise identification requires skill and training.
- Aquatic invertebrates are highly variable in response to disturbance and natural conditions making it difficult to attribute subtle changes to a specific cause.
- Assessments of aquatic invertebrates are site-specific. These assessments must be performed at each site and are performed more than once to evaluate potential on-going changes in the ecosystem.

SPECIFIC FIELD SAMPLING TECHNIQUES/EQUIPMENT: A variety of sampling techniques/approaches are available to evaluate aquatic invertebrate communities and the factors that influence their organization at any given site.

Aquatic invertebrate sampling in the field- several methods are available to capture aquatic invertebrates in the field for characterization of the community. Single and/or multiple habitat (i.e., pool, riffle, run) sampling approaches can be used. See citations for specifics.

a) **Quantitative samples**:
   i. **Hess sampler-** (presence/absence, densities, composition, richness and diversity per unit area) (Barbour et al., 1999; Cuffney et al., 1993; Moulton et al., 2002)
   ii. **Surber sampler-** (presence/absence, densities, composition, richness and diversity per unit area) (Barbour et al., 1999; Cuffney et al., 1993; Moulton et al., 2002)

b) **Qualitative samples:**
   i. **D-net-** (presence/absence, densities, composition, richness and diversity per unit area, time or effort) (Barbour et al., 1999; Cuffney et al., 1993; Moulton et al., 2002)

Collection of data in the field- a variety of information can be obtained through measurements obtained in the field and through proper preservation of tissues.

a) **Habitat assessment**- several protocols exist for quantifying habitat quality, a critical component to determining which factors limit aquatic invertebrates (Barbour et al., 1999; Cuffney et al., 1993; Moulton et al., 2002)
b) **Chemical characterization of algae, water, sediment**- chemical characterization of metal sources to invertebrates can be used to determine what limits the aquatic invertebrate community (Farag et al., 1998; Schmidt et al., 2002)
c) **Collection of tissue samples**- metal in invertebrate tissue can be used to assess the exposure to and bioavailability of metals (Cain et al., 2004; Luoma and Rainbow, 2005; Rainbow, 2002; Hare, 1992)

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STUDY DESIGN: The aquatic invertebrate community present at a given location is a function of the biological, physical and chemical conditions that exist at that site, which all may vary over time. Several experimental approaches exist to evaluate the invertebrate community response to a stressor such as ARD while accounting for the influence of the spatial/temporal dynamics of such communities.

a) BACI (Before, After, Control, Impact)- comparison with a control or reference site, or before and after the influence of ARD (Underwood, 1994)
b) Gradient-sample a large number of locations variously affected by ARD such that sample locations include little or no ARD to heavily affected by ARD (Schmidt et al., 2010)
c) Sequential sampling- sample over a period of time, probably seasonal to evaluate temporal variability in the aquatic community (Clark and Clements, 2006)
d) In-situ sampling- colonize microcosms in one stream and place them in an ARD influenced stream to measure the direct effect of ARD on an aquatic community from a stream not influenced by ARD (Clark and Clements, 2006)
e) Predictive model- model developed to measure the difference between the invertebrate communities expected verses that observed (Hawkins et al., 2000)

LABORATORY TECHNIQUES: Although field evaluations of aquatic invertebrate communities are an important component to understanding the impacts of ARD on aquatic ecosystems, they are more credible when paired with laboratory analyses (Clements and Kiffney, 1994)

Toxicity testing- exposing invertebrates to contaminants in a controlled laboratory setting -can yield valuable information about factors limiting aquatic invertebrates in the field (Long and Chapman, 1985).

a) Toxicity testing of water

i. **"Whole effluent" testing**- exposure of invertebrates to waters (and a complete dilution series) collected from the field site of interest or manufactured (USEPA, 2002a)
ii. **Sediment toxicity testing-** exposure of invertebrates to sediments from the field site of interest or manufactured (Burton, 1991; ASTM E1706)
iii. **Reference testing**- exposure of an invertebrate to individual contaminants of interest to isolate the effect of the contaminant of concern (USEPA, 2002a)
iv. **Acute vs. chronic-** different methods and endpoints for evaluating the toxicity of short-term (USEPA, 2002a) and prolonged exposures (USEPA, 2002b)
v. **Non-standard toxicity tests**-more ecologically relevant approach to measuring a biological response to a toxicant (Buchwalter et al., 2007; Clements et al., 1988; Kennedy et al., 2004)

Tissue analysis- Laboratory analyses of tissues collected in the field can produce lines of evidence linking contaminant exposure to biochemical effects.

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a) **Tissue metals concentrations**- Concentrations of metals in whole bodies or cellular fractions can provide evidence of metal (Cain et al., 2004; Farag et al., 1998)
b) **Metal-specific biochemical responses**- Measurement of parameters such as metallothionein can document physiological malfunction (Roesijadi, 1992)

ADDITIONAL CONSIDERATIONS: The observed condition of an aquatic invertebrate community at any given time represents an integrated response to the range of physical, chemical, and biological conditions present and previously-present at the site. The tools described above can be used as complementary lines of evidence in a comprehensive assessment of factors limiting the resident ecosystem (Clements and Kiffney, 1994; Long and Chapman, 1985).

BIBLIOGRAPHY:

ASTM E1706-05e. 2003. *Standard Test Method for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates*. West Conshohocken, PA: ASTM International. Available from www.astm.org. accessed 8/23/13

Barbour, M.T., Gerritsen, J., Snyder, B.D., and Stribling, J.B. 1999. *Rapid Bioassessment Protocols for Use in Streams and Wadeable Rivers: Periphyton, Benthic Macroinvertebrates and Fish*, Second Edition. EPA 841-B-99-002. Washington, D.C.: U.S. Environmental Protection Agency, Office of Water.

Buchwalter, D.B., Cain, DJ, Clements, WH, and Luoma, SN. 2007. Using biodynamic models to reconcile differences between laboratory toxicity tests and field biomonitoring with aquatic insects. *Environmental Science and Technology* 41:4821-4828.

Burton, G.A. 1992. Assessing the toxicity of fresh-water sediments. *Environmental Toxicology and Chemistry* 10:1585-1627.

Cain, D.J, Luoma, S.N, and Wallace, W.G. 2004. Linking metal bioaccumulation of aquatic insects to their distribution patterns in a mining-impacted river. *Environmental Toxicology and Chemistry* 23:1463-7143.

Clark, J.L., and Clements, W.H. 2006. The use of in situ and stream microcosm experiments to assess population- and community-level responses to metals. *Environmental Toxicology and Chemistry* 25:2306-2312.

Clements, W.H., Cherry, D.S., and Cairns, J. 1988. Structural alterations in aquatic insect communities exposed to copper in laboratory streams. *Environmental Toxicology and Chemistry* 7:715-722.

Clements, W.H., and Kiffney, P.M. 1994. Integrated laboratory and field approach for assessing impacts of heavy metals at the Arkansas River, Colorado. *Environmental Toxicology and Chemistry* 13:397-404.

Cuffney, T.F., Gurtz, M.E., and Meador, M.R. 1993. *Methods for Collecting Benthic Macroinvertebrate Samples as Part of the National Water-Quality Assessment Program*. Open-File Report 93-406. Reston, VA: U.S. Geological Survey.

Farag, A.M., Woodward, D.F., Golstein, J.N., Brumbaugh, W., and Meyer, J.S. 1998. Concentrations of metal associated with mining waste in sediments, biofilm, benthic macroinvertebrates, and fish from the Coeur d'Alene river basin, Idaho. *Archives of Environmental Contamination and Toxicology* 34:119-127.

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Farag, A.M., Nimick, D.A., Kimball, B.A., Church, S.E., Skaar, D., Brumbaugh, W.G., Hogstrand, C., and MacConnell, E. 2004. Aquatic health and exposure pathways of trace elements. In *Integrated Investigations of Environmental Effects of Historical Mining in the Basin and Boulder Mining Districts, Boulder River Watershed, Jefferson County, Montana*. Professional Paper 1652-A. Reston, VA: U.S. Geological Survey.

Hare, L. 1992. Aquatic insects and trace-metals- Bioavailability, bioaccumulation and toxicity. *Critical Reviews in Toxicology* 22(5-6):327-369.

Hawkins, C.P., Norris, R.H., Hogue, J.N., and Feminella, J.W. 2000. Development and evaluation of predictive models for measuring the biological integrity of streams. *Ecological Applications* 10(5):1456-1477.

Kennedy, A.J., Cherry, D.S., and Currie, R.J. 2004. Evaluation of ecologically relevant bioassays for a lotic system impacted by a coal-mine effluent, using Isonychia. *Environmental Monitoring and Assessment* 95(1-3):37-55.

Long, E.R., and Chapman, P.M. 1985. A sediment quality triad- Measures of sediment contamination, toxicity, and infaunal community composition in Puget-Sound. *Marine Pollution Bulletin* 16(10):405-415.

Luoma, S.N., and Rainbow, P.S. 2005. Why is metal bioaccumulation so variable? Biodynamics as a unifying concept. *Environmental Science and Technology* 39(7):1921-1931.

Moulton, S.R., Kennen, J.G., Goldstein, R.M., and Hambrook, J.A. 2002. *Revised Protocols for Sampling Algal, Invertebrate, and Fish Communities as Part of the National Water-Quality Assessment Program*. Open-File Report 02-150. Reston, VA: U.S. Geological Survey.

Rainbow, P. 2002. Trace metal concentrations in aquatic invertebrates: why and so what? *Environmental Pollution* 120(3):497-507.

Roesijadi, G. 1992. Metallothioneins in metal regulation and toxicity in aquatic animals. *Aquatic Toxicology* 22(2):81-114.

Schmidt, T.S., Clements, W.H, Mitchell, K.A., Church, S.E., Wanty, R.B., Fey, D.L., Verplanck, P.L., and San Juan, C.A. 2010. Development of a new toxic-unit model for the bioassessment of metals in streams. *Environmental Toxicology and Chemistry* 29(11):2432-2442.

Schmidt, T.S., Soucek, D.J., and Cherry, D.S. 2002. Integrative assessment of benthic macroinvertebrate community impairment from metal-contaminated waters in tributaries of the Upper Powell River, Virginia, USA. *Environmental Toxicology and Chemistry* 21(10):2233-2241.

Schmidt, T.S., Clements, W.H, and Cade, B.S. 2012. Estimating risks to aquatic life using quantile regression. *Freshwater Science* 31(3):709-723.

Underwood, A.J. 1994. On beyond BACI: Sampling designs that might reliably detect environmental disturbances. *Ecological Applications* 4(1):3-15.

USEPA (U.S. Environmental Protection Agency). 2002a. *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms*, Fifth Edition. Office of Water, EPA-821-R-02-012.

USEPA (U.S. Environmental Protection Agency). 2002b, *Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms*, Fourth Edition. Office of Water, EPA-821-R-02-013.

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Biotic Ligand Model

(Kathleen S. Smith)

MEDIUM: Water

PRINCIPLE: The biotic ligand model (BLM) is a computational model that determines metal speciation and predicts acute metal toxicity to biota in aqueous systems (DiToro et al., 2001; Santore et al., 2001). The U.S. Environmental Protection Agency (EPA) incorporated the BLM into its recently revised aquatic life ambient freshwater quality criteria for Cu (EPA, 2007a), replacing the previous hardness-based criteria for Cu. Currently (*circa* 2012), Cu is the only metal that has a BLM incorporated into its USEPA aquatic life criteria; BLMs for Ag, Cd, Pb, Ni, and Zn are under development (Paquin et al., 2002). The BLM is being considered for regulatory and risk-assessment frameworks worldwide. There are a variety of non-regulatory BLMs under development for a variety of applications. This discussion is limited to the regulatory version of the BLM.

The free ion activity model (FIAM), described by Morel (1983), demonstrates that the free metal ion activity reflects the overall reactivity of the metal. This reactivity is a major control on metal bioavailability and toxicity and is the conceptual basis for the BLM. The BLM combines (1) calculations of thermodynamic speciation to determine the free and complexed (inorganic and organic) forms of dissolved aqueous species, (2) calculations of metal uptake by biological receptors, and (3) computation of water-quality criteria based upon the speciation and uptake calculations. The BLM combines an equilibrium geochemical model, a metal-organic binding model, and a physiological/toxicological model. Required input parameters for the BLM include temperature, pH, dissolved organic carbon (DOC), percent DOC as humic acid, dissolved major cations and anions (i.e., Ca, Mg, Na, K, SO_4, and Cl), and alkalinity. Dissolved concentration of the metal of interest is required if the BLM is run in speciation mode. The BLM represents an improvement over previous hardness-based criteria in that it produces criteria for site-specific conditions, it is an alternative to expensive toxicological testing, it links chemical speciation with bioavailability/toxicity, and it is relatively easy to use.

APPLICATION: Two versions of the BLM are available. The research BLM version can be downloaded from the HydroQual, Inc. website (www.hydroqual.com/wr_blm.html; accessed 8/23/13) and has the capability to output aqueous speciation data, water-quality criteria computations, and LC_{50} values, which is the metal concentration that is lethal to 50% of a group of test organisms within a given timeframe. The water-quality criteria computations include final acute values (FAV), criterion continuous concentrations (CCC), criterion maximum concentrations (CMC), and acute toxic units (TU)] for Cu. In addition to Cu speciation and LC_{50} values, this research version of the BLM predicts acute toxicity and speciation of Ag, Cd, and Zn for a variety of aquatic organisms including fathead minnow, rainbow trout, *Daphnia magna*, *Daphnia pulex*, and *Ceriodaphnia dubia*. A second BLM version is available from the USEPA (http://water.epa.gov/scitech/swguidance/standards/criteria/aqlife/pollutants/copper/2007_index.cfm: accessed 8/23/13) that computes water-quality criteria for Cu.

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LIMITATIONS: The complete set of BLM input parameters has not historically been measured at most sites and estimation techniques are not well established. DOC is especially important for the Cu-BLM and is not a parameter commonly collected for site characterization. The BLM does not consider all known influences on metal bioavailability and toxicity. For example, computations can only be made for one metal and one organism at a time, so the influences of multiple metal toxicity, competition among metals, and competition among organisms are not addressed. Also, the influence of dissolved Fe and Al on metal binding to DOC (which can be an important factor in mined and mineralized areas, Smith et al., 2009) is not taken into account. The BLM considers only dissolved equilibria, so dietary metal toxicity, metal/solid-phase interactions (e.g., mineral precipitation, sorption/desorption reactions), and non-equilibrium conditions (e.g., kinetic reactions) are not considered. Finally, although the BLM can compute criteria for site-specific conditions, it is still necessary to consider the influence of spatial and temporal variability.

BIBLIOGRAPHY:

Di Toro, D.M., Allen, H.E., Bergman, H.L., Meyer, J.S., Paquin, P.R., and Santore, R.C. 2001. A biotic ligand model of the acute toxicity of metals. I. Technical basis. *Environ. Sci. Technol.* 20:2382–2396.

HydroQual, Inc. 2007. *Biotic Ligand Model, Windows Interface, Version 2.2.3, User's Guide and Reference Manual*. Mahwah, NJ: HydroQual, Inc. www.hydroqual.com/wr_blm.html. Accessed 8/23/13.

Morel, F.M.M. 1983. *Principles of Aquatic Chemistry*. New York: John Wiley & Sons, 446 p.

Niyogi, S., and Wood, C. M. 2004. Biotic ligand model, a flexible tool for developing site-specific water quality guidelines for metals. *Environ. Sci. Technol.* 38:6177–6192.

Pagenkopf, G. K. 1983. Gill surface interaction model for trace-metal toxicity to fishes: role of complexation, pH, and water hardness. *Environ. Sci. Technol*. 17:342–347.

Paquin, P.R., Gorsuch, J.W., Apte, S., Bately, G.E., Bowles, K.C., Campbell, P.G.C., Delos, C.G., Di Toro, D.M., Dwyer, R.L., Galvez, F., Gensemer, R.W., Goss, G.G., Hogstrand, C., Janssen, C.R., McGeer, J.C., Naddy, R.B., Playle, R.C., Santore, R.C., Schneider, U., Stubblefield, W.A., Wood, C.M., and Wu, K.B. 2002. The biotic ligand model: a historical overview. *Comp. Biochem. Physiol. Part C*. 133:3–35.

Santore, R.C., Di Toro, D.M., Paquin, P.R., Allen, H.E., and Meyer, J.S. 2001. A biotic ligand model of the acute toxicity of metals. II. Application to acute copper toxicity in freshwater fish and Daphnia. *Environ. Sci. Technol*. 20:2397–2402.

Slaveykova, V. I., and Wilkinson, K. J. 2005. Predicting the bioavailability of metals and metal complexes: Critical review of the biotic ligand model. *Environ. Chem*. 2:9–24.

Smith, K.S., Ranville, J.F., Diedrich, D.J., McKnight, D.M., and Sofield, R.M. 2009. Consideration of iron-organic matter interactions when predicting aquatic toxicity of copper in mineralized areas. In *Proceedings, Securing the Future and 8th*

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International Conference on Acid Rock Drainage (ICARD). Sweden: SveMin. http://www.proceedings-stfandicard-2009.com/. Accessed 8/23/13.

USEPA (U.S. Environmental Protection Agency). 2007a. *Aquatic Life Ambient Freshwater Quality Criteria—Copper: 2007 Revision*. Washington, D.C.: U.S. Environmental Protection Agency, EPA-822-R-07-001. www.epa.gov/waterscience/criteria/copper/2007/index.htm. Accessed 8/23/13.

USEPA (U.S. Environmental Protection Agency). 2007b. *Framework for Metals Risk Assessment*. Washington, D.C.: U.S. Environmental Protection Agency, EPA/120/R-07/001. www.epa.gov/raf/metalsframework/pdfs/metals-risk-assessment-final.pdf. Accessed 8/23/13.

Fish and Fish Habitat

(Andrew S. Todd)

MEDIA: Biological

MEASUREMENT OBJECTIVES: Measurements of resident fish populations and their environment can be used to evaluate the impact of mining-related aquatic stressors, such as acid rock drainage, on the aquatic ecosystems downstream of that mining influence. Sampling objectives could be to answer any of the following types of questions:

- What fish would we expect to be present at a given site?
- Are fish currently present at a given site resident at the expected temporal frequency (year-round or seasonally)?
- How healthy is the fishery at a given site?
- Is poor water quality resulting from ARD causing observed impacts to the fishery?
- What other factors (e.g., lack of suitable habitat) could be causal for an impaired fishery at a given site?

SPECIFIC FIELD SAMPLING TECHNIQUES/EQUIPMENT: A variety of sampling techniques/approaches are available to evaluate fisheries and the factors that influence their well-being.

Fish capture in the field—several methods are available to capture fish in the field for characterization of organism metrics (e.g., length vs. weight) and collection of samples for later laboratory analysis.

a) **Hook and line**- qualitative (presence/absence of fish), lentic (standing or still water) and lotic (moving water) habitats

b) **Seines and gill nets**- qualitative/quantitative, primarily lentic habitats (Hubert, 1996; Hayes et al., 1996)

c) **Electrofishing**- lentic and lotic habitats (Reynolds, 1996; USEPA, 1993)

i. Qualitative (e.g., spot-shocking), yields presence/absence information

ii. Quantitative (e.g., multiple-pass depletion methods), can yield population, biomass, species composition estimates

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Collection of data in the field—a variety of information can be obtained through measurements obtained in the field and through proper preservation of fish tissues.

a) **Habitat assessment**- several protocols exist to evaluate habitat quality for fish (Barbour et al., 1999; McMahon et al., 1996)
b) **Fish "fitness"**- length and weight measurements useful for evaluating the health/condition of individual fish (Barton et al., 2002; Anderson and Neumann, 1996)
c) **Fish age and growth**- collection of non-lethal (scales) and lethal (otolith) samples can yield estimates of the age of resident fish (DeVries and Frie, 1996; USEPA, 1993)
d) **Collection of tissue samples**- proper specimen processing and preservation is essential for later lab analysis (USEPA, 1993)
e) **Marking fish**- release of "marked" or tagged fish after capture can be helpful in tracking their survival/behavior (Guy et al., 1996)

SPATIAL OR TEMPORAL CONSIDERATIONS: The fishery present at a given location is a function of the biological, physical and chemical conditions that exist at that site, which all may vary over time. Several experimental approaches exist to evaluate a fish population's response to a stressor such as ARD (Power, 2002).

- Compare and contrast- comparison with reference site, upstream/downstream of ARD input
- Graded exposure/response- measure fish community over a range exposure severities
- Sequential sampling- evaluate temporal variability in fish community
- In-situ fish exposure- directly observe fish response to stressors (Todd et al., 2007)

LABORATORY TECHNIQUES: A field evaluation of fisheries is an important component to understanding the fishery impacts of stressors such as ARD, analyses completed in the laboratory can add significantly to that understanding.

Toxicity testing- exposing fish to contaminants in a controlled laboratory setting can yield valuable information about factors limiting a fishery in the field (Sprague, 1990).

a) **"Whole effluent" testing**- exposure of fish to waters (and a complete dilution series) collected from the field site of interest (USEPA, 2002a)
b) **Reference testing**- exposure of fish to individual contaminants of interest to isolate the effect of the contaminant of concern (USEPA, 2002a)
c) **Acute vs. chronic-** different methods and endpoints for evaluating the toxicity of short-term (USEPA, 2002a) and prolonged exposures (USEPA, 2002b)

Tissue analysis- Laboratory analyses of tissues collected in the field can produce lines of evidence linking contaminant exposure to biochemical effects

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a) **Tissue metals concentrations**- Concentrations of metals in tissues such as gill, liver, kidney, and fillet can provide evidence of metal exposure (USEPA, 1993; Todd et al., 2007; Todd et al., 2009)
b) **Metal-specific biochemical responses**- Measurement of parameters such metallothionein and lipid peroxidation can document physiological malfunction (Farag et al., 2004)
c) **Histology**- documentation of increased incidence of morphological changes (gill erosion, fin rot) can be used to link contaminants to observed degradation of a fishery (Myers and Fournie, 2002)

ADDITIONAL CONSIDERATIONS: The observed condition of a fishery at any given time represents an integrated response to the range of physical, chemical, and biological conditions present and previously-present at the site. The tools described above can be used as complementary lines of evidence in a comprehensive assessment of factors limiting the resident ecosystem.

BIBLIOGRAPHY:

Anderson, R.O., and Neumann, R.M. 1996. Length, weight, and associated structural indices. *In* B.R. Murphy and D.W. Willis, editors. *Fisheries Techniques*, 2nd edition. Bethesda, MD: American Fisheries Society.

Barbour, M.T., Gerritsen, J., Snyder, B.D., and Stribling, J.B. 1999. *Rapid Bioassessment Protocols for Use in Streams and Wadeable Rivers: Periphyton, Benthic Macroinvertebrates and Fish*, 2nd edition. EPA 841-B-99-002, Office of Water. Washington, D.C.: U.S. Environmental Protection Agency.

Barton, B.A., Morgan, J.D., and Vijayan, M.M. 2002. Physiological and condition-related indicators of environmental stress in fish. *In* S.M. Adams, editor. *Biological Indicators of Aquatic Ecosystem Stress*. Bethesda, MD: American Fisheries Society.

DeVries, D.R., and Frie, R.V. 1996. Determination of age and growth. *In* B.R. Murphy and D.W. Willis, editors. *Fisheries Techniques*, 2nd edition. Bethesda, MD: American Fisheries Society.

Farag, A.M., Nimick, D.A., Kimball, B.A., Church, S.E., Skaar, D., Brumbaugh, W.G., Hogstrand, C., and MacConnell, E. 2004. Aquatic health and exposure pathways of trace elements. *In Integrated Investigations of Environmental Effects of Historical Mining in the Basin and Boulder Mining Districts, Boulder River Watershed, Jefferson County, Montana*. Professional Paper 1652-A. Reston, VA: U.S. Geological Survey.

Guy, C.S., Blankenship, H.L., and L.A. Nielsen. 1996. Tagging and marking. *In* B.R. Murphy and D.W. Willis, editors. *Fisheries Techniques*, 2nd edition. Bethesda, MD: American Fisheries Society.

Hayes, D.B., Ferreri, C.P., and Taylor, W.W. 1996. Active fish capture methods. *In* B.R. Murphy and D.W. Willis, editors. *Fisheries Techniques*, 2nd edition. Bethesda, MD: American Fisheries Society.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Hubert, W.A. 1996. Passive capture techniques. *In* B.R. Murphy and D.W. Willis, editors. *Fisheries Techniques*, 2nd edition. Bethesda, MD: American Fisheries Society.

McMahon, T.E., Zale, A.V., and Orth, D.J. 1996. Aquatic habitat measurements. *In* B.R. Murphy and D.W. Willis, editors. *Fisheries Techniques*, 2nd edition. Bethesda, MD: American Fisheries Society.

Myers, M.S., and Fournie, J.W. 2002. Histopathological biomarkers as integrators of anthropogenic and environmental stressors. *In* S.M. Adams, editor. *Biological Indicators of Aquatic Ecosystem Stress*. Bethesda, MD: American Fisheries Society.

Power, M. 2002. Assessing fish population response to stress. *In* S.M. Adams, editor. *Biological Indicators of Aquatic Ecosystem Stress*. Bethesda, MD: American Fisheries Society.

Reynolds, J.B. 1996. Electrofishing. *In* B.R. Murphy and D.W. Willis, editors. *Fisheries Techniques*, 2nd edition. Bethesda, MD: American Fisheries Society.

Sprague, J.B. 1990. Aquatic toxicology. *In* C.B. Schreck and P.B. Moyle, editors. *Methods for Fish Biology*. Bethesda, MD: American Fisheries Society.

Todd, A.S., Brinkman S., Wolf, R.E., Lamothe, P.J., Smith K.S., and Ranville, J.F. 2009. Use of an enriched stable-isotope approach to determine the gill-zinc binding properties of juvenile rainbow trout (*O. mykiss*) during acute zinc exposures in hard and soft waters. *Environmental Toxicology and Chemistry* 28(6):1233-1243.

Todd, A.S., McKnight, D.M., Jaros, C.L., and Marchitto, T.M. 2007. Effects of acid rock drainage on stocked rainbow trout (*O. mykiss*): an in-situ, caged fish experiment. *Environmental Monitoring and Assessment* 130:111-127.

USEPA (U.S. Environmental Protection Agency). 1993. *Fish Field and Laboratory Methods for Evaluating the Biological Integrity of Surface Waters*. Office of Research and Development, EPA-600-R-92-111. Washington, D.C.: U.S. Environmental Protection Agency.

USEPA (U.S. Environmental Protection Agency). 2002a. *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms*—Fifth Edition. Office of Water, EPA-821-R-02-012. Washington, D.C.: U.S. Environmental Protection Agency.

USEPA (U.S. Environmental Protection Agency). 2002b. *Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms*—Fourth Edition. Office of Water, EPA-821-R-02-013. Washington, D.C.: U.S. Environmental Protection Agency.

In Vitro Bioaccessibility Tests (IVBA)

(Suzette A. Morman)

MEDIUM: Solids (dust, soil, mine waste and tailings, and plant materials)

PRINCIPLE: *In vitro* bioaccessibility tests (IVBA) or physiologically based extraction tests (PBET). Studies to determine toxicity are generally conducted using animal surrogates or cell line tests which are time consuming and expensive to complete. IVBA are generally defined as the fraction of a potential toxicant that becomes soluble in the stomach and is then available for absorption (Ruby et al., 1993). Most IVBA studies

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model an ingestion exposure pathway using a simulated gastric fluid (SGF). This method is based on the concept that the amount of metal(loid)s soluble in the leachate is likely to be an important determinant of the *in vivo* bioavailability or uptake to target organs in the body. Some SGF IVBA tests endeavor to model ingestion throughout the gastrointestinal tract using sequential extractions (Oomen et al., 2002), whereas others simulate only the low pH conditions of the stomach (Drexler and Brattin, 2007).

Limited research has been conducted to determine bioaccessibility from other exposure pathways, such as inhalation by using a simulated lung fluid (SLF) (Gray et al., 2010) and for other materials, e.g., vegetation (Brumbaugh et al., 2011). These methods have not been approved for use by regulatory agencies. Other recent research interests include their application in evaluating exposure to birds(Nordwick, 2008)and other mammals (Suedel, 2006) as there are similarities in gastric properties (e.g., pH).

PRIMARY USES: IVBA may be used to estimate bioaccessibility of metal(loids) in a variety of materials. The extraction will produce a concentration, but in order to obtain a bioaccessibility value (percent leached from sample material), the total elemental concentration must be measured and the bioaccessible fraction calculated (see equation below). The results express the amount of an element that is available to the body for uptake by target organs such as the kidneys and liver. This is a very simplified process as many physiological factors control the uptake of elements by the body.

These tests were originally developed to determine the bioaccessibility of lead (Pb) in mine waste in the early 90's (Ruby et al., 1993) and their use has been primarily explored at the university level in the United States not only for Pb, but a variety of other metal(loids) as well. These tests may be available commercially in the European Union but may not yet be commonly available through commercial laboratories in the U.S.

LIMITATIONS: Although this method has been used to examine the bioaccessibility of numerous metal(loid)s (arsenic, cadmium, chromium, copper, mercury, nickel, antimony, uranium and zinc) in a variety of settings (mines, smelter impacted, other contaminated sites and urban areas) from a regulatory perspective, the USEPA has accepted only one method (Drexler and Brattin, 2007) to determine lead bioaccessibility for site specific assessments (USEPA, 2008). Relative bioavailability of Pb may be calculated from the bioaccessibility results.

In terms of physiology, these tests produce estimates of what is available to the body for uptake(bioaccessibility), not actual uptake (bioavailability) to the target organs such as liver or kidneys. In the United States, there are currently no guidelines, either health or regulatory, for comparison of bioaccessibility values.

INTERFERENCES: As several 'recipes' are available only general guidelines are described. Factors influencing analytical error include sample heterogeneity, solid: solution ratio and leaching parameters.

SCOPE: IVBA have been used to estimate bioaccessibility of metal(loids) in a variety of materials.

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DETECTION LIMITS: Detection limits are specific to the instrument used for analysis of the extract, either ICP-MS or ICP-AES.

DATA FORMAT: The data are generally presented as the percentage of the total concentration of the element that is soluble (bioaccessible) defined as:

$$\% \text{ Bioaccessibility} = \frac{\text{(mg leached by extraction fluid per kg soil)}}{\text{(total concentration in soil, mg/kg)}} \times 100$$

The data may also be presented as mg leached per kg solid.

COST: Costs are considered lower when compared to animal surrogate studies. It is uncertain at this time how many commercial laboratories have this method available and what the exact costs are.

BIBLIOGRAPHY:

Brumbaugh, W.G., Morman, S.A., and May, T.W. 2011. Concentrations and bioaccessibility of metals in vegetation and dust near a mining haul road, Cape Krusenstern National Monument, Alaska. *Environmental Monitoring and Assessment* 182:325-340.

Drexler, J.W., and Brattin, W.J. 2007. An *in vitro* procedure for estimation of lead relative bioavailability: with validation. *Human Ecology Risk Assessment* 13:383-401. See also http://www.colorado.edu/geolsci/legs/invitro1.html. Accessed 8/23/13.

Gray, J.E., Plumlee, G.S., Morman, S.A., Higueras, P.L., Crock, J.G., Lowers, H.L., and Witten, M.L. 2010. In vitro studies evaluating leaching of mercury from mine waste calcine using simulated human body fluids. *Environ. Sci. Technol.* 44:4782–4788.

Oomen, A.G., Hack, A., Minekus, M., Zeijdner, E., Cornelis, C., Schoeters, G., Verstraete, W., Van DeWiele, T., Wragg, J., Rompelberg, C.J.M., Sips, A.J.A.M., and Van Wijnen, J.H., 2002, Comparison of five in vitro digestion models to study the bioaccessibility of soil contaminants. *Environ. Sci. Technol*. 36:3326-3334.

Ruby, M.V., Davis, A., Link, T., Schoof, R., Chaney, R., Freman, G., and Bergstrom, P. 1993. Development of an *in vitro* screening test to evaluate the *in vivo* bioaccessibility of ingested mine-waste lead. *Environ. Sci. Technol*. 27:2870-2875.

Suedel, B.C., Nicholson, A., Day, C.H., and Spicer, J., II. 2006. The value of metals bioavailability and speciation information for ecological risk assessment in arid soil. *Integrated Environmental Assessment and Management* 2:355-364.

USEPA (U.S. Environmental Protection Agency). 2008. *Linking Waterfowl with Contaminant Speciation in Riparian Soils*. Mine Waste Technology Program. EPA/600/R-08/060. http://www.epa.gov/nrmrl/pubs/600r08060.html. Accessed 8/23/13.

USEPA (U.S. Environmental Protection Agency). 2008. Standard Operating Procedure for an *In Vitro* Bioaccessibility Assay for Lead in Soil. EPA 9200.1-86.

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http://www.epa.gov/superfund/health/contaminants/bioavailability/pb_ivba_sop_final.pdf. Accessed 8/23/13.

Microorganisms as Indicators for Metal(loid) Impacted Environments

(Timberley M. Roane and Raina M. Maier)

MEDIA: Soil and Water

MEASUREMENT OBJECTIVES: The measurement of certain populations of microorganisms can indicate the level of stress within an environment. Microorganisms which are too small to be seen with the naked eye, at least as single cells, include algae, bacteria, fungi, protozoa and viruses. Common properties of microorganisms are that they are ubiquitous in the environment, and are capable of surviving environmental conditions, such as high metal concentrations, that exclude other organisms. Despite their presence in contaminated environments, relatively little is understood about the types of microorganisms associated with metal- and metalloid-impacted systems. As such, there are no established microbiological standards or guidelines related to metal(loid) toxicity, and yet microorganisms, in particular the bacteria, can be used as indicators of toxicity and remediation potential. Bacteria can resist metal(loid) toxicity through the production of extracellular compounds that complex the metal(loid), reducing its bioavailability for plants and animals; through the production of intracellular compounds that also bind and reduce metal(loid) toxicity; and through the conversion of various forms of metal(loid)s into solids (e.g., elemental metal(loid) forms), gaseous (e.g., volatilized metal(loid) forms), or altered oxidation states (in some cases oxidized, in some cases reduced) that also reduce toxicity (Maier et al., 2009). As a result, the presence of certain types of metal(loid)-resistant bacteria can be used as an indicator of metal(loid) stress in a system. When compared to unimpacted equivalent environments, assessing the types or diversity of microorganisms present in a metal(loid)-impacted environment is the first step in determining the effect metal(loid)s are having on other biological systems. Several methods are available to do this, including culture-based and molecular-based techniques.

It is important to understand that microorganisms exist as complex communities in the environment with each community having a diverse mix of individual microbial populations. The presence of organic matter and nutrients, as well as the dominant environmental characteristics of the environment (e.g. pH, temperature, oxygen availability) provide selective pressures that determine the community present at any given site. For example, in a normal forest soil which is slightly acidic and has normal levels of organic matter and nutrients (including background levels of metal(loid)s), it is expected that the community will be dominated by chemoheterotrophic bacterial and fungal populations that make a living via the oxidation of the organic matter present. These microorganisms are the cornerstone for normal carbon cycling activities in soil and water systems. However, in metal(loid)-contaminated environments, particularly those associated with mining, levels of organic carbon and nutrients are often low to nonexistent whereas levels of metal(loid)s are higher than normal. This will result in a community dominated by populations of chemolithoautotrophic microorganisms that make a living through the oxidation of reduced iron and sulfur compounds combined with the fixation of carbon dioxide into organic matter used to build cells. Thus, it is

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possible to determine the level of environmental and metal(loid) stress in an environment by examining the dominant populations present in a soil or water community to determine whether: 1) populations with metal(loid) resistance mechanisms are common; and 2) whether chemoheterotrophic or chemolithoautotrophic populations dominate the system.

ANALYTICAL TECHNIQUES:

Culture-based methods: Culture-based methods use specialized growth media to isolate and quantify particular populations of microorganisms within the community (Maier et al., 2009). For metal(loid)-impacted samples, it can be useful to:

- Enumerate chemoheterotrophic (carbon cycling) bacterial populations to indicate the level of stress in the environment. For example, a normal soil chemoheterotrophic count will range from 10^5 to 10^7 bacteria per gram soil. In mine tailings or waste rock piles this number normally ranges from 10^1 to 10^5 depending on pH and nutrient content. The lower the number of chemoheterotrophs present, the higher the stress level in the site. High stress levels and low chemoheterotrophic counts are associated with difficulty in establishing a vegetative cap on the site to bring it to closure (Mendez et al., 2007).
- Enumerate chemolithoautotrophic populations such as iron or sulfur-oxidizers. The presence of *Acidothiobacillus ferrooxidans*, for example, is linked to acidic conditions and solubilization of metal(loid)s in mine waste or water samples (Mendez et al., 2007; Campos et al., 2011). In a normal soil iron- and sulfur-oxidizers are not detectable using culturable counts. However, iron- and sulfur-oxidizer counts in mine tailings, waste rock piles and acid mine drainage can reach up to 10^6 per gram (Kock and Schippers, 2006). The higher the number of chemolithoautotrophic bacteria the higher the stress level associated with the site.
- Isolate metal(loid)-resistant bacteria (those bacteria capable of growth when high levels of metal(loid)s are present). Such bacteria can be used to aid in remediation of mine waste or water samples through precipitation of metal(loid)s and reduction of sulfate associated with acid mine drainage (Hallberg, 2010; Kassab and Roane, 2006).

Molecular-based methods: Although useful, culturing is limited by the complex growth media many microorganisms require. In recent years, analysis of nucleic acids (DNA) in environmental samples has become a common way to evaluate the bacterial populations within microbial communities (Maier et al., 2009). Most of these approaches are based on analysis of a single gene, the 16S ribosomal RNA gene (16S rRNA gene), which is currently used to classify bacteria. To accomplish this several approaches have been developed:

- Fingerprint or profiling approaches rapidly provide a snapshot of community structure but do not identify the member populations in the community. Fingerprinting approaches are good for comparing differences between a large number of samples. Approaches used include denaturing gradient gel electrophoresis (DGGE) or terminal restriction length polymorphism (T-RFLP) profiling of the 16S rRNA gene (Maier et al., 2009). Profiling techniques such as

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DGGE or T-RFLP are often used to screen a large number of samples to support the choice of a smaller number of samples that are analyzed further, using more expensive approaches, to identify populations within the community.

- There are currently three main approaches, based on the 16S rRNA gene, used to identify a bacterial community. Phylochip microarray (Affymetrix, Inc.) analysis, clone library analysis, and high-throughput sequencing analysis (Maier et al., 2009). Each of these approaches has advantages and disadvantages. For example, the Phylochip microarray contains thousands of known bacterial probes that can rapidly determine which populations are present within a sample. The disadvantage is that it cannot detect "unknown" microorganisms even if they make up a significant part of the community. In contrast, high throughput sequencing analysis rapidly determines the populations within a sample at great depth. This means that the technique in many cases does a good job at detecting most of the different types of bacteria present. The disadvantage to this technique is that only a small part of the 16S rRNA gene is analyzed which is often not enough to make an exact identification. Finally, a clone library analyzes the entire 16S rRNA gene and so identification is more definitive. The disadvantage is that the analysis takes much more time and has only a small fraction of the "depth" achieved by high-throughput sequencing (Mendez et al., 2008).

As an example, a recent study used clone library analysis to compare the bacterial communities present in extremely (pH 2.7) and moderately (pH 5.7) acidic lead-zinc mine tailing samples with those from a vegetated off-site control soil sample (pH 8) (Mendez et al., 2008)[&]. The number of unique populations identified in each of these communities decreased from 42 in the control soil to 24 in the moderately acidic sample and 8 in the extremely acidic tailing sample. The clones in the extremely acidic tailing sample were most closely related to acidophiles (known iron- and sulfur-oxidizers), none of which were detected in the control soil sample. This comparison provided a reference point to better understand the revegetation of the mine tailings (Mendez et al., 2007).

As a second example, an on-going study by Roane et al. (unpublished data)[§] using Phylochip-based bacterial community analysis has found that elevated sediment-associated metal(loid) concentrations select for a specialized group of bacteria called the Archaea. Particularly adapted to harsh environmental conditions, the Archaea are an understudied group of bacteria associated with acid mine drainage; however, they may serve as key indicators of metal(loid) toxicity. Other studies are finding promising uses for the Archaea in bioleaching and metal(loid) remediation (Bini, 2010).

ADDITIONAL CONSIDERATIONS: Among the advantages of using microorganisms as indicators of metal(loid) toxicity is the ability of microorganisms to immediately respond to changes in their environment. If metal(loid) concentration, for example, changes, the microbial community will very quickly respond to the change. If regular microbial monitoring is occurring, the resulting microbial response can be detected.

BIBLIOGRAPHY:

Bini, E. 2010. Archaeal transformation of metals in the environment. FEMS Microbiology Ecology, 73, 1-16.

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Campos, M.B., H. de Azevedo, M.R.L. Nascimento, C.V. Roque, and S. Rodgher. 2011. Environmental assessment of water from a uranium mine (Caldas, Minas Gerais State, Brazil) in a decommissioning operation. Environmental Earth Science, 62, 857-863.

Hallberg, K.B. 2010. New Perspectives in acid mine drainage. Hydrometallurgy, 104, 448-453.

Kassab, D.M., and T.M. Roane. 2006. Differential responses of a mine tailings *Pseudomonas* isolate to cadmium and lead exposures. Biodegradation, 17, 379-387.

Kock, D. and A. Schippers. 2006. Geomicrobiological investigation of two different mine waste tailings generating acid mine drainage. Hydrometallurgy, 83, 167-175.

Maier, R.M., I.L. Pepper and C.P. Gerba. 2009. Environmental Microbiology, 2nd edition. Academic Press, San Diego, CA.

Mendez, M.O., E.P. Glenn, and R.M. Maier. 2007. Phytostabilization potential of quailbush for mine tailings: growth, metal accumulation and microbial community changes. Journal of Environmental Quality, 36, 245-253.

Mendez, M.O., J.W. Neilson, and R.M. Maier. 2008. Bacterial community characterization of a historic semiarid lead-zinc mine tailings site. Applied and Environmental Microbiology, 74, 3899-3907.

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Toxicity Test Kits

(Andrea L. Foster)

PRINCIPLE: Off-the-shelf water and sediment toxicity test kits employ dormant prokaryotes or eukaryotes that are revived for measurement of chronic or acute toxic response to one or more constituents. Prior to the advent of these kits, expensive and time-consuming toxicity testing was restricted to laboratories with staff and facilities for the maintenance of continuous test cultures. In many cases, off-the-shelf kits provide a simple and cost-effective mechanism for evaluating the absolute and relative toxicities of mine-waste materials.

Many off-the-shelf kits are self-contained and require very simple equipment or facilities, and could conceivably be used in a mobile lab in the field. Organisms used in commercially-available kits include strains of *E. coli, Vibrio fischeri,* algae, protozoans, rotifers, several types of crustaceans, and a variety of higher plant seedlings. Most testing is directed towards freshwater environments, but tests using estuarine/marine invertebrate species are also available. Acute toxicity is measured by immobility (death), or lack of growth, and chronic toxicity is measured by several means including enzymatic response (*E. coli*), production of luminescence (*V. fischeri*), reproductive ability (*Daphnia*), and extent of growth (ostracods, plants) under controlled conditions.

PRIMARY USES: In the past, toxicity tests were primarily used to derive acute toxicity values such as LC_{50}, (the concentration of toxicant required to produce 50% mortality

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among the test organisms) for chemicals in isolation. This allows for important comparisons among organisms (to identify the most sensitive species) or among reagents (Connell and Miller, 1984). With the advent of off-the-shelf kits, direct measurement of the toxicity of waters/effluents and sediments (including mine wastes) can be measured at low cost and with a higher throughput. As an example of the use of these tests, the USEPA developed the whole effluent toxicity (WET) test to measure toxic effect of an effluent without requiring the identification of specific pollutants. The test is used along with other water quality criteria to set discharge limits in permits through its National Pollutant Discharge Elimination System (NPDES). USEPA recommends the use of representative vertebrate, invertebrate and plant species as ecological receptors in its WET test (http://cfpub.epa.gov/npdes/wqbasedpermitting/wet.cfm, accessed 8/23/13). Toxicity test data are also used in screening for pollutants, compliance biomonitoring, and laboratory studies (USEPA, 1993).

LIMITATIONS: Each test organism has limited life tolerance conditions (mainly pH, but other factors such as dissolved oxygen level, temperature, and sensitivity to low levels of non-tested chemicals) that must be maintained in order to have a valid result. Particularly with regard to mine wastes, the role of pH in toxicity should be assessed as well as the role of metals. Regular validation of the test method and outcome must be made using a standard reference material with known toxicity. During longer (> 24 hr) tests, toxicants may be lost via sorption on containers or biota, may volatilize, or otherwise become degraded and inactive (USEPA, 1993). The toxic effect of a chemical may also vary considerably between organisms, making comparison difficult (Kenaga, 1981). Results derived from long-term laboratory cultures may present toxic responses not aligned with that of the native community due to genetic drift; commercial labs should be queried as to the measures taken to preserve or assess the genetic diversity of cultures used for toxicity testing.

INTERFERENCES: Interference is taken broadly to mean confounding factors in execution of method or interpretation of results. Positive or negative synergistic effects in toxicant mixtures can easily be missed unless the pure end-members' toxicities are also evaluated. Also, care must be taken to evaluate the species of the toxicant under consideration. Aquatic toxicity tests are designed to measure dissolved species, and use test organisms that feed only on dissolved species (e.g., microbes) or suspended particles (*Daphnia*). Sediment (contact) toxicity tests specifically use detritus-feeders such as ostracods, but are usually conducted in aqueous slurry, which introduces ambiguity into the results unless it can be demonstrated that dissolution of the toxic phase in the aqueous mileu (outside the organism) is negligible.

SCOPE: Off-the-shelf toxicity tests have been used to measure toxicity of mining waste effluents in Alaska and Colorado, among other places (Choate et al., 2008). They have also been used to assess toxicity in other industrial settings. Microbial-based tests have been used to measure microbial response to organic and inorganic chemicals (Bitton et al., 1994; Bitton et al., 1992). LC_{50} values from a variety of related water samples can be used to identify site-specific areas of concern or suggest the mechanism of toxic effect (Choate et al., 2008; Bitton et al., 1994).

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DETECTION LIMITS: The ability to measure a toxic response in the organism and the sensitivity of the chemical analysis method set the operational detection limit, which will vary by element. Field-based, visual indicator test kits (i.e., MetPlate) are less sensitive and accurate than laboratory tests where color development in solution can be quantified by visible spectrophotometer or luminescence by a fluorometer.

MASS/VOLUME REQUIREMENTS: Depending on the concentration of toxicants, as little as 10 mL to as much as 100 mL of high-concentration test solution will be needed for the entire aquatic test (including replicates) using *Daphnia*, *E. coli*, or plant seeds. The invertebrate sediment toxicity test we have used requires a fixed amount of sediment in each test well, which could lead to difficulties at sites with dilute or low-toxicity metals. At sites with high-toxicity or high concentration metals, dilution of the sediment with cleaned sand is recommended.

SAMPLE PRESERVATION/PREPARATION: Aqueous and sediment samples to be tested should be collected into acid-washed plastic containers, stored under ambient conditions, and used as soon as possible. Where storage is required, 4º C is recommended. Sediment samples should additionally be sieved to remove larger objects and create a more homogeneous sample. Dry sieving is preferred to wet due to potential loss of labile species from sediment.

DATA FORMAT: Data are usually reported as LC_{50} (defined above) or as an observed effect among 50% of the test organisms (EC_{50}).

COST: The test costs vary, but are typically a few hundred dollars (US) for a kit that allows a single complete acute or chronic definitive test, or several scoping/survey tests. The following is a list of known vendors of off-the-shelf toxicity test kits, and is not meant to be comprehensive. Use of trade names does not constitute endorsement by the U.S. Geological Survey.

Epi-Biodetection Products (www.ebpi-kits.com/, accessed 8/23/13): daphnia, ostracod, rotifer, and plant test kits
MBL Aquaculture (www.mblaquaculture.com/content/about/index.php, accessed 8/23/13): daphnia, shrimp, fish, rotifer, and others
Sdix (www.sdix.com/ , accessed 8/23/13): Microtox test kits
Dr. Gabriel Bitton (www.ees.ufl.edu/homepp/bitton/, accessed 8/23/13): Metpad, MetPlate, MetStick (*E. coli*)

BIBLIOGRAPHY:

Bitton, G., Jung, K. and Koopman, B. 1994. Evaluation of a microplate assay specific for heavy metal toxicity. *Arch. Environ. Cont. Toxicol*. 27:25–28.

Bitton, G., Koopman, B. and Agami, O. 1992. MetPAD: A bioassay for rapid assessment of heavy metal toxicity in wastewater. *Water Environ. Res*. 64:834–836.

Choate, L.M., Ranville, J.F., Blumenstein, E.P., and Smith, K.S. 2008. Chapter E. Simple screening tests to determine the potential metal toxicity of mining-waste

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leachates. In *Understanding Contaminants Associated with Mineral Deposits*. Edited by P.L. Verplanck. U.S. Geological Survey Circular 1328. http://pubs.usgs.gov/circ/1328/. Accessed 8/23/13.

Connell, D.W. and Miller, G.J. 1984. *Chemistry and Ecotoxicology of Pollution*. New York: John Wiley and Sons.

Kenaga, E. 1981. Aquatic test organisms and methods useful for assessment of chronic toxicity of chemicals. *Regulatory Toxicol. Pharmacol*. 1:277–292.

USEPA (U.S. Environmental Protection Agency). 1993. *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms*. EPA/600/4-90/027F. Washington, D.C.

Vegetation

(James G. Crock)

MEDIUM: Vegetation, including both vascular and non-vascular species. This section does not address aquatic vegetation.

SAMPLING OBJECTIVES: There are many variables associated with sampling vegetation. Many of these variables must be defined and controlled to ensure an effective sampling design and sample collection. Of utmost importance is that the question to be answered by this sampling must be clearly and well defined before any sampling is done. Only in this way can the population that will answer the question be defined and appropriately sampled. For vegetation what part of the plant to be sampled must be defined – leaves, twigs, bark, woody parts, fruit, roots or combination - all react differently to given elements. The target elements must be defined since essential verses non-essential elements for that plant react differently in the different parts of the plant. Also whether or not to composite multiple plants in a given area or multiple plants from multiple areas must be defined. Also one must consider the prevalence of a given species across the study area. The plant (same species, genus) must be present throughout the study area for usage for complete area coverage. An additional question would be to sample a native species or an agricultural/domestic species. All these issues must be defined to answer the appropriate question. Also one must be aware of the historical usage of land, e.g., has the area been historically treated with pesticides or herbicides prior to sampling as with orchards or cotton fields.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUES: Once the part/parts of the plant and compositing variables are defined, stainless steel shears or knives are commonly used to collect the sample. These should be washed and rinsed in water or denatured alcohol. Snip and place in the collection bag, usually a plain paper or clean cloth bag. Be sure to collect sufficient material – usually more than 200 g. Up to 90% weight loss can be observed upon drying the material. Ashing the sample can also cause up to an additional 99% loss in weight. Under these extreme conditions, 100 g of sample can yield as little as 0.1 g ash. Most analytical techniques require more than this, especially for duplicate analyses if required. If roots are collected, remove as much of the soil as possible in the field. Root samples are usually washed with demineralized water in the laboratory prior to analysis. Also remove extraneous organic and inorganic material from the sample in the field. This is especially true if collecting lichens or mosses.

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SPATIAL OR TEMPORAL CONSIDERATIONS: The most important spatial question for vegetation sampling and collection is whether or not the given genus (and species) is present throughout the study area. To get full areal coverage with the same plant helps guarantee a meaningful study. Many studies focus on collection of the last year or two of growth of the plant. Collection time is a critical factor in vegetation collection, especially if the part collected is the fruit or the leaves. Plant samples should be collected during a small of a time frame as possible. Elemental content is a function of plant maturity. This is especially true if more than one harvesting is done, e.g., alfalfa.

ANALYTICAL TECHNIQUES: Most modern analytical methods are appropriate for vegetation analysis. Most common elements of interest are at very low concentrations in the dry plant material, commonly at the sub mg/Kg or µg/Kg (part per million or part per billion) levels. A goal of any survey or sampling is that the analytical results be quantifiable for as many analytes for as many of the samples as possible. This requires very sensitive methods of determination for plant analysis. Usually vegetation material is ashed under high temperatures (450^{o}C is common) to preconcentrate the elemental content commonly up to 100 fold (1% ash yield). Microwave assisted digestions of the ashed material or the raw material are a common way to digest the material prior to analysis. Multi-acid digestions at elevated temperatures are also common. For most studies multielement techniques are favored, since more analytical data are collected for a smaller sample. Inductively coupled plasma-optical emission spectroscopy (ICP-OES) and inductively coupled plasma-mass spectroscopy (ICP-MS) are the common choices. Both techniques can cover the periodic table, either simultaneously or sequentially. ICP-MS tends to have lower limits of determination but tends to be more finicky. ICP-OES is a more mature method of analysis and may prove more robust. Both methods are complementary. If these methods are not available, atomic absorption spectroscopy (AAS) is a mature, proven technique. Graphite furnace AAS offers very good limits of determination but is operator intensive. Flame AAS if very reliable and robust, but may not be sensitive enough for many of the study's analytes. Hydride generation AAS offers excellent limits of determination for an important suite of elements (e.g., As, Se, Sb). Cold vapor AAS is the preferred method of determination of Hg. Solid sampling or digested samples are also analyzed for Hg using atomic fluorescence spectrometry. The common drawback to all AAS methods is that they all are single element determinations and are more labor intensive. Whatever the method of analysis chosen, sufficient and appropriate standard reference materials, laboratory and field duplicates, and blanks must be analyzed to ensure the highest quality of data.

CONTAINER: Vegetation samples should be collected into breathable containers. Plain paper bags work well. Examples would be plain white or brown paper lunch or grocery bags. Non-coated, breathable cloth bags also will work, e.g., Hubco® style. It is important that vegetation samples be kept in breathable storage until they are sufficiently dry to deter mold or bacterial growth.

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CONTAINER PRECLEANING: New paper bags are usually sufficiently clean for plant sampling purposes. Bags should be ink-free. New glass jars as described below are also usually sufficiently clean for plant samples.

PRESERVATION: If possible, it is best to dry any vegetation samples in the field to ambient air conditions. If samples cannot be air dried in the field, they should be refrigerated to less than 4°C to prevent mold or bacterial growth. Once the material is dried and milled to the necessary size, raw material is best stored in glass jars fitted with a plastic lined metal lid. Usually the 16 to 32 oz. (pint to quart) Ball® or Mason® jars work very well. For the dried, prepared and ashed material a smaller (4 oz.) glass jar with the plastic lined lid is optimal. Simple cardboard containers may work for the short term, but since the ash is very hydrophilic, there will be sample degradation, especially in humid climates.

HANDLING AND PREPARATION: A critical question must be answered before any sample preparation is done: Should the samples be washed or not? This question is part of the answer of what the question of the study is. For example, if the question is: "What is bioavailable for cattle grazing on forbs that are growing on recovered mine lands?" one would not wash the material since the whole plant is ingested. However, if one is concerned only with the elemental content taken up by the plant through its root system, one would wash the plants to remove as much as possible of the surface contamination. Non-vascular plants, such as lichens and mosses, obtain their elemental content through areal deposition. To remove any surface contamination, mosses and lichens are commonly washed before preparation. If the samples are washed, usually gentle manual agitation in tap water followed by several rinsing with demineralized/deionized/distilled water is sufficient to remove most surface contamination. However, some particles remain in the plant as occluded material. This occluded material can sometimes be accounted for by normalizing the elemental data to elements not normally thought of being included in vegetation growth, i.e., Sc, Ce, La, or Y. Samples are dried under forced air at ambient temperature only to constant moisture content. Additional heat should not be used because of the possible loss of volatile elements. Normal sample preparation requires milling of the dried material in a suitable mill, such as a blender, hammer mill or Wiley® Mill. Samples are usually reduced in particle size to less than 10-mesh (2 mm). Samples should be stored in a controlled temperature and humidly environment. Absolute environment control is not as important if the prepared samples are stored in appropriate glass jars. Once properly prepared, plant samples should be viable for over a year.

ADDITIONAL CONSIDERATIONS: Definition and control of as many variables as possible is critical for vegetation sampling design, collection, preparation, analysis, and final interpretation of the data to be successful.

BIBLIOGRAPHY:

Barth, R.C., and Topper, K. 1993. *Sampling and Monitoring of Environmental Contaminants*. New York: College Custom Series, McGraw-Hill, Inc., 222 p.

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Crock, J.G., Severson, R.C., and Gough, L.P. 1992. Determining baselines and variability of elements in plants and soils near the Kenai National Wildlife Refuge, Alaska: *Water, Air, and Soil Pollution* 63:253–271.

Crock, J.G., Gough, L.P., Wanty, R.B., Day, W.C., Wang, B., Gamble, B.M., Henning, M., Brown, Z.A., and Meier, A.L. 2000. *Regional Geochemical Results from the Analyses of Rock, Water, Soil, Stream Sediment, and Vegetation Samples—Fortymile River Watershed, East-Central, Alaska, 1998 Sampling.* Open File Report 00-511. Reston, VA: U.S. Geological Survey, 157 p.

Gough, L.P., Day, W.C., Crock, J.G., Gamble, B.M., Henning, M.W., Ager, C.M., Meier, A.L., Briggs, P.H. Brown, Z.A., and Adams, M. 2005. *Regional Geochemical Results from the Analyses of Rock, Soil, and Vegetation Samples—Big Delta B-2 Quadrangle, Alaska*. Open-File Report 2005-1431. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/of/2005/1431/. Accessed 8/23/13.

Kabata-Pendias, A., and Pendias, H. 1984. *Trace Elements in Soils and Plants*. Boca Raton, FL: CRC Press, Inc., 315 p.

Keith, L.H. (ed.). 1988. *Principles of Environmental Sampling*. Washington D.C.: ACS Professional Reference Book, American Chemical Society, 458 p.

Peacock, T.R., and Crock, J.G. 2002. Chapter B: Plant material preparation and determination of weight percent ash. In *Analytical Methods for Chemical Analysis of Geological and Other Materials*. Edited by J.E. Taggart. Open-File Report 02-0223. Reston, VA: U.S. Geological Survey. http://pubs.usgs.gov/of/2002/ofr-02-0223/. Accessed 8/23/13.

Reuter, D.J., Robinson, J.B., and Dutkiewicz, C. 1997. *Plant Analysis—An Interpretation Manual*. Collingwood, Australia: CSIRO Publishing, 572 p.

Whole Effluent Toxicity (WET)

(Barbara Butler and Lareina Guenzel)

MEDIUM: Water and Biological Organisms

OBJECTIVES: Whole Effluent Toxicity (WET) is a term used to describe the aggregate toxic effect of an aqueous sample (e.g., whole effluent wastewater discharge) as measured by an organism's response upon exposure to the sample (e.g., lethality, impaired growth or reproduction). WET tests replicate the total effect and actual environmental exposure of aquatic life to toxic pollutants in an effluent without specificity to individual contaminants. WET tests are an important permitting requirement for evaluating a water sample relative to a State's narrative water quality standards, which prohibit the discharge of pollutants in toxic amounts or pollutants that will cause harm to the aquatic life use designation. Narrative toxicity criteria are cited as a basis for establishing WET controls in USEPA permitting regulation at 40 CFR 122.44(d)(1)(v); however, the narrative criteria that apply to specific water bodies vary by State. There are two basic types of WET tests: an acute test and a chronic test. USEPA has developed WET test protocols using both freshwater and marine and estuarine test species. It is recommended to run tests using an invertebrate, vertebrate and a plant to identify the most sensitive species. Organisms most often used in WET tests are

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indicators or surrogates for the aquatic community to be protected, and a measure of the real biological impact from exposure to the toxic pollutants.

SPECIFIC EQUIPMENT/TECHNIQUES Aquatic toxicity tests are laboratory experiments that measure acute or chronic biological effects (e.g., growth, survival, and reproduction) of a water sample (effluents or receiving waters) on aquatic organisms. In aquatic toxicity tests, organisms of a particular species and age are held in test chambers and exposed to different concentrations of an aqueous sample. Observations are then made at predetermined exposure periods. At the end of the test, the responses of test organisms are used to estimate the effects of the test sample, either in absolute terms or relative terms (i.e., the effects of an effluent in receiving waters). Because water samples may contain a mixture of many toxic compounds, these methods are designed to investigate the total toxicity of the water rather than the toxicity of a single compound contained within. These methods (USEPA, 2000; USEPA, 2002a, b) are used to identify effluents and ambient receiving waters containing toxic materials that are present in concentrations that result in acute or chronic toxicity.

To carry out these methods, water samples are brought to the laboratory and a dilution series is prepared by mixing it with control laboratory water. In some situations, it may be appropriate to use the receiving water for the dilution water, if the receiving water meets specific criteria (USEPA, 2000). For an effluent WET test, five sample dilutions plus a control are recommended. If a National Pollutant Discharge Elimination System (NPDES) permit has a limit for whole effluent toxicity, it is prudent for the dilution series to bracket that limit (USEPA, 2002a, b). Receiving water (i.e., ambient water) tests usually contain the undiluted water sample plus a laboratory water control sample, but may consist of a series of dilutions of the receiving water (USEPA, 2002a, b). USEPA has published test methods for various standard test organisms in 40 CFR § 136 (Table A3.15). These organisms have been chosen to represent field organisms (e.g., *C. dubia* are ubiquitous in freshwater aquatic ecosystems throughout the U.S.); they are easy to culture in a laboratory setting; and give reliable and consistent results such that toxicity comparisons between different water samples can be made. Standard laboratory methods used to test waters have been shown to correlate well with field biological assessments representing many species that are known to inhabit local streams.

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TABLE A3.15 USEPA approved WET test organisms in 40 CFR § 136.

Test Organism	Common Name	EPA Method	
		Acute	Chronic
Freshwater			
Ceriodaphnia dubia	Invertebrate, water flea	2002.0	1002.0
Daphnia puplex	Invertebrate, water flea	2021.0	NA
Daphnia magna	Invertebrate, water flea	2021.0	NA
Pimphales promelas	Fathead minnow	2000.0	1000.0/1001.0
Cyprinella leedsi	Bannerfin shiner	2000.0	NA
Oncorhynchus mykiss	Rainbow trout	2019.0	NA
Salvelinus fontinalis	Brook trout	2019.0	NA
Selenastrum capricornutum	Green alga	NA	1003.0
Estuarine and Marine			
Mysidopsis bahia	Mysid shrimp	2007.0	1007.0
Cyprinodon variegatus	Sheepshead minnow	2004.0	1004.0/1005.0
Menidia beryllina, M. menidia, M. peninsulae	Silverside	2006.0	1006.0*
Arbacia punctulata	Sea urchin	NA	1008.0

**M. beryllina* only

To initiate the acute toxicity test, a minimum of 20 individual organisms of a given species (e.g., *C. dubia*) are exposed to each effluent concentration (USEPA, 2002a). To initiate the chronic toxicity test, individual organisms of a given species (e.g., fathead minnow) are added to test units and their survival and reproduction over 7 days is followed; specific numbers of test organisms and test units required depend on the species being tested (USEPA, 2002b). These tests provide dose-response information, expressed as the percent effluent concentration that affects survival and/or reproduction during the 7-day test.

A large amount of data is collected during these tests, including NOEC (no observed effect concentration), LC (lethal concentration) and IC (inhibition concentration) levels. A NOEC is the highest concentration, or percentage (volume), of the water sample tested with a response that is not statistically significantly different from the control response. An LC50 represents the volume percentage of a water sample that results in mortality of 50% of the organisms tested. An IC25 describes the percent of the water sample that causes a 25% reduction in the mean number of young per female, or reduction of growth, depending on the specific endpoint being evaluated.

Toxicity often is expressed in toxic units (TU), where toxic unit acute (TUa) is the reciprocal of the LC50 and toxic unit chronic (TUc) is the reciprocal of the NOEC or the IC25. The USEPA technical support document (USEPA, 1991) recommends that a TUc of 1.0 and a TUa of 0.3 not be exceeded for the most sensitive of at least three different test species as criteria to protect aquatic life against chronic and acute toxicity, respectively.

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SPATIAL OR TEMPORAL CONSIDERATIONS:

Because of potential temporal variability in the toxicity of effluents, a negative test result for a particular water sample does not preclude the possibility that a sample collected at a different time might exhibit acute or chronic toxicity. The frequency of testing required under a NPDES permit is determined by the regulatory agency and based on factors such as the variability and degree of toxicity of the waste, production schedules, and process changes (USEPA, 2002a, b). The sampling dates and times should span a sufficient duration to represent the full range of effluent variability, and the samples must be tested according to USEPA's WET test methods in 40 § CFR 136.

ADDITIONAL CONSIDERATIONS:

It is possible that *C. dubia* or other USEPA approved WET species may be less or more sensitive to one or more toxic components in a given water sample, as compared to an indigenous species (those present at a given site). The use of indigenous species might provide a better site-specific toxicity assessment; however, many species are not amenable to laboratory testing. USEPA allows use of indigenous species only where State regulations require their use or prohibit importation of recommended species (USEPA, 2002a, b). Furthermore, observed toxicity does not result in identification of a specific toxicant; therefore, if an effluent or receiving water has persistent and consistent toxicity, then toxicity reduction evaluation (TRE) testing is conducted to identify (toxicity identification evaluation (TIE) is a component of TRE) and reduce the component responsible for causing the toxicity.

BIBLIOGRAPHY:

USEPA, 1991. *Technical support document for water quality-based toxic controls. Office of Water Enforcement and Permits and Office of Water Regulations and Standards.* U.S. Environmental Protection Agency, Washington, D.C., 20460. EPA/505/2-90/001.

USEPA. 1995. *Whole Effluent Toxicity: Guidelines Establishing Test Procedures for the Analysis of Pollutants* (FR Vol. 60 Number 199, pages 53529-53544), 10/16/95. Office of Science and Technology, EAD.

USEPA, 1999. *Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms; Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters*. U.S. Environmental Protection Agency, Washington, D.C., EPA-600/R-98/182

USEPA. 2000 *Method Guidance and Recommendations for Whole Effluent Toxicity (WET) Testing* (40 CFR Part 136). Office of Water, Office of Science and Technology. Washington, DC. EPA/821/B-00-004.

USEPA. 2002a. *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms*, 5th edition. U.S. Environmental Protection Agency, Washington, D.C., 20460 EPA 821/R-02/012. Office of Science and Technology.

USEPA, 2002b. Short-term methods for estimating the chronic toxicity of effluents and receiving waters to freshwater organisms. 4th Edition. Office of Water, U.S. Environmental Protection Agency, Washington D.C. 20460. EPA/821/R-02/013.

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Assessments of Habitats

(Virginia T. McLemore)

Habitats are the collective environment that allows species to evolve or the place where an organism or a community of organisms lives, including all living and nonliving factors or conditions of the surrounding environment. Generally species live in communities that depend on each other. The survival of species generally depends upon water, soil, local climate, altitude, and areas of food production. Mining can impact habitats, both in the long- and short-term, but mining companies today attempts to minimize these impacts. Habitat fragmentation, common in mining sites, is when large areas are broken up into smaller units, making use by species that depend on specific units difficult. Isolation of species or prevention of migration into certain areas can be detrimental to the overall health of the habitat.

MEDIUM: The following media in a habitat needs to be sampled and monitored:

- Percent vegetation cover by species
- Percent total vegetation cover (sum of all species)
- Percent total ground cover (vegetation, litter, rock)
- Percent bare ground
- Herbaceous production
- Shrub density
- List of species observed during sampling visit (animal and plant)
- Availability of water
- Season in which sampling and monitoring occurred (fall, winter, summer, spring). Ideally habitats should be sampled throughout the year to cover all seasons.
- Bioturbation
- Root penetration
- Burrowing

In an active mining area, the following needs to be monitored:

- Number of mining operations, such as number of placer dredges
- Royalties
- Number of claims staked or licenses granted and area covered

SAMPLING OBJECTIVES: Assessments of habitats are required to check on the health of the habitat and how well it may be responding to remediation or reclamation efforts. Vegetation development is documented. The area can be assessed for readiness for grazing or other land uses. Vegetation monitoring should include species composition, indices of stand structure, age or size distributions, indices of condition, and related physical variables such as depth to water.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUES: A variety of methods can be employed using appropriate sampling, collecting, and analytical equipment and techniques.

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SPATIAL OR TEMPORAL CONSIDERATIONS:

- Each area should have a unique identification number so that all sampling and monitoring activities can be documented and recorded. The site number, date, sampler, location, revegetation practices, and site specific data must be recorded at each site visit.
- Cover data can be collected using a line transect or point intercept method. In the line transect method, samples are collected at a given interval (such as 1 m) along the entire transect length. In the point intercept method, a grid is laid out and locations are randomly selected within the grid. In both methods, the sample locations should be located on maps.
- Production data can be collected using square plots or a given distance from a transect line.
- A list of all species observed during the cover and production sampling should be recorded as encountered. Species diversity and intensity should be recorded.

ADDITIONAL CONSIDERATIONS: Abandoned or inactive mines can be excellent bat habitats (Altenbach and Milford, 1995; Altenbach, 1998). Mines can be closed using bat-compatible closures. Construction work on abandoned or inactive mines should be done during specific times of the year when the bats (and other wildlife) are not using these features, so as not to seal the animals inside.

BIBLIOGRAPHY:

Altenbach, J.S., 1998, Abandoned mines as bat habitat: Cultural Resource Management Magazine, no. 7, http://www.cerrilloshills.org/mines/bats.html, accessed 12/13/10

Altenbach, J.S. and H. E. Milford, 1995 Evaluation and management of bats in abandoned Mines in New Mexico. In: D. Pate, ed. Proceedings of the 1993 Cave Management Symposium, Carlsbad, New Mexico, pp. 326-330.

Ferris, F. K., and others, 1996, Handbook of western reclamation technologies: Office of Surface Mining Reclamation and Enforcement, 504 p. (CD-ROM).

The Yukon Placer Economic Health Working Group, 2008, Fish habitat management system for Yukon placer mining: Yukon Placer Secretariat, 11 p., http://www.yukonplacersecretariat.ca/pdf/economic_health_monitoring_protocol_nov08.pdf, accessed 8/23/13.

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ANALYTICAL METHODS

This section summarizes some of the analytical methods used in sampling and monitoring programs. Figure A3.8 indicates some of the suggested analytical procedures for each element.

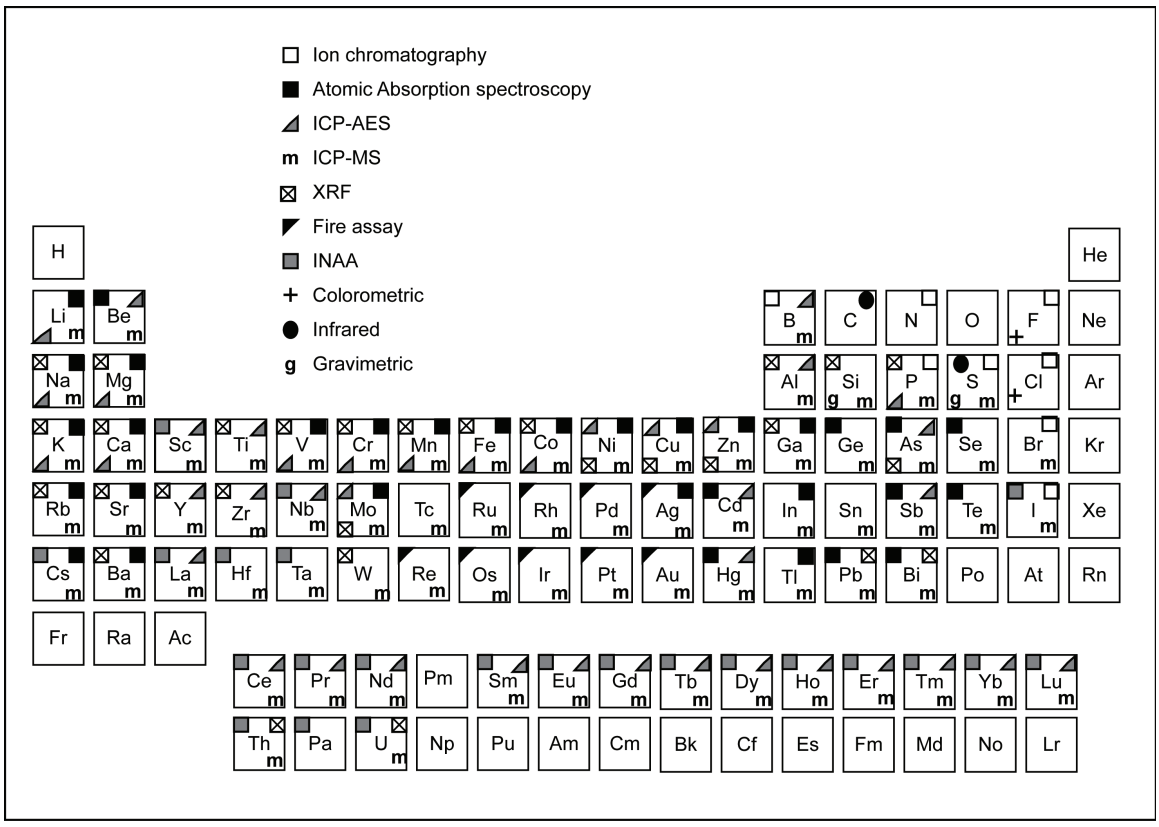


FIGURE A3.8. Summary of suggested analytical procedure for each element. This is not completely inclusive of all analytical methods that can be used for each method.

Cyanide Determination by Flow Injection Analysis with Gas Diffusion Separation and Amperometric Detection

(William Lipps)

MEDIA: Aqueous solutions

MEASUREMENT OBJECTIVE: To determine free, available, and/or total cyanide in water samples, or aqueous extracts. With minor modifications, the available methods are applicable to the determination of cyanide in mineral process solutions.

Free Cyanide – form of cyanide that is bioavailable and known for its toxic effects on organisms. At pH 7 or less in water, free cyanide is present as HCN.

SPECIFIC SAMPLING EQUIPMENT/TECHNIQUES: Flow Injection Gas diffusion amperometric cyanide methods minimize interferences encountered in leaching, detoxification, and environmental discharges from precious metal processing solutions.

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Sample pre-treatment may be necessary to mitigate potential loss of cyanide during collection, transport, and storage of samples prior to analysis. A holding time study should be conducted on all matrices; in many cases immediate analysis may be necessary. Refer to ASTM D7365 for detailed instructions.

ANALYTICAL TECHNIQUES:

Free Cyanide – The preferred method is ASTM D7237. The sample is injected into a reagent stream buffered at the pH of the aquatic environment (or at pH 6-8). The released hydrogen cyanide gas (HCN) diffuses through a hydrophobic membrane into an alkaline acceptor stream where CN^- is captured and passes through an amperometric flow cell. The current measured is proportional to the cyanide concentration.

Available Cyanide – Also known as WAD cyanide, the preferred method is OIA1677 and/or ASTM D6888. Nickel and mercury cyanide complexes are released by ligand displacement. Copper, silver, zinc, cadmium, and other weak cyanide complexes do not require ligand exchange reagents. Iron, gold, and platinum group metal complexes are not measured. The treated sample is injected into an acidic reagent releasing cyanide as HCN. The HCN diffuses through a hydrophobic membrane into an alkaline acceptor stream where CN^- is captured and passes through an amperometric flow-cell. The current measured is proportional to the cyanide concentration.

Total Cyanide – The preferred method is ASTM D7511. The method measures free cyanide, available cyanide, and cyanide from iron complexes. Cobalt, gold, and platinum group metal cyanide complexes are only partially recovered. The sample is injected into an acidic reagent stream and exposed to narrow band UV irradiation. Cyanide is liberated from the metal-cyanide complexes to form HCN. The HCN diffuses through a hydrophobic membrane into an alkaline acceptor stream where CN^- is captured and passes through an amperometric flow-cell. The current measured is proportional to the cyanide concentration.

Total Cyanide after distillation – An alternative method to ASTM D7511 is ASTM D7284. The method measures free cyanide, available cyanide, and cyanide from iron complexes. Cobalt, gold, and platinum group metal cyanide complexes are only partially recovered. The sample is first distilled from a strong sulfuric acid magnesium chloride reagent and captured in a basic absorber solution as CN^-. The distilled sample is injected into an acidic reagent releasing cyanide as HCN. The HCN diffuses through a hydrophobic membrane into an alkaline acceptor stream where CN^- is captured and passes through an amperometric flow-cell. The current measured is proportional to the cyanide concentration.

LIMITATIONS: Samples are usually aqueous, however, solid samples can be analyzed after a preliminary extraction into reagent water or basic solution. Sulfide can interfere with the free cyanide method (ASTM D7237), and should be removed using lead carbonate. Sulfide does not interfere with OIA1677, ASTM D6888, ASTM D7511, or ASTM D7284 at concentrations below 50 mg/L Sulfide. Thiocyanate interferes with all total cyanide methods causing both positive and negative interferences. Interferences from thiocyanate are generally greater with preliminary distillations.

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SCOPE: Quantitation ranges can vary by method, and by instrument manufacturer, but are usually in the low ppb to mid ppm. Methods can be modified to enable quantitation of cyanide as high as 2000 ppm. Accuracy may be ±30% at the lower calibration levels (depending on the method), or as good as ± 1% at mid-range calibration for some analytes.

DETECTION LIMITS: Detection limits vary by method and/or analyte but are usually in the low ppb to low ppm range in solution.

BIBLIOGRAPHY:

ASTM International 2010, Standard Guide for Understanding Cyanide Species, ASTM D6696, Philadelphia, Pennsylvania, ASTM.

ASTM International 2009, Standard Practice for Sampling, Preservation, and Mitigating Interferences in water Samples for Analysis of Cyanide, ASTM D7365, Philadelphia, Pennsylvania, ASTM.

ASTM International 2010, Standard Test Method for Free Cyanide with Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection, ASTM D7237, Philadelphia, Pennsylvania, ASTM

ASTM International 2009, Standard Test Method for Available Cyanide with Ligand Exchange Displacement and Flow Injection Analysis (FIA) Utilizing Gas Diffusion Separation and Amperometric Detection, ASTM D6888, Philadelphia, Pennsylvania, ASTM

ASTM International 2009, Standard Test Method for Total Cyanide by Segmented Flow Injection Analysis, In-Line Ultraviolet Digestion and Amperometric Detection, ASTM D7511, Philadelphia, Pennsylvania, ASTM

ASTM International 2008, Standard Test Method for Total Cyanide in Water by Micro Distillation followed by Flow Injection Analysis with Gas Diffusion Separation and Amperometric Detection, ASTM D7284, Philadelphia, Pennsylvania, ASTM

OI Analytical, Available Cyanide by Ligand Exchange and Flow Injection Analysis (FIA, OIA 1677, College Station, TX

OI Analytical, Determination of Total Cyanide by Segmented Flow Injection Analysis, In-line Ultraviolet Digestion and Amperometric Detection, OIA1678, College Station, TX.

Electron Microprobe Analysis

(Nelia Dunbar)

PRINCIPLE: Electron microprobe analysis (EMPA) is a non-destructive technique that allows quantitative analysis of a wide range of elements on very small (1 micron) area on a sample surface, as well as allowing chemical mapping of a sample surface. The technique uses a beam of electrons that is either focused onto the sample surface (for quantitative analysis), or is rastered, or swept, across the sample surface (for chemical mapping). The electron microprobe can also be backscattered electron imaging, which produces an image showing the mean atomic number of a flat sample surface, and secondary electron imaging, which produces a high-resolution three-dimensional image of a rough sample surface. With an additional detector, the electron microprobe can also

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be used to produce cathodoluminescent images (visible light produced by electron bombardment of a sample surface).

PRIMARY USES: The electron microprobe is used primarily for petrological and material science applications. The main strength of the electron microprobe is its microbeam capability, which allows quantitative determination of the chemical composition of 1 micron spot on a sample surface, as well as producing high-resolution images of a sample surface. The technique is well suited to mineral identification, studies of mineral alteration and the relationship between different mineral phases, studies of mineral zonation, geothermometry and geobarometry, particle analysis and location of trace phases.

LIMITATIONS: EMPA is not well suited to bulk geochemical analyses. The detection limits are higher than those typical for bulk analytical techniques. Analyses only provide information about the atomic concentrations of elements in a sample; no information on isotopic composition and limited information on the bonding states of elements can be determined. For high-quality quantitative analysis, high-quality standard reference materials must be available for all analyzed elements.

INTERFERENCES: Due to the simplicity of the x-ray spectra, there are few interferences that affect normal electron microprobe analysis. A few notable interferences that affect geological analyses are Ca and P, P and F, Ti and V. With modern software, determining potential interferences for a given sample composition is straightforward.

SCOPE: Traditionally, the electron microprobe has been using for analyzing and producing chemical maps of elements with atomic numbers between F and U. Crystals are now also available that can detect, map, and analyze C, N, and O. The electron microprobe is suitable for analyzing solid, polishable materials that are capable of withstanding a vacuum of $4x10^{-5}$ Pa. Aqueous, organic, or wet samples are not suitable for EMPA.

DETECTION LIMITS: There are no upper detection limits for EMPA, given that suitable standard reference materials are available. For normal analyses, detection limits are around 200-300 ppm for most elements. Lower detection limits of between 50-10 ppm can be obtained by using longer counting times, which increases the length of time for an individual analysis from 3-4 minutes to 15-20 minutes, or longer.

BIBLIOGRAPHY

Goldstein, J., 1979, Introduction to Analytical Electron Microscopy: New York and London, Plenum Press, 83 p.

Potts, P. J., Bowles, J. F. W., Reed, S. J. B., and Cave, M. R., 1995, Microprobe Techniques in Earth Sciences: London, Chapman and Hall, 419 p.

Reed, S. J. B., 1990, Recent developments in geochemical microanalysis: Chemical Geology, v. 83, p. 1-9.

Reed, S. J. B., 1993, Electron Microprobe Analysis: Cambridge, Cambridge University Press, 326 p.

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Reed, S. J. B., 1996, Electron Microprobe and Scanning Electron Microscopy in Geology: Cambridge, Cambridge University Press, 201 p.
Scott, V., and Love, G., 1983, Quantitative Electron-probe microanalysis: West Sussex, Ellis Horwood, 345 p.

Fire Assaying

(Ibrahim Gundiler)

PRINCIPLE: Fire assaying is an ancient form of chemical analysis for quantitative determination of precious metals, which is still widely used in the mining industry. It involves fusion of a known quantity of finely pulverized solid sample with a mixture of fluxes in a fireclay crucible at 1,000 C. Gold, silver, and platinum group metals are collected in molten lead, and the impurities are rejected into a complex borosilicate slag. Subsequently, lead is separated from slag, subjected to oxidizing fusion in a porous dish (cupel) made of magnesia or bone ash and lead oxide is absorbed into cupel. An alloy of noble metals is recovered as residue, and gravimetric methods, atomic-absorption spectrometry, or neutron activation analysis determines concentration of the individual elements.

PRIMARY USES: Fire assay is used as the primary method of analysis for gold bearing concentrates, loaded carbon, and bullion. For most other applications, it is a pre-concentration method, followed by modern analytical techniques.

LIMITATIONS: Requires certain skills and experience for formulating appropriate flux charges for different types of materials. Requires high temperature furnaces, assay supplies, microbalance, or atomic absorption spectrophotometer.

INTERFERENCES: High concentrations of copper, nickel, sulfur, arsenic, and tellurium may cause difficulties during fusion and cupellation.

SCOPE: Applicable for a variety of material such as gold bullion, concentrates, loaded carbon, analysis of geological materials, low-grade ores or tailings, process solutions.

DETECTION LIMITS: Depending on sample size and final finishing technique; gravimetric - 50 ppb, AAS - 5 ppb, NAA - 1 ppb.

BIBLIOGRAPHY

Beamish, F.E., and Van Loon. J.C., 1977, Analysis of Noble Metals, Overview of Selected Methods: Academic Press, New York.
Bugbee, E.E., 1940, A Textbook of Fire Assaying: John Wiley and Sons, New York, Reprinted by Legend Metallurgical Laboratory, Inc., 1991, Reno, Nevada.
Donaldson, E.M., 1982, Methods for the Analysis of Ores, Rocks and Related Material Energy: Mines and Resources Canada, CANMET, Ottawa.
Gilbert, T.J., (ed.), 1989, Methods Manual v. 2, Gold Analysis on Mill Process Solids, Society of Mineral Analysts: Available from Legend Metallurgical Inc., Reno, Nevada.

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Hafty, J., Riley, L.B., and Goss, W.D., 1977, A Manual for Fire Assaying and Determination of the Noble Metals in Geological Materials: U.S. Geological Survey, Bull. 1445, U.S. Government Printing Office, Washington, D.C.
Moloughney, P.E., 1986, Assay Methods Used in CANMET for the Determination of Precious Metals: Energy, Mines and Resources Canada, CANMET, Ottawa.
Shepard, O.C., and Dietrich, W.F., Fire Assaying: McGraw-Hill Book Co, New York.

Flame Atomic Absorption Spectrophotometry (FAAS)

(Lynn A. Brandvold)

PRINCIPLE: Production of ground state atoms in a flame, which absorb light at wavelengths characteristic of that element. The extent of that absorption is a measure of the concentration of the element in the light path.

PRIMARY USES: Minor and trace metal determinations in liquids and digested solids. Particularly useful for mineralized samples and high dissolved solids samples where matrix matching is difficult.

SAMPLE PREPARATION: Solid samples must be digested by acid into liquid form.

LIMITATIONS: Sample must be in liquid form and is consumed by analysis. Solid samples may be solubilized with acid treatment or by alkaline fusion. Method determines single elements only and is limited to about 45 metals. Dynamic concentration range is limited to about 2 orders of magnitude, although dilution and concentration can expand this. FAAS is not a good method to use for high concentrations of metals (>20%). Precision is only about ± 5%. Refractory metals whose oxides do not break down at high temperatures have poor sensitivity.

INTERFERENCES: In theory there are no interferences, in practice there can be some and they are of three types; spectral, physical and chemical. Spectral interferences can result from spectral line overlap; this is of particular importance in the case of gold and platinum because iron, which is universally present in rock samples, has lines, which overlap those of gold and platinum. Gold and platinum can be determined by FAAS but the iron must be removed first. Physical interferences can result because of density or surface tension differences between samples and standards. Reactions that prevent the formation of ground state atoms in the flame can cause chemical interferences; such as the formation of oxides or ions.

SCOPE: Analytical concentration range differs by element, but generally ranges from 0.1-100 ppm in solution. Metals that form refractory oxides in the flame are generally less sensitive. Sodium, lithium, copper, cadmium, and zinc are very sensitive. Mercury by cold vapor AA and arsenic and selenium but hydride generation AA are very sensitive adaptations of this technique.

DETECTION LIMITS: Detection limits that differ by element are generally in the ppm range in solution.

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BIBLIOGRAPHY:
Potts, P. J., 1987, A handbook of silicate rock analysis: Chapman and Hall, New York, NY, 610 p.
Reeves, R. D. and Brooks, R. R., 1978, Trace element analysis of geological materials: Chemical Analysis, Monographs on analytical chemistry and its applications, v. 51, John Wiley & Sons, NY, NY.
Riddle, C., ed., 1993, Analysis of geological materials: Marcel Dekker, Inc., New York, 67 p.
Slavin, Morris, 1978, Atomic absorption spectroscopy: Chemical Analysis, v. 25, John Wiley and Sons, New York, NY, 187 p.
Smith, Keith A., ed., 1983, Soil analysis: Marcel Dekker, Inc. New York, NY, 511 p.
Van Loon, Jon C., 1980, Analytical atomic absorption spectroscopy: selected methods: Academic Press, Inc., New York, NY, 330 p.

Graphite Furnace Atomic Absorption (GFAA)

(Lynn A. Brandvold)

PRINCIPLE: The principle is the same as that of FAAS. The method differs in that ground state atoms are produced by rapidly heating a graphite tube rather than using a flame.

PRIMARY USES: Trace metal determinations in water and digested solids. The method can determine USEPA's Primary Pollutant Metals to the required detection limits whereas frequently ICP-AES and FAAS cannot.

SAMPLE PREPARATION: Solid samples must be digested by acid into liquid form.

LIMITATIONS: Sample must be in liquid form. Solid samples may be solubilized with acid treatment. Method determines single elements only but with the appropriate hardware and software up to six elements may be determined sequentially. The method is limited to about 45 metals. Dynamic concentration range is limited to about 2 orders of magnitude, although dilution and concentration can expand this. Samples can be concentrated during a determination by multiple injections of sample. Precision ranges from ± 5-20 %. Refractory elements have poor sensitivities.

INTERFERENCES: Some type of background correction is needed for GFAA to correct for complex matrices and the resulting high background absorption. For example, NaCl will result in high broad range background absorbance. Matrix modifiers are also used to reduce interferences. Through the use of background correction and appropriate matrix modifiers high dissolved solids and viscous solutions can be analyzed.

SCOPE: Analytical concentration range differs by element, but generally ranges from 0.1-100 ppb in solution.

DETECTION LIMITS: Detection limits differ by element and are generally in the 1-2 ppb range in solution and sensitive elements can be determine to 0.1 ppb.

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BIBLIOGRAPHY:

Slavin, Walter, 1984, Graphite furnace AAS: a source book:. Perkin-Elmer Corp., Ridgefield, CT.

Reeves, R. D. and Brooks, R. R., 1978, Trace element analysis of geological materials: Chemical analysis, Monographs on analytical chemistry and its applications, v. 51, John Wiley and Sons, NY, NY.

USEPA (U.S. Environmental Protection Agency), 1983, Methods for chemical analysis of water and wastes: USEPA, EPA-600/4-79-020, Environmental Monitoring Support Lab, Cincinnati, Ohio.

USEPA (U.S. Environmental Protection Agency), 1994, Test methods for evaluating solid waste, physical/chemical methods (SW-846): 3rd edition, update 2B, Environmental Protection Agency, National Center for Environmental Publications, Cincinnati, Ohio, 800-553-6847, order number EPASW-846.3.2B, accessed at URL http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm, accessed 8/23/13.

Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)

(Lynn A. Brandvold)

PRINCIPLE: An inductively coupled plasma (ICP) torch serves to atomize and ionize the sample. Liquids are introduced by a nebulizer and solids by laser ablation. Metal ions produced by the torch are sampled through a differentially pumped interface linked to a quadrupole mass spectrometer (MS). The MS separates the ions on the basis of their mass-to-charge ratio. Detection is accomplished by converting the separated ions to an electrical signal.

PRIMARY USES: Metals in solutions and some digested solids can be determined at trace and ultra-trace levels including rare earth elements and platinum group elements. Isotopes of many of the metal elements can be determined as well.

SAMPLE PREPARATION: Solid samples must be digested by acid into liquid form.

LIMITATIONS: The ICP-MS is a trace and ultra-trace metal analytical technique. Most samples must be in liquid form. It requires a much larger liquid sample size (5 ml) than GFAA (1 ml). However, because of excellent sensitivity, samples can often be diluted with little effect in sensitivity.

INTERFERENCES: Matrix effects become noticeable at concentrations of greater than about 500 to 1000 part per million (ppm). This effect is quite general in that a high concentration of nearly any element results in a matrix effect. This effect can be minimized by dilution or separation of the offending species. There are also mass spectroscopic interferences with isobars (overlaps of mass-to-charge ratio), polyatomic molecules, and doubly-charged ions.

SCOPE: ICP-MS is a multi-element technique for determining trace and ultra-trace elements. Isotopic analyses can also be performed. It is used for a wide variety of media; water, soils, plants, and rocks. Laser ablation may be used for rock samples prepared as

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thin sections and slurry nebulization may be used for fine solid particles. Plants must be solubilized.

DETECTION LIMITS: The detection limits for most elements is in the sub-parts per billion (ppb). For some elements it lies in the sub-part per trillion (ppt) range. This excellent sensitivity makes ICP-MS the method of choice for elements that have low environmental concentration standards.

BIBLIOGRAPHY:

Skoog, Douglas A., Holler, James F., and Nieman, Timothy A., 1998, *Principles of Instrumental Analysis,* 5th Edition, Harcourt Brace College Publishers.

Taylor, Howard E., 2001, *Inductively Coupled Plasma-Mass Spectrometry*, Academic Press.

Agilent Technologies Student Manual, 2001, *Agilent 7500 Inductively Coupled Plasma Mass Spectrometry*, Manual Part Number H8974-90000.

Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

(Lynn A. Brandvold)

PRINCIPLE: ICP-AES is a multi-element analytical method appropriate for most sample media with proper sample preparation. It has a wide range of element concentration that can be determined in one analysis (wide dynamic range). Solid samples must be decomposed or digested by various combinations of acids before analysis. To reach the low environmental concentration standards that are established for some elements such as copper, arsenic, selenium, and thallium, liquid samples must be pre-concentrated by evaporation before analysis.

PRIMARY USES: Offers detection limits frequently better than flame atomic absorption, particularly for refractory elements such as Al, Ba, Mo, and W. It also offers low detection limits for some elements that can't be determined by flame or graphite atomic absorption such as U, Th, and rare earth elements. Because of the simultaneous multi-element capability of ICP-AES, it is the workhorse for routine analysis of natural waters.

SAMPLE PREPARATION: Solid samples must be digested by acid into liquid form.

LIMITATIONS: Highly mineralized samples pose a problem in analyzing for trace elements because of matrix interferences. Brines and solid samples which must be decomposed by a fusion require special techniques not only because of matrix interferences but because they pose sample introduction problems and salt build-up on the torch.

INTERFERENCES: ICP-AES interferences can result from spectral interferences, background shifts, and matrix effects in the sample. Spectral interference corrections, inter-element correction factors, and background corrections are applied using data system software specific to the manufacture of the instrument being used. Matching of

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standard and sample matrices can minimize matrix effects. Spectral overlap interferences are difficult to correct for.

SCOPE: Analysis by ICP-AES is appropriate for major, minor, and trace elements in a variety of sample media.

DETECTION LIMITS: Detection limits that differ by element are generally in the ppm range in solution.

BIBLIOGRAPHY

Briggs, P. H., 1996, Forty elements by inductively coupled plasma-atomic emission spectrometry; *in* Arbogast, B. F., Analytical methods manual for the Mineral Resource Surveys program: U.S. Geological Survey: U.S. Geological Survey, Open-file Report 96-525, p. 77-94.

Briggs, P. H. and Fey, D. L., 1996, Twenty-four elements in natural and acid mine waters by inductively coupled plasma-atomic emission spectrometry; *in* Arbogast, B. F., Analytical methods manual for the Mineral Resource Surveys program: U.S. Geological Survey: U.S. Geological Survey, Open-file Report 96-525, p. 95-101.

Lichte, F. E., Golightly, D., and Lamothe, P. J., 1987, Inductively coupled plasma-atomic emission spectrometry; *in* Baedecker, P. ed., Methods for geochemical analysis: U. S. Geological Survey, Bulletin 1770, p. B1-B10.

Potts, P. J., 1993, Laboratory methods of analysis; *in* Riddle, C., ed., Analysis of geological materials: Marcel Dekker, Inc., New York, New York, p. 123-220.

Thompson, M. and Walsh, J. N., 1989, Handbook of inductively coupled plasma spectrometry: Blackie and Son Ltd., London, England, p. 201-210.

Instrumental Neutron Activation Analysis (INAA)

(Virginia T. McLemore)

PRINCIPLE: When a sample is irradiated in a nuclear reactor by a high flux of neutrons, neutron capture reactions occur. Specific isotopes of most elements undergo neutron capture reactions during irradiation. Radioactive decay with a characteristic half-life begins that emits gamma radiation and produces stable daughter elements. INAA involves the measurement of the intensity of the characteristic gamma emissions associated with the radioactive decay, which is proportional to the elemental composition.

PRIMARY USES: INAA analyses are performed on powdered solid media and require access to a reactor in order to irradiate the samples.

LIMITATIONS: INAA is applicable for trace element analysis of solid media only. The samples are radioactive and must be properly disposed of.

INTERFERENCES: INAA interferences can result from spectral interferences and background shifts. Matching of standard and sample matrices can minimize matrix interferences. Highly mineralized samples, especially lead, cause analytical interferences, which are difficult to correct for. INAA is sensitive to oxidation state and chemical

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bonding effects. Neutron flux in the nuclear reactor is a variable that must be accounted for.

SCOPE: INAA is a nondestructive technique used for quantitative analysis of trace analysis (sensitivity) to 0.1 ppm levels.

DETECTION LIMITS: Detection limits differ by element and are generally in the 0.1-5 ppm range.

BIBLIOGRAPHY

Parry, S. J., 1991, Activation spectrometry in chemical analysis: Wiley, Chichester, 243 p.

Riddle, C., ed., 1993, Analysis of geological materials: Marcel Dekker, Inc., New York, 67 p.

Ion Chromatography (IC)

(Lynn A. Brandvold)

PRINCIPLE: Ions in a liquid are separated by a resin column; and detected and quantified by conductivity, uv/vis spectrophotometry, or electrochemical methods. The most common detection method is conductivity. Both cations and anions can be determined by this method. Separations involve a mobile liquid traveling through a packed column containing a stationary phase. The sample mixture, introduced into the mobile liquid, undergoes a series of interactions with the stationary phase as it moves through the column. Separated ions emerge in the order of increasing interaction with the stationary material—the least retarded component elutes first, the most strongly retained material elutes last.

PRIMARY USES: The method of choice for determining F^{-1}, Br^{-1}, Cl^{-1}, NO_3^{-1}, SO_4^{-2}, and $PO4^{-3}$ in water samples. With the proper instrumentation these anions can be analyzed in a single determination.

LIMITATIONS: The usual method of sample preservation for nitrate and nitrite, addition of sulfuric acid, cannot be used. Nitrate and nitrite must be determined within 48 hours. Fluoride in the presence of large amounts of bicarbonate in some instances may not be able to be determined. Newer techniques of automatic ranging of the conductivity detector allow for the determinations of high concentrations of ions and low concentrations of ions in the same sample.

INTERFERENCES: Interferences can be caused by substances with retention times similar to those of the ions of interest. A large concentration of one ion may cause baseline or peak resolution problems for an ion present in low concentration. High concentration of an ion may also change the retention time for an ion present in low concentration. Small organic compounds and high amounts of carbonate/bicarbonate are only slightly retained by the column and can elute with fluoride and interfere.

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SCOPE: This method is applicable to the analysis of major and minor ions in waters and leach solutions. It has the advantage that all the cations or all the anions can be determined with a single run.

DETECTION LIMITS: Detection limits vary depending on the ion; chloride can be determined in the 1 ppb range; fluoride in the 10 ppb range; nitrate, and sulfate in the 100 ppb range.

BIBLIOGRAPHY:

Her Majesty's Stationery Office, 1983, Methods for the examination of waters and associated materials: high performance liquid chromatography, ion chromatography, thin layer and column chromatography of water samples, Her Majesty's Stationery Office, London.

Mulik, J. D. and Sawicki, E., 1979, Ion chromatographic analysis of environmental pollutants: v. 2, Ann Arbor Science Publishers, Ann Arbor, MI.

Pohl, C. A. and Johnson, E. L., 1982, Review of ion chromatography: Journal of Chromatographic Science, v. 18, p. 442.

Sawicki, E., Mulik, J. D., and Wittgenstein (eds.), 1978, Ion chromatographic analysis of environmental pollutants: v. 1, Ann Arbor Science Publishers, Ann Arbor, MI.

Smith, F. C. and Chang, R. C., 1982, The practice of ion chromatography: John Wiley & Sons, Chichester, England.

Petrographic Analyses

(Virginia T. McLemore)

PRINCIPLE: Field observations and petrographic analysis of soils, rocks, and other solid materials are critical in understanding the composition, especially mineralogy, of the rocks and the processes the samples were subjected to (i.e. alteration, diagenesis, weathering, etc.). Petrographic analysis differentiates various rock units, determines the mineral phases present, determines the rank and intensity of alteration, determines the chemistry of alternating fluids, describes the cementation, describes textural relationships between mineral phases, and determines the paragenesis of mineralization, alteration, and cementation.

PRIMARY USES: Mineralogical data is helpful in understanding weathering and alteration processes, selecting samples for humidity cells, and for developing geotechnical, hydrological, and geologic (mineralogy, stratigraphy, internal structure) conceptual models of the site.

LIMITATIONS: Petrographic analysis of rocks has been traditionally performed with optical microscopy using thin sections and point counting thereby providing a quantitative modal mineralogy (Delvigne, 1998). However, these methods do not always provide accurate mineral proportions for sedimentary, volcanic, and soil-like material, because rock fragments, groundmass, and matrix are typically identified as separate phases, not as specific minerals. In addition, groundmass phases in volcanic rocks can be too fine to identify individually by these methods, and making thin sections of intact soil-

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like material can be difficult because much of the material is not well cemented. In addition, variable modal analyses can be obtained by different petrographers.

INTERFERENCES: Determination of fine-grained minerals, especially clay minerals and groundmass by petrographic analysis is difficult and other methods should be used.

DETECTION LIMITS: Mineral abundances are reported in weight percent and the detection limits vary on the petrographer and method of determining the mineralogy.

DATA FORMAT: Mineral concentrations are estimated using standard charts and data are summarized as weight percent. Estimates of both primary and alteration minerals are determined, cementation is described, diagenesis is described, porosity is estimated, and the alteration intensity is determined from the concentration of alteration minerals. Any special features are noted. Pyrite and carbonate minerals are described in detail because of their importance in generating or preventing acid drainage. The abundance, texture, grain size, contact relationships, shape, integrity, paragenesis, composition, surface area, and occurrence or association are described in detail in the petrographic form. Specific carbonate minerals are identified. Work sheets, descriptive text or relational databases can be used to document petrographic analysis.

SCOPE: Petrography generally is performed using standard petrographic and reflected ore microscopy techniques, using rocks. Mineral concentrations are estimated using standard charts and data are summarized. Estimates of both primary and alteration minerals are determined, cementation described, diagenesis described, porosity estimated, and the alteration intensity are determined from the concentration of alteration minerals. Any special features will be noted. Photographs are taken.

Petrographic analysis is improved by additional methods including electron microprobe analysis, clay mineral analysis (Moore and Reynolds, 1989), Rietveld method (Bish and Howard, 1988; Bish and Post, 1993; Young, 1993; Oerter et al., 2007), other mineralogical determinations, and the whole-rock chemistry of the sample. Another approach that can be used to determine quantitative mineralogy is to calculate a normative mineralogy from the whole-rock chemical composition. A normative mineralogy is the quantity of idealized minerals that are calculated from a whole-rock chemical analysis, although not all calculated minerals are necessarily actually present in the sample (Neuendorf et al., 2005). Normative mineralogy is usually calculated for volcanic rocks that have glass and/or very small crystals, for which it is difficult to determine a modal mineralogy. A normative mineralogy also is calculated for altered or metamorphosed igneous rocks that no longer have the original igneous mineralogy. One of the most widely used normative calculations is the CIPW norm. Another normative method involves the combination of X-ray diffraction and whole-rock chemical analysis and attempts to provide a better quantitative mineralogy (SEDNORM; Cohen and Ward, 1991; Ward et al., 1999). The modified ModAn quantitative mineralogy method is a normative mineralogy that makes use of and is consistent with the available petrographic observations, electron microprobe analysis, clay mineral analysis, other mineralogical determinations and the whole-rock chemistry of the sample (Paktunc, 1998, 2001; McLemore et al., 2009).

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Soil petrography also is used to describe the lithology, alteration, and mineralogy of mine soil samples through the use of a binocular microscope. Soils are rinsed in cool tap water to remove adhering fine material and to aid in the examination of rock fragment lithology and mineral identification. Examine approximately 10 g of a representative portion of mine soil sample prior to rinsing. Place this same split in a glass beaker, add enough water to cover the sample, and swirl beaker. Decant water, which will include some clay and silt sized material, if present. Repeat this procedure until water is relatively clear and adhering fines are mostly removed. To limit the dissolution of gypsum and soluble salts, do not repeat rinsing more than 10 times or leave mine soil immersed in water for extended periods of time. Write the sample number on a piece of filter paper with indelible ink. Place the filter paper in the funnel and pour mine soil and any remaining water into the funnel. Rinse the beaker repeatedly until all material is removed and deposited on the filter paper in the funnel. Drain any water from the funnel and air-dry the sample on the filter paper.

Examine unrinsed mine soil sample under the binocular microscope and note the abundance and color of any fine material present, describe cohesion and/or cementation, and note grain shape based on AGI data sheet 18. Test for calcite by placing dilute HCl on a representative portion of sample. Note if there is any reaction to HCl and how vigorous the reaction. Take a general photo of unrinsed sample and any other pertinent features. Document these and any other observations of the unrinsed sample.

After rinsing, examine rinsed and dried mine soil under the binocular microscope. Document observations and measurements.

Modal Mineralogy

Modal mineralogy is the abundance (in volume %) of all minerals found in a rock or soil based on observations and measurements. For mine soil samples, several analytical techniques are used to estimate the modal mineralogy, including soil petrography, electron microprobe, clay analysis (XRD), Reitveld analysis, and whole-rock geochemistry. Modal mineralogy for mine soils is an estimation only, because it is not determined by point counting due to the unconsolidated nature and heterogeneity of the material. Point counting can be used in rock samples.

Description of alteration

Describe the rank and intensity of hydrothermal alteration. Alteration is a general term describing the mineralogical, textural, and chemical changes of a rock as a result of a change in the physical, thermal, and chemical environment in the presence of water, steam, or gas (Henley and Ellis, 1983). The nature of the alteration depends upon (a) temperature and pressure at the alteration site, (b) composition of the parent rock, (c) composition and temperature of the alteration (invading) fluids, (d) permeability of the parent rock, and (e) duration of the alteration process. Recognition and genesis of alteration are important in mineral exploration and understanding the formation of ore deposits because specific alteration types are associated with specific ore deposits. Furthermore, alteration halos surrounding ore deposits are typically more widespread and easier to recognize than some of the orebodies themselves (Guilbert and Park, 1986).

Intensity of alteration is a measure of how much alteration has occurred that can be estimated by determining the percentage of newly-formed secondary minerals by

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visual examination (McLemore, 1993). For example, a parent rock that has not been affected by any alteration would have zero intensity of alteration, whereas a parent rock in which all primary minerals have been replaced by secondary minerals would have an alteration intensity of 100%.

Alteration rank is based upon the identification of new secondary minerals and their significance in terms of alteration conditions such as temperature, pressure, and permeability. The intensity of alteration is independent of the rank of alteration (McLemore, 1993). It is possible to have rocks with a high rank but low intensity (hot, impermeable zones) or other rocks of low rank but high intensity (cooler, permeable zones; McLemore, 1993).

Each alteration rank is associated with a particular mineral assemblage. The term mineral assemblage implies mutual equilibrium growth of mineral phases, whereas mineral association implies that the mineral phases are only in physical contact. A number of terms are applied to various alteration assemblages.

- Deuteric alteration refers to the interaction between volcanic or magmatic rocks and magmatic-hydrothermal fluids during the cooling of the igneous rocks. A variety of alteration minerals may be produced during deuteric alteration.
- Propylitic alteration is identified by the presence of epidote and chlorite. Carbonate is also part of this alteration assemblage, but is often not present due to prior dissolution.
- Sericitic alteration is defined by the dominance of illite, sericite, and/or muscovite. The major difference between these three K-micas is grain size: illite is a clay-size K-mica, whereas muscovite is larger. Sericite is of intermediate size. Some minor compositional differences also occur between the three minerals.
- Argillic alteration consists of kaolinite, smectite (montmorillonite clays), chlorite, and sericite. Advanced argillic alteration consists of kaolinite, quartz, alunite, pyrophyllite, and other aluminosilicate minerals.
- Silicic alteration is produced by the addition of silica, predominantly as quartz.
- QSP alteration is identified by the mineral assemblage of quartz-sericite-pyrite. However, pyrite is often partially or wholly replaced by Fe-oxides or Fe-oxyhydroxides.

Alteration of parent rock occurs by several processes: (1) direct deposition, (2) replacement, (3) leaching, and (4) ejecta (Browne, 1978). Evidence of all four processes is found in the Questa district.

Direct deposition occurs by precipitation of new minerals in open spaces, such as vugs or fractures. Replacement occurs when one mineral is converted to a new mineral by fluids entering the rock. These two processes are common and depend upon permeability and duration of the process. Complete fluid/mineral equilibrium is rarely achieved because of these factors. Leaching and supergene enrichment occurs locally where steam condensate reacts to form acidic solutions by the oxidation of H_2S or CO_2 which then attacks the parent rock and dissolves primary or secondary minerals. Silica residue is a common result of leaching and is a spongy or vuggy altered rock predominantly consisting of quartz, iron and titanium oxides. Direct deposition or replacement may occur after leaching, thereby producing overlapping alteration types.

Ejecta, hydrothermal brecciation, and hydrofracturing are another form of alteration where hot water and/or steam physically break apart the parent or altered rock. If this

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forceful ejection of fluids occurs at or near the surface, hydrothermal eruptions of water, steam, and rock can occur. Silicification following the brecciation is common.

After hydrothermal alteration, the rocks and minerals may be subjected to supergene alteration (long-term weathering). The term "weathering" is used in this document to describe chemical and physical changes that have occurred since the material was emplaced in rock piles, a process which continues today. However, distinguishing between supergene alteration and modern weathering may be difficult. If possible, also note whether minerals are primary, secondary, alteration, and/or weathering.

Description of pyrite and carbonate minerals

Pyrite and carbonate minerals are described in detail because of their importance in generating or preventing acid drainage. The abundance, texture, grain size, contact relationships, shape, integrity, paragenesis, composition, surface area, and occurrence or association are described in detail. Specific carbonate minerals are identified. Note whether pyrite occurs as individual grains or within rock fragments or clasts. Test for calcite with dilute HCl after rinsing and note where calcite occurs (e.g., within fine material, as discrete grains, or within altered rock fragments). Use a hand lens or binocular microscope to observe fizzing. Describe any other carbonate minerals present. Note the proportions of different forms of gypsum.

The form and texture of gypsum indicate its origin as being either detrital (incorporated before material was emplaced in the rock pile) or authigenic (grown in the rock pile as a result of in-situ weathering). Some soil samples contain both detrital and authigenic gypsum. It is important to describe the types of gypsum within each sample and estimate the relative percentages of each type.

COST: Petrographic analysis of a single sample can take several hours to a day to complete depending upon the complexity of the sample.

BIBLIOGRAPHY:

AGI Data Sheets for Geology in the Field, Laboratory, and Office, 1982, Compiled by R.V. Dietrich, J.T. Dutro, Jr., and R.M. Foose.

Bish, D.L. and Howard, S.A., 1988, Quantitative phase analysis using the Rietveld method: Journal of Applied Crystallography, v. 21, p. 86-91.

Bish, D.L. and Post, J.E., 1993, Quantitative mineralogical analysis using the Rietveld full-pattern fitting method: American Mineralogist, v. 78, p. 932-940.

Browne, P. R. L., 1978, Hydrothermal alteration in active geothermal fields: Annual Reviews of Earth and Planetary Science, v. 6, pp. 229–250.

Cohen, D.R. and Ward, C.R., 1991, SEDNORM—a program to calculate a normative mineralogy for sedimentary rocks based on chemical analyses: Computers and Geosciences, v. 17, p. 1235-1253.

Cross, W., Iddings, J.P., Pirsson, L.V., and Washington, H.S., 1903, Quantitative classification of igneous rocks, University of Chicago Press, London, 286 p.

Delvigne, J.E., 1998, Atlas of micromorphology of mineral alteration and weathering: Canadian Mineralogist, Special Publication 3, 494 p.

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Donahue, K, 2007, Origins of clay minerals in the Molycorp Mine Goathill North Rock Pile, Questa, NM. SME annual meeting.

Guilbert, J. M., and Park, C. F., Jr., 1986, The geology of ore deposits: W. H. Freeman and Company, New York, New York, 985 p.

Henley, R. W., and Ellis, A. J., 1983, Geothermal systems, ancient and modern: a geochemical review: Earth Science Reviews, v. 19, p. 1–50.

McLemore, V. T., 1993, Geology and geochemistry of the mineralization and alteration in the Steeple Rock district, Grant County, New Mexico and Greenlee County, Arizona: Ph.D. dissertation, University of Texas at El Paso; also New Mexico Bureau of Mines and Mineral Resources, Open File Report 397, 526 p.

McLemore, V., Sweeney, D., Dunbar, N., Heizler, L. and Phillips, E., 2009, Determining quantitative mineralogy using a combination of petrographic techniques, whole rock chemistry, and MODAN: Society of Mining, Metallurgy and Exploration Annual Convention, preprint 09-20, 19 p.

Moore, O.M. and Reynolds, R.O., Jr., 1989, X-ray diffraction and the identification and analyses of clay minerals: Oxford University Press, New York, 378 p.

Neuendorf, K.K.E., Mehl, J.P., Jr., and Jackson, J.A., 2005, Glossary of Geology: American Geological Institute, 5th ed., Alexandria, Virginia, 779 p.

Oerter, E., Brimhall, G.H., Jr., Redmond, J., and Walker, B., 2007, A method for quantitative pyrite abundance in mine rock piles by powder X-ray diffraction and Rietveld method: Applied Geochemistry, v. 22, p. 2901-1915.

Paktunc, A.D., 1998, MODAN: An interactive computer program for estimating mineral quantities based on bulk composition: Computers and Geoscience, v. 24 (5), p. 425-431.

Paktunc, A.D., 2001, MODAN—A Computer program for estimating mineral quantities based on bulk composition: Windows version. Computers and Geosciences, 27, 883-886.Van der Plas, D. and Tobi, A.C., 1965, A chart for judging the reliability of point counting results: American Journal of Science, v. 263, p. 87-90.

Ragland, P.C., 1989, Basic Analytical Petrology, Oxford University Press, Inc., New York, 369 p.

Richard, Linda R., MINPET version 2.02, Mineralogical and Petrological Data Processing System.

Simmons, S. F., Browne, P. R. L., and Brathwaite, R. L., 1992, Active and extinct hydrothermal systems of the North Island, New Zealand: Society of Economic Geologists, Guidebook Series, v. 15, 121 pp.

Van der Plas, D. and Tobi, A.C., 1965, A chart for judging the reliability of point counting results: American Journal of Science, v. 263, p. 87-90.

Ward, C.R., Taylor, J.C., and Cohen, D.R., 1999, Quantitative mineralogy of sandstones by X-ray diffractometry and normative analysis: Journal of Sedimentary Research, v. 69, no. 5, p. 1050-1062.

Williams, H., Turner, F.J., and Gilbert, C.M., 1954, Petrography; an introduction to the study of rocks in thin sections, W.H. Freeman, San Francisco, 406 p.

Young, R.A., ed., 1993, the Rietveld method: Oxford, U.K., Oxford University Press, 298 p.

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Scanning Electron Microscopy-Based Quantitative Mineralogical Analysis (MLA, QEMSCAN)

(Karin Olson Hoal)

PRINCIPLE: The method is based on the high-speed (up to 150 determinations/second) acquisition of backscatter electrons and X-rays at user-defined resolution (to 2 micrometers), and the production of false-colored images and data sets for sample quantification and interrogation. The technology was developed principally for applications related to quantification of metal distribution for mining projects, and developmental work into oil and gas, environmental, and other applications has been successful.

PRIMARY USES: The method is used to portray and quantify mineralogical parameters such as modal abundance, mineral associations, locking and liberation, mineral texture, microfractures and porosity in particulate and solid samples including drill core and geological samples, metallurgical feed, concentrate and tailings samples, as well as dust, soil, oil shale, coal and other naturally occurring and some synthetic materials from diverse industries.

LIMITATIONS: Limitations of the method include a reliance on high-speed acquisition of semi-quantitative data (up to 1000 counts per second) by energy-dispersive spectrometry, reliance on user-refined mineral libraries, pixel resolution to 2 micrometers (at the present time), and complex software requiring training and mineralogical background. Many of these limitations are being addressed for future versions.

INTERFERENCES: Key analytical issues relate to being a comparative (non-absolute) method of analysis, requiring understanding of X-ray peak overlaps and boundary phase identification. The elemental sample activation volume and beam size (0.5 micrometer) limit resolution capability.

DETECTION LIMITS: Lower limits of detection are based on elemental X-ray counts per 1000, and equate to about 1 wt% element abundance in a mineral.

DATA FORMAT: Data are presented in false-colored images, charts and graphs, and data sets that are exportable into spreadsheet format or other applications.

SPECIFIC QA/QC CONSIDERATIONS: The method should be used in tandem with other mineralogical methods such as optical microscopy, electron microprobe, and x-ray diffraction for mineral species validation and quality control. Sample sizes are typically thin-section sized (for solids) or 1-2 g mounted in resin and graphite blocks (for particulates). Therefore sample representativeness and density settling provide additional QA/QC concerns.

BIBLIOGRAPHY:

Butcher, A.R., Gravestock, D.I., Gottlieb, P., Gubitt, C., Edwards, G.V. 2000. A new way to analyse drill cuttings: A case study from Deparanie-1, Cooper Basin, South

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Australia. *Australian Petroleum Production & Exploration Association Limited (APPEA) Journal* 40:774-775.

Camm, G.S., Glass, H.J., Bryce, D.W., Butcher, A.R. 2005. Characterisation of a mining-related arsenic-contaminated site, Cornwall, U.K. *Journal of Geochemical Exploration* 82:1-15.

Gottlieb, P., Wilkie, G., Sutherland, D., Ho-Tun, E., Suthers, S., Perer, K., Jenkins, B., Spencer, S., Butcher, A., Rayner, J. 2000. Using quantitative electron microscopy for process mineralogy applications. *Journal of The Minerals, Metals & Materials Society (JOM)* April, p. 24-25.

Hoal, K.O., Stammer, J.G., Appleby, S.K., Botha, J., Ross, J.K., and Botha, P.W. 2009. Research in quantitative mineralogy: Examples from diverse applications. *Mineral Engineering* 22:402-408.

Soil Gas Analyses

(Virginia T. McLemore)

PRINCIPLE: Soil gas surveys involve the collection of vapors or gases from soil pore spaces in the vadose zone. Soil gas surveys are used to measure and determine the spatial distribution of specific unwanted soil gases emitting from the soil as well as identify the source. Soil gases provide an excellent means of characterizing the soil environment. Studies have shown that high soil gas anomalies are concentrated over buried fractures and faults because these geologic features generally have high permeability (Sibley, 1992; Fu et al., 2005; Walia et al., 2010). The gases detected are generally methane and ethane and the ratio of ethane/methane is used to distinguish from methane that is produced from decay of organic matter. Other gases that can be collected and analyzed include hydrogen sulfide, radon, mercury, helium, oxygen, carbon dioxide, argon, nitrogen, and carbon monoxide. The gases are measured using a gas chromatograph.

PRIMARY USES: Locate faults, especially in seismically active areas. Locate areas of high hydrogen sulfide and other gases that could affect water quality. Can be used to characterize the soil environment after reclamation (Jacinthe and Lal, 2006). Can be used to locate underground workings (Sibley, 1992).

LIMITATIONS: It is difficult to measure soil gas in impermeable soils and wet soils. Variations in sampling protocols make it difficult to compare regional soil gas data unless the same protocols are used. Because soil gases are not homogeneous, soil gas analysis can change with a variety of factors resulting in spatial and vertical variations.

INTERFERENCES: It is important to characterize the site and the methodology, especially lithology, sample spacing, sample depth, type of sample collection, emplacement methods, duration of sample collection, and decontamination procedures. Soil parameters, including density, organic content, soil moisture, permeability, porosity, clay content, and grain size, should be described. All of these parameters can affect the soil gas measurement.

SCOPE: Three types of methods are used: active, passive, and flux chambers. Active methods allow for real time measurement of soil gases. Passive soil gas methods involve

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the burial of a collection device into the soil for a period of time and the subsequent retrieval and measurement. Passive methods are generally used as screening measurements. Flux chambers provide a direct measurement of the soil flux at a specific location.

DATA FORMAT: Be careful of units. Analyses measured in ppb are not the same as ug/L or ug/m^3 because the conversion depends on the molecular weight of the compound.

BIBLIOGRAPHY

American Society for Testing and Materials (ASTM), 2006, Standard Guide for Soil Gas Monitoring in the Vadose Zone: ASTM Standard D5314-92 (2006), January 1993; Reapproved 2006; website http://www.astm.org/Standards/D5314.htm, accessed 8/23/13.

Bailey, E.A., Gray, J.E., and Theodorakos, P.M., 2002, Mercury in vegetation and soils at abandoned mercury mines in southwestern Alaska, USA: Geochemistry: Exploration, Environment, Analysis, v.2. p. 275-285.

California Environmental Protection Agency, 2012, Advisory—active soil gas investigations: http://www.dtsc.ca.gov/SiteCleanup/upload/VI_ActiveSoilGasAdvisory_FINAL_043012.pdf, accessed 8/23/13.

Duddridge, G., Grainger, P., and Durrance, E., 1991, Fault detection using soil gas geochemistry: Quarterly Journal of Engineering Geology, v. 24, p. 427-433.

Eijkelkamp, undated, Soil gas sampling and analyses: http://www.surechem.com.my/download/eijkelkamp/P1/P1-65e.pdf, accessed 12/13/10.

Fountain, J. C. and Jacobi, R. D., 2000, Detection of buried faults and fractures using soil gas analysis: Environmental Geology, v. 6, p. 201-208.

Fu, C., Yang, T.F., Walia, V., and Chen, C., 2005, Reconnaissance of soil gas composition over the buried fault and fracture zone in southern Taiwan: Geochemical Journal, v. 39, p. 427-439.

Jacinthe, P.A., and Lal, R., 2006, Spatial variability of soil properties and trace gas fluxes in reclaimed mine land of southeastern Ohio: Geoderma, v. 136, p. 598-608.

Sibley, R.D., 1992, Gas migration studies at a disused mine to detect possible rock fracture patterns and the extent of underground workings: Quarterly Journal of Engineering Geology and Hydrogeology, v.25, p. 47-56.

Tasse, N.M., Germain, D., and Bergeron, M., 1994, Impact of seasonal variations on the geochemistry of pore waters and pore gases of acid mine tailings; a field study; *in* Proceedings of the International land reclamation and mine drainage conference and Third international conference on The abatement of acidic drainage; Volume 2 of 4; Mine drainage: U.S. Bureau of Mines, Special Publication p. 426.

U.S. Environmental Protection Agency, 2010, Soil Gas Sampling: Operating procedure, SESDPROC-307-R2, http://www.epa.gov/region04/sesd/fbqstp/Soil-Gas-Sampling.pdf, accessed 8/23/13.

Walia, V., Lin, S.J., Fu, C.C., Yang, T.F., Hong, W., Wen, K., and Chen, C., 2010, Soil-gas monitoring: A tool for fault delineation studies along Hsinhua fault (Tainan), southern Taiwan: Applied Geochemistry, v. 25, p. 60-607.

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Stable Isotopes By Mass Spectrometry

(Virginia T. McLemore)

PRINCIPLE: A mass spectrometer separates ions on the basis of their masses that are dependent upon their motions in a magnetic and/or electric field. Stable isotopes are nonradioactive isotopes that are stable in the environment with respect to the element. There are approximately 300 stable isotopes in nature. However, only a few are useful in studying geologic materials (H, C, O, N, S and to a lesser extent Se, Si, B, Cl, Ca). Stable isotopic geochemistry is an important tool in environmental sciences because isotopes of the same element are sometimes slightly fractionated during physical and chemical processes occurring in nature. The fractionation is due to slight variations in the physical and chemical properties of the isotopes and is proportional to differences in their isotopic masses. Therefore, stable isotopes can be used to identify sources and identify the chemical processes involved, among other uses. The basic formula in stable isotope geochemistry is

$$\delta^{13}C = \left(\frac{\left(\frac{^{13}C}{^{12}C}\right)_{sample}}{\left(\frac{^{13}C}{^{12}C}\right)_{standard}} - 1 \right) * 1000\ ‰$$

Where C is the concentration of the element carbon and the ratio δ is measured in per mil or parts per thousands.

MEDIA: gas, water, solid, organic materials

PRIMARY USES: Stable isotope geochemistry can be used to identify the source of various solids, liquids, and gases. In addition, by combining isotopic data of different isotopes, such as S and C, other chemical parameters can be measured, such as pH and oxygen fugacity.

LIMITATIONS: Uses a small sample size, so sample collection and preparation is vital.

INTERFERENCES: Varies.

DETECTION LIMITS: Varies.

BIBLIOGRAPHY

Cravotta, C.A., III, 1995, Use of stable isotopes of carbon, nitrogen, and sulfur to identify sources of nitrogen in surface waters in the Lower Susquehanna River Basin, Pennsylvania: U.S. Geological Survey Open-File Report 94-510, 103 p.

Drever, James (2002). *The Geochemistry of Natural Waters*. New Jersey: Prentice Hall. pp. 311–322. ISBN 0-13-272790-0.

Faure, G., 1986, Principles of isotope geology: John Wiley and Sons, Inc., New York, New York, 589 p.

Fritz, P., Basharmal, G.M., Drimmie, R.J., Ibsen, J., and Qureshi, R.M., 1989, Oxygen isotope exchange between sulfate and water during bacterial reduction of sulfate: Chemical Geology, v. 79, p. 99-105.

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Fry, B., Ruf, W., Gest, H., and Hayes, J.M., 1988, Sulfur isotope effects associated with oxidation of sulfide by O2 in aqueous solution: Chemical Geology, v. 73, p. 205-210.

Hoefs, J., 1987, Stable isotope geochemistry: Springer-Verlag, Berlin, Germany, 241 p.

Kyser, T. K., ed., 1987, Short course in stable isotope geochemistry of low temperature fluids: Mineralogical Society of Canada, Short Course Handbook, v. 13, 452 p.

Nelson, S. and Dettman, D., 2001, Improving hydrogen isotope ratio measurements for on-line chromium reduction systems, Rapid Communications in Mass Spectrometry; 15: 2301-2306

Rye, R.O. and Alpers, C.N., 1997. The stable isotope geochemistry of jarosite. U.S. Geol. Surv. Open-file Rept. 97-88.

Sharp Z., 2006. Principles of Stable Isotope Geochemistry: Prentice Hall

Taylor B E, Wheeler M C and Nordstrom D K. 1984. Stable isotope geochemistry of acid mine drainage: Experimental oxidation of pyrite. Geochim. et Cosmochim. Acta 48: 2,669-2,678.

Taylor, B.E., and Wheeler, M.C., 1994, Sulfur- and oxygen-isotope geochemistry of acid mine drainage in the western United States, in Alpers C.N., and Blowes, D.W., eds., Environmental geochemistry of sulfide oxidation: Washington, D.C. American Chemical Society Symposium Series 550, p. 481-514.

Taylor, B.E., Wheeler, M.C., and Nordstrom, D.K., 1984, Oxygen and sulfur isotope compositions of sulfate in acid mine drainage--evidence for oxidation mechanisms. Nature 308:538-541.

U.S. Geological Survey, 1999, Stable Isotopes and Mineral Resource Investigations in the United States: http://pubs.usgs.gov/info/seal2/, accessed 8/23/13.

Wasserman, M.D., Rye, R.O., Bethke, P.M., and Arribas, Jr., A., 1992, Methods for the separation and total stable isotope analysis of alunite: United States Geological Survey, Open-File Report 92-9.

X-Ray Diffraction Analysis (XRD)

(Virginia T. McLemore)

DEFINITIONS: Radiation—energy in transit, either as particles or electromagnetic waves

Radioactivity—characteristic of materials to emit ionizing radiation

Ionization—the removal of electrons from an atom

PRINCIPLE: Diffraction occurs as X-rays of a specific wavelength interact with crystalline solids. X-rays are electromagnetic radiation of wavelength about 1 Å (10^{-10} m), which is about the same size as atoms and inter-atomic spacings. They occur in that portion of the electromagnetic spectrum between gamma rays and the ultraviolet. Each crystalline solid has its unique characteristic X-ray powder pattern, which may be used as a "fingerprint" for its identification. Once the material has been identified, X-ray crystallography may be used to determine its structure, i.e. how the atoms pack together in the crystalline state and what the interatomic distance and angle are etc. X-ray diffraction can determine the size and the shape of the unit cell for any compound using the diffraction of X-rays of a specific wavelength.

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PRIMARY USES: X-ray diffraction has been in use in two main areas, for the fingerprint characterization of crystalline materials and the determination of their structure. XRD analyses are used to identify minerals in solid media and require relatively simple sample preparation techniques.

SAMPLE PREPARATION: Sample can be solid or liquid. Solid sample must be crushed to powder. Thin sections can be used.

LIMITATIONS: To obtain an x-ray diffraction pattern, one needs a crystalline phase in an abundance above 1 %.

INTERFERENCES: Overlapping peaks from multi-minerals may produce interferences. Quantitative mineral abundance determination by whole rock X-ray diffraction analysis can be difficult, because the effects of mineral crystallinity, preferential orientation in the sample mount, differential absorption of X-rays, and overlapping peaks by different minerals, affect the diffractogram patterns and make their interpretation difficult. For example, some minerals, such as hematite and other iron oxides, have poor crystallinity and are not always detected by X-ray diffraction.

SCOPE: X-ray diffraction analysis requires an X-ray source, the sample under investigation and a detector to pick up the diffracted X-rays. A diffractometer can be used to make a diffraction pattern of any crystalline solid. With a diffraction pattern an investigator can identify an unknown mineral, or characterize the atomic-scale structure of an already identified mineral. The X-ray radiation most commonly used is that emitted by copper, whose characteristic wavelength for the K radiation is =1.5418Å.

COST: The cost involves instrument time, interpretation, and can cost several hundred dollars per sample.

BIBLIOGRAPHY

Azaroff, L. V. and Buerger, M. J., 1958, The Powder Method in X-ray Crystallography: McGraw-Hill Book Co.

Bertin, E. P., 1975, Principles and practice of X-ray spectrometric analysis: Plenum Press, New York, 1079 p.

Buhrke, V. E., Jenkins, R., and Smith, D. K., 1998, Preparation of specimens for X-ray fluorescence and X-ray diffraction analysis: John Wiley and Sons, Inc., New York.

Cullity, B. D., 1978, Elements of X-ray Diffraction: Addison-Wesley Publishing Company, Inc.

Jenkins, R. and Snyder, R. L., 1996, Introduction to X-ray Powder Diffractometry: vol. 138, Wiley Interscience, John Wiley and Sons, New York.

Klug, H. P. and Alexander, L. E., 1974, X-ray diffraction procedures for polycrystalline and amorphous materials: Wiley Interscience, John Wiley and Sons, New York, 2nd Edition.

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X-Ray Fluorescence Analysis (XRF)

(Virginia T. McLemore)

PRINCIPLE: Samples are excited by X-ray spectrum emitted by an X-ray tube, which causes ionization of inner-shell electrons. When a primary excitation source from an X-ray tube or a radioactive source strikes a sample, the resulting radiation can be either absorbed by the atom or scattered through the material. The process in which an X-ray is absorbed by the atom by transferring all of its energy to an innermost electron is called the "photoelectric effect." During this process, if the primary X-ray had sufficient energy, electrons are ejected from the inner shells, creating instabilities in the atom, called vacancies. The excited atom is unstable and decays by an electron moving into the vacant spot in the inner shell. A loss of excess energy results in the emission of an X-ray photon, which has a characteristic energy for that specific element. Because each element has a unique set of energy levels, each element produces X-rays at a unique set of energies, allowing one to measure the elemental composition of a sample. The process of emissions of characteristic X-rays is called X-ray fluorescence (XRF). Analysis using X-ray fluorescence is called X-ray Fluorescence Spectroscopy. In most cases the innermost K and L shells are involved in XRF detection. A typical X-ray spectrum from an irradiated sample will display multiple peaks of different intensities, which are related to its elemental composition. There are two types of XRF instrumentation: energy dispersive (EDXRF) and wavelength dispersive (WDXRF) spectrometers.

PRIMARY USES: XRF analyses are performed on solid media and require relatively simple sample preparation techniques.

SAMPLE PREPARATION: Solid sample must be crushed to powder. Major elements are typically determined on powders that have been fused with a flux and cast as discs. Trace elements are typically determined on compressed powder pellets.

LIMITATIONS: XRF is applicable for solid media only.

INTERFERENCES: XRF interferences can result from spectral interferences, spectral overlap, background shifts, matrix absorption and enhancement effects in the sample, and sample inhomogeneity. Matching of standard and sample matrices can minimize matrix interferences. Highly mineralized samples, especially lead, cause analytical interferences, which are difficult to correct for.

SCOPE: XRF is a nondestructive technique used for quantitative analysis of bulk elemental composition and trace analysis (sensitivity) to ppm levels. It can be used to detect metallic surface contamination on polymers. Portable EDXRF units provide acceptable field measurements.

DETECTION LIMITS: Detection limits differ by element and are generally in the 1-5 ppm range for WDXRF units. Detection limits are higher for EDXRF units.

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BIBLIOGRAPHY

Azaroff, L. V. and Buerger, M. J., 1958, The powder method in X-ray crystallography: McGraw-Hill Book Co.

Bertin, E. P., 1975, Principles and practice of X-ray spectrometric analysis: Plenum Press, New York, 1079 p.

Buhrke, V. E., Jenkins, R., and Smith, D. K., 1998, Preparation of specimens for X-ray fluorescence and X-ray diffraction analysis: John Wiley & Sons, Inc., New York.

Potts, P. J., 1993, Laboratory methods of analysis; *in* Riddle, C., ed., Analysis of geological materials: Marcel Dekker, Inc., New York, New York, p. 123-220.

Riddle, C., ed., 1993, Analysis of geological materials: Marcel Dekker, Inc., New York, 67 p.

Taggart, J. E., Jr., Lindsay, J. R., Scott, B. A., Vivit, D. V., Bartel, A. J., and Stewart, K. C., 1987, Analysis of geologic materials by wavelength-dispersive X-ray fluorescence spectrometry; *in* Baedecker, P. ed., Methods for geochemical analysis: U. S. Geological Survey, Bulletin 1770, p. E1-E19.

Field Portable X-ray Fluorescence (FPXRF) Instruments

(Richard K. Glanzman)

MEDIA: Solids

PRINCIPLE: This method involves the use of a handheld instrument weighing about three pounds to analyze most of the elements in the periodic table. FPXRF instruments use the same x-ray fluorescence spectroscopy as the laboratory instruments. Two excitation methods are available: radioisotopes and miniaturized x-ray tubes. Almost all current instruments use the x-ray tube.

X-rays from the instrument impinging on a sample excite the individual atoms within the sample. The FPXRF quantitatively determines the element-specific photons dominantly emitted from the K and L shell electrons. The emitted photons in counts per second (cps) are directly proportional to the amount of that element in the sample. The counts are converted to concentrations in parts per million or percentages by two computer software programs within the FPXRF for approximately 30 elements but the detected peaks for all elements are saved in a separate spectrum file. Therefore, there are two files stored for every sample analysis.

PRIMARY USES: The FPXRF is primarily used for the inorganic analysis of solid media such as powders, soils, rocks, stream sediments, construction debris, etc. Analysis does not require sample preparation but some preparation may be selectively desirable. Specific FPXRF analytical methods include metal alloys, lead in paint, and plastics. It can be used on any media as a screening tool. FPXRF instruments usually have about a one centimeter diameter window of analysis but newer instruments equipped with a camera can analyze a spot only a few millimeters in diameter. Its dominant strength is the capability of taking the FPXRF to the sample instead of the more conventional method of taking the sample to the laboratory. However, when sample preparation such as drying, sieving or crushing/grinding are considered, the FPXRF may be set up at an onsite location for chemical analysis. Potts and West (2008) and Jenkins (1998) provide the principles of, and operational guides for, XRF and FPXRF analysis.

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FPXRF instruments are very user friendly and can be operated by non-technical personnel with about half-hour training but for credible chemical analysis the operator should have a basic understanding of x-ray fluorescence limitations and interferences. In effect, the operator becomes an analyst. USEPA Method 6200 protocol is recommended reading for both those planning to use the FPXRF and those operating the FPXRF. USEPA Method 6200 is readily available from any internet search software.

LIMITATIONS: FPXRF limitations involve light elements, calibration, moisture content and particle size. Most FPXRF instruments are capable of analyzing elements as low as sulfur, commonly as low as potassium but lighter elements are more difficult. Newer instruments are capable of determining elements as light as magnesium but lighter elements may be possible in the future.

Instruments can be procured for specific purposes (modes) and calibrated for specific elements for each purpose. Most instruments have been calibrated for trace element analysis of about 30 elements on dry soils with relatively low organic content. Therefore, visible organic matter like twigs and leaves should be removed from the sample prior to analysis. This can most easily be done by coarse sieving. However, highly organic material (greater than about 10 percent organic carbon) and particularly peat decrease the reported elemental concentration in proportion to the organic carbon content. The content can be monitored and corrected by the Compton backscatter peak height in the sample spectrum. The trace element (soils) instrument mode is usually capable of determining elemental concentrations up to about three to five percent. The bulk mode determines concentrations from about this range to 100 percent. Other instrument calibration modes can include plastics (organic and density issues), metal alloys (analysis and alloy identification) and lead in paint (surface and near surface area concentration). Instruments can be designed for other specific purposes by contacting manufacturers.

Similar to organic material, moisture content of more than 15 percent or when water is seeping from the sample also decreases the reported trace element concentrations in proportion to the excessive moisture content. The Compton backscatter peak in the sample spectrum is the best way to monitor the moisture content and can be used to correct for excessive moisture content. The drier the sample is, the better the chemical analysis. Air or low temperature drying is recommended where drying is deemed appropriate to minimize vaporization of the more volatile elements.

Finally, a broad range and larger particle sizes create a heterogeneity problem. A fine-grained (less than about 100 mesh) grain size involving a single mineralogy is ideal, but generally these are not the characteristics of the samples being analyzed. Presenting a dry, homogenized, coarse sand grain size or less for analysis through the one centimeter FPXRF window can be both precise and accurate. Pebble and larger sized particles frequently have a sufficient mineralogical heterogeneity that both precision and accuracy with a laboratory confirmation sample can be highly variable. Coarse sand size sieving of the sample is the easiest method of removing coarse grained particulates and promoting homogeneity. Confirmation samples sent to the laboratory should specify homogenization of the sample during sample preparation and prior to weighing the sample for digestion. A FPXRF analyzed XRF sample cup of a particulate sample is recommended for sending a confirmation sample to the laboratory.

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INTERFERENCES: There are many potential elemental energy levels that overlap and are both best monitored and clearly shown on the FPXRF spectrum screen. For example, the major arsenic peak and the major lead peak overlap one another so the FPXRF software compensates for this by using secondary peaks to estimate the arsenic and lead concentrations. This is typically successful until the lead concentration is greater than about 10 times the arsenic concentration. This problem can usually be further corrected by using the laboratory confirmation analyses to construct a linear least square fit equation for the FPXRF data. Another potentially more serious problem is the secondary iron peak overlapping the primary cobalt peak. It is unlikely that cobalt concentrations can be determined on samples containing iron even as a principal trace element. Other interferences occur and the spectral database needs to be included in the downloaded FPXRF database along with the Excel spreadsheet of analytical values and their error estimate.

SCOPE: Almost all FPXRF instruments come equipped to analyze trace elements (less than 3 to 5 percent) using the Compton Normalization software. This software uses the Compton backscatter peak as a measure of matrix composition. As described in the above sections, elevated moisture and organic content can significantly affect this peak. Elemental concentrations above about 2 to 3 percent to 100 percent are calculated using the Fundamental Parameter software. The specific sample characteristics that are to be analyzed by the FPXRF need to be discussed with the instrument manufacturer/supplier to fit the FPXRF for the intended sample analysis.

DETECTION LIMITS: Detection limits for chemical elements above titanium in the periodic table range from less than 10 to about 100 ppm. Detection limits for lighter elements like magnesium to titanium range from about tenths of percent to about 100 ppm. Detection limits and both precision and accuracy improve with longer counting times. Screening level analytical work can involve a 10 to 20 second counting time whereas quantitative work may require 100 to 200 seconds. Little is gained by counting longer than 200 seconds.

DATA FORMAT: The mean and standard deviation of the elemental concentrations being analyzed can be observed on the FPXRF view screen as the sample is being analyzed. This is useful for determining the appropriate counting time. The elemental peaks for all the detected elements can also be observed on the FPXRF view screen as the sample is being analyzed. Either screen can also be viewed at the end of the sample analysis. The analytical data and peak heights of all detected elements (spectrum) are both stored for each sample analysis. Instruments are capable of storing tens of thousands of sample analyses. Either file for any previous sample can also be viewed on the FPXRF view screen. Both the analytical data and spectra for all the sample analyses can be downloaded in excel spreadsheet form. In addition the spectra for all samples can be downloaded into a searchable and peak identifiable file for visually inspecting for elemental interferences.

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BIBLIOGRAPHY:

Glanzman, R.K. 2010. *Application of Field Portable X-Ray Fluorescence to the Mining Industry*. Unpublished Ph.D. dissertation, Colorado School of Mines, 125 p.

Jenkins, R. 1999. *X-Ray Fluorescence Spectrometry*, Second Ed. New York: John Wiley & Sons, Inc., 207 p.

Potts, P.J., and West, M. 2008. *Portable X-ray Fluorescence Spectrometry, Capabilities for In Situ Analysis*. Cambridge: RSC Publishing, The Royal Society of Chemistry. ISBN: 978-0-85404-552-5, 291 p.

USEPA (U.S. Environmental Protection Agency). 2007. Field Portable X-Ray *Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment*. Test Methods SW846, EPA Method 6200-1 Revision 0, February, 2007, 32 p.

GEOPHYSICAL AND REMOTE SENSING TECHNIQUES

Overview

(Bruce D. Smith)

Geophysical methods can be used at different scales of geoenvironmental investigations of abandoned mine lands ranging from dump sites (centimeters) watersheds (kilometers). Applications at these different scales are illustrated by studies from Animas watershed in Colorado (Church and others, 2007) and in the Boulder watershed, Montana, (Nimick and others 2004). In general geophysical surveys can be carried out by airborne, ground, and borehole methods. Usually airborne surveys are done at watershed scales whereas ground and borehole are applied at the site scale. The application of geophysical methods depends on mapping contrasts in physical property of rocks. Hoover, Klein, and Campbell (2000) outlined physical properties and depth of investigation for various geophysical methods that can be applied to mining environmental investigations given in Table A3.16.

Standard text books on near surface geophysics are given in the below Bibliography. Geophysical applications generally focus on near surface methods which map contrasts in physical properties to depths of a few kilometers at most. Mainly applications are within a few hundred meters. An important class of applications is to groundwater and hydrologic studies which include acid mine drainage. Other geophysical applications are to engineering and infrastructure issues associated with mining environmental problems. Geophysical case histories of applications in mapping contaminants from a variety of sources demonstrate possible applications in mining environmental studies.

Survey costs are difficult to estimate for particular methods since scale of applications is an important aspect of costs. Figure A3.9 shows a generalization of costs for electrical and electromagnetic geophysical surveys. In general geophysical measurements are recorded digitally and processed. General cost of instrumentation for ground surveys has been provided in order to provide some basis for costs. One can estimate a rental or use cost based on the instrumentation price of say 5% per week of rental. Post processing and report writing generally should be at least twice the time to acquire the data. Again this depends on the depth required for interpretation.

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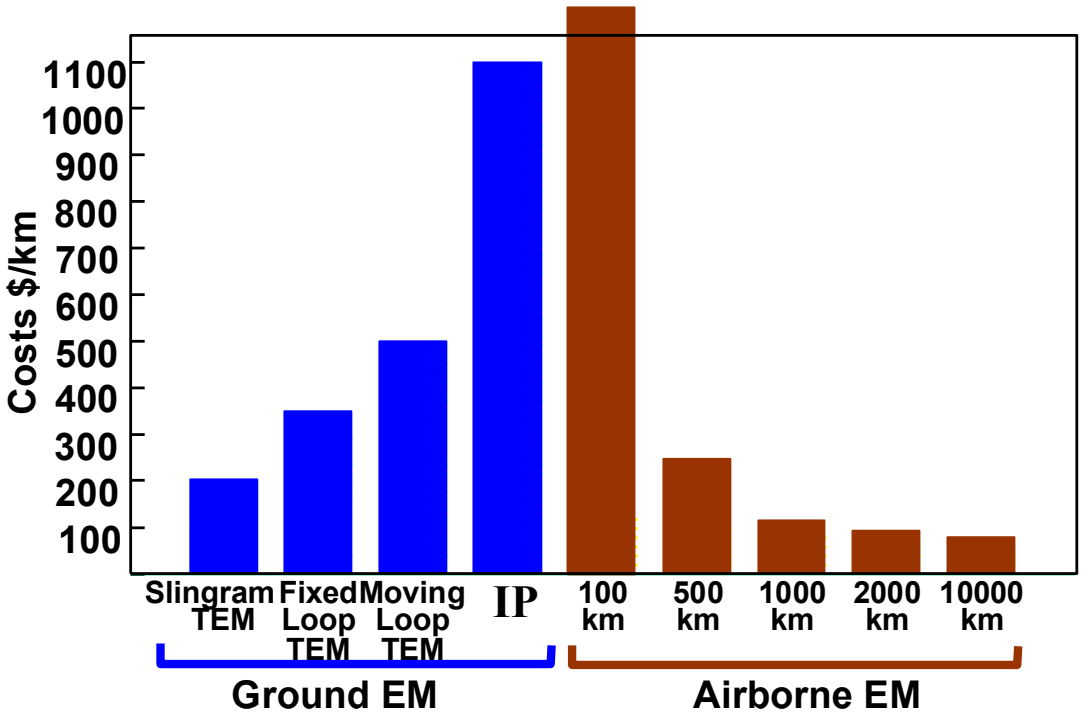


FIGURE A3.9 Generalized comparative costs for ground and airborne electromagnetic (EM) surveys (Greg Hodges, written communication, 2009).

BIBLIOGRAPHY:

American Society for Testing and Materials, 1999. Standard Guide for Selecting Surface Geophysical Methods, ASTM D6429-99, West Conshohocken, Pennsylvania.

Butler, D.K. (ed.), 2005, Near- Geophysics, Society of Exploration Geophysicists, Monograph 5, 752p.

Campbell, D.L., Horton, R.J., Bisdorf, R.J., Fey, D.L., Powers, M.H., and Fitterman, D.V., 1999, Some geophysical methods for tailings/mine waste work, Tailings and Mine Waste '99: Proc. of 6th Int'l Conf.: Rotterdam, A.A. Balkema, p.35-43.

Church, S.E., von Guerard, P., and Finger, S.E. (eds.), 2007, Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado, U.S. Geological Survey Professional Paper 1651, 1096 p., 6 plates, 1 DVD. Available online at : http://pubs.usgs.gov/pp/1651/.

Keys, S.W., 1997, A Practical Guide to Borehole Geophysics in Environmental Investigations: CRC Press, Boca Raton, Florida, 171p.

Kirsch, R. (editor), 2005, Groundwater Geophysics: A Tool for Hydrogeology, Springer, New York, 491.

Hoover, D.B., Klein, D.P., and Campbell, D.L., 2000, Geophysical Methods in Exploration and Mineral Environmental Investigations, Chapter 3, *in* USGS mineral deposit models, eds., D.B. Stoeser and W.D. Heran, USGS Digital Data Series 64.

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Milson, J., 2003, Field Geophysics: John Wiley & Sons, Inc. Hoboken, New Jersey, 232p.

Mwenifumbo, C.M., 1993, Borehole geophysics in environmental application: Canadian Institute of Mining and Metallurgy Bulletin, v. 86, no. 966, p. 43-49

Nimick, D.A., Church , S.E., and Finger, S.E., 2004, Integrated Investigations of Environmental Effects of Historical Mining in The Basin and Boulder Mining Districts, Boulder River Watershed, Jefferson County, Montana: U.S. Geological Survey Professional Paper 1652, 524 p. Available online at : http://pubs.usgs.gov/pp/1652/

Rubin, Y. and Hubbard, S. S., Hydrogeophysics: Springer, New York, 513 p.

Sharma, P.V. and Reddy, K.R., 2004, Geoenvironmental Engineering: Site Remediation, Waste Containment, and Emerging Waste Management Technologies: John Wiley & Sons, Inc. Hoboken, New Jersey, 961p.

Ward, S.H. (ed.), 1990, Geotechnical and Environmental Geophysics (Volumes I, Review and tutorial; II Environmental and Groundwater; III Geotechnical), Society of Exploration Geophysicists, 981 p.

TABLE A3.16 Summary of geophysical methods and their characteristics as applied to mineral deposit geoenvironmental studies [in method column; A, airborne surveys, B, borehole surveys, and G, ground surveys]

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Method	Physical Parameter Measured	Typical Units	Relevant Physical Property	Typical Source of Anomaly	Depth of Investigation
Gravity: (A,B,G)	Total attraction of Earth's gravity field (the vertical attraction of anomalous masses) Gradient of Earth's Field	Milligals or gravity unit (0.1 mGal) Eotvos Unit (10^9 gal/cm)	Density	Rock density contrasts	All
Magnetic: (A,B,G)	Vector component , or total attraction of Earth's magnetic field. Gradient of Earth's magnetic field.	Nanotesla or gammas Nanotesla/m	Magnetic susceptibility and (or) remnant magnetization (same)	Magnetic susceptibility and (or) remnant magnetization contrasts (same)	Surface to Curie isotherm (loss of magnetization)
Gamma-ray Scintillometery : (A,B,G) Gamma-ray Spectrometry: (A,B,G)	Rate of gamma-ray photons received Rate of gamma-ray photons received and their energy	Counts per second Counts/sec in spectral regions. If calibrated %K and PPM equivalent U and Th	Quantity of K+U+Th and daughters Quantity of K,U,Th K,U, and Th	K+U+Th contrasts in Earth's upper 50 cm K,U,Th, contrasts in Earth's upper 50 cm	Upper 50 cm
Seismic refraction (B,G) Seismic reflection (B,G)	Seismic energy and travel time	Meters, milliseconds	Velocity of P or S waves	Structures, or velocity layer contracts	All
Thermal borehole (B) Thermal Remote Sensing (A, G)	Thermal gradient or temperature Surface temperature day and night	Degrees C/m, degrees Degrees C	Thermal Conductivity Thermal Inertia	Thermal flux or conductivity variations Thermal inertial contrasts	Hole depth About 5 cm
Electrical , direct current (B,G)	Electrode position (m), current (A), and electric field (mV)	Meter, amps, millivolts; typically converted to units of resistivity (Ohm-m)	Resistivity	Lateral or vertical changes in resistivity	About 2 km
Mise-a-la-masse (B,G)	Applied DC or low frequency AC field	Millivolts	Resistivity	Conductive body	Few hundred meters
Induced Polarization (B,G)	Resistivity change w/frequency (PFE), phase angle between transmitted and received signal, Normalized part of received voltage decay waveform (Chargeability)	Percentage change or chargeability	Interface ionic polarization, electron transfer	Metallic luster mineralization, high ion exchange clays, redox potential, and pore water	About 2 km
Electromagnetic (A,B,G) many variations of induction	Dependent on method; ratio of received (secondary) to applied (primary) electric and magnetic	Impedance (ohms) or dimensionless ration; units of conductivity	Electrical conductivity (inverse of resistivity	Lateral or vertical changes in Earth electrical properties	Shallow (<1m) using controlled sources; deep

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instrumentation	fields in frequency or time domain; natural field	(Siemens/m) or resistivity (Ohm-m)			(>10km) using natural fields
Self potential (B,G)	Natural near-static (direct current) electric field	Millivolts	Eh/pH electronic conductor; steaming-potential and thermal coupling coefficients	Vertical change in Eh/pH caused by electronic conductor, groundwater flow; thermal flux	A few hundred meters
Nuclear Magnetic Resonance (B,G)	Spin decay of water molecules	Seconds, milliseconds	Free and bound water	Changes in porosity, amount of water,	<200 m

Source: adapted from Hoover and others (2000).

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Gravity Methods

(Bruce D. Smith)

PRINCIPLE: Gravity measurements define anomalous density within the Earth; in most cases, ground-based gravimeters are used to precisely measure variations in the gravity field at different points. Gravity anomalies are computed by subtracting a regional field from the measured field, which result in gravitational anomalies that correlate with source body density variations. Positive gravity anomalies are associated with shallow high density bodies, whereas gravity lows are associated with shallow low density bodies. The gravity method also enables a prediction of the total anomalous mass responsible for an anomaly. Gravity methods detect only lateral contrasts in density. Applications of gravity to environmental considerations related to mineral deposit development include identification of lithologies, structures, and, at times, ore bodies themselves (Wright, 1981). Small anomalous bodies, such as underground workings, are not easily detected by gravity surveys unless they are at shallow depth. In recent years gravimeters have increased in precision which has enabled greater use of microgravity measurements. A microgravity survey provides a measure of change in subsurface density (ASTM D6430-99). Natural variations in subsurface density include lateral changes in soil or rock density, buried channels, large fractures, faults, dissolution-enlarged joints and cavities. The microgravity method comes closest of all the geophysical methods for allowing a positive statement to be made regarding the presence or absence of subsurface cavities at a site (Butler, 1984). A microgravity survey consists of making sensitive gravity measurements at the microGal (μGal) level (1/1000 of a milliGal or 10-9f the Earth's gravitational field) with a gravimeter. Gravity measurements are acquired at discrete points along a profile line or within a grid, and are corrected for instrument drift, tidal effects, elevation changes, and latitude. Recent advances in gravity surveying include a number of airborne applications summarized in a recent workshop (Lane, 2004).

MEDIA: density of rocks and soil.

PRIMARY USES:

- **Watershed scale hydrogeology** – mapping faults, structure, and hydrostratigraphy. Mapping hydrothermal alteration. Detection of ore deposits (such as massive sulfide deposits) that have a high density relative to host rocks.
- **Mapping dump or site scale features** - investigations of dump density variations and volume where sufficient density contrast exists.
- **Detection of Mine voids and tunnels –** shallow mine tunnels detected by high precision microgravity.

LIMITATIONS: Data acquisition is relatively slow compared with other geophysical methods; Irregular topography will produce artifacts in the data unless accounted for in the processing;

INTERFERENCES: local sources of vibrations, wind, storms, and distant earthquakes can produce interference.

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DETECTION LIMITS/AERIAL RESOLUTION: Standard gravity surveys
DATA FORMAT: Digital records.

COST: most surveys can be done by one person with perhaps high precision GPS locations and elevations. High precision meters can cost in excess of $100,000.

BIBLIOGRAPHY:

Butler, D. K, 1980, Microgravimetric techniques for geotechnical applications, Miscellaneous Paper GL-80-13: U.S. Army Engineer Waterways Experiment Station, Vicksburg, MS, 121pp.

Benson, R.C., Kaufman, R.D., Yuhr, L., and Hopkins, R., 2003, Locating and characterizing abandoned mines using microgravity; Proceedings, Geophysical Technologies for Detecting Underground Coal Mine Voids Forum, July 28-30, 2003 – Lexington, Kentucky, Federal Highway Administration publication, p1-9.

Lane, R., 2004, Airborne Gravity 2004 workshop proceedings; Geoscience Australia, available http://www.ga.gov.au/about/corporate/ga_authors/index_2004_18.jsp, accessed December 2009.

Webring, M.W., Kucks, R.P., and Abraham, J.D., 2004, Gravity study of the Guernsey landfill site, Guernsey, Wyoming; U.S. Geological Survey Open-File Report 2004-1383, 34 p.

Wright, P.M., 1981, Gravity and magnetic methods in mineral exploration, in Skinner, B.J., ed., Economic Geology, 75th Anniversary volume, p. 829-839.

Magnetic Methods

(Bruce D. Smith)

PRINCIPLE: There are two primary applications for magnetic measurements: (1) locating and mapping buried ferrous metals, and (2) mapping geologic structures.

Location and Mapping of Buried Ferrous Metals; magnetic measurements can be used for locating and mapping buried ferrous metals (e.g. waste, drums or underground structures and utilities). The presence of buried ferrous metals creates a local variation in the strength of the earth's magnetic field, permitting the detection and mapping of buried ferrous metal.

Total field measurements (made with one magnetometer) and gradient measurements (made with two magnetometers) are commonly used. Magnetic gradient measurements are made by a gradiometer, which is simply two magnetic sensors separated by a constant offset. Magnetic gradient measurements are often used to enhance the anomalies produced by shallow buried objects.

Mapping Geologic Structures; Magnetic measurements can be used for geologic mapping by responding to the magnetic susceptibility of soil and rock. Generally total field measurements are used for geologic mapping. The primary application is for mineral exploration and for characterizing geologic structure such as faults.

MEDIA: Digital Data.

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PRIMARY USES:

- **Watershed scale hydrogeology** – mapping faults, structure, and hydrostratigraphy. Mapping hydrothermal alteration. Detection of ore deposits (such as massive iron formations) that have a high magnetization relative to host rocks.
- **Mapping dump or site scale features** - investigations of dump compositional variations and volume where sufficient magnetization contrast exists.

LIMITATIONS: Gradient measurements are less sensitive to deeper objects than total field measurements. total field measurements susceptible to natural fluctuations in the Earth's magnetic field (a base station must be used to remove natural fluctuations in Earth's field).

INTERFERENCES: Magnetic measurements are susceptible to interference from steel pipes, fences, vehicles and buildings. Solar flares produce intense magnetic field variations.

DETECTION LIMITS/AERIAL RESOLUTION: Standard magnetic surveys can be carried out by one person or by a vehicle with a magnetometer mount. Airborne surveys are carried out with variable line spacing depending on the target to be mapped.

DATA FORMAT: Digital records.

COST: most ground surveys can be done by one person. Instrumentation costs are from $20,000. Airborne surveys can be conducted for a minimum of $20,000 excluding mobilization.

BIBLIOGRAPHY:

Hansen, R. O., Grauch, V.J.S. and Racic, L., 2005, Magnetic Methods in Near-Surface Geophysics, in, *Near Surface Geophysics*, D. Butler ed., Society of Exploration Geophysics, p 151-175.

Nabighian, M.N. (ed.), 1987, Magnetic prospecting; Society of Exploration Geophysicists, 622 p.

Ground Penetrating Radar (GPR)

(Bruce D. Smith)

PRINCIPLE: Ground penetrating radar (GPR) uses high frequency electromagnetic waves to acquire subsurface information (ASTM D6432-11). Energy is radiated downward into the ground from a transmitter and is reflected back to a receiving antenna. The reflected signals are recorded and produce a continuous cross-sectional "picture" or profile of shallow subsurface conditions. Reflections of the radar wave occur where there is a change in the dielectric constant or electrical conductivity between two materials. Changes in conductivity and in dielectric properties are associated with natural hydrogeologic conditions such as bedding, cementation, moisture, clay content, voids, and fractures. Large changes in dielectric properties often exist between geologic materials and manmade structures such as buried utilities or tanks.

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The ground penetrating radar (GPR) method involves the use of a transmitter to emit high frequency electromagnetic waves into the subsurface. The transmitter is either moved slowly across the ground surface or moved at fixed station intervals. When the waves encounter an interface between materials of differing dielectric properties, they are reflected back. Travel times of the waves provide a profile of shallow conditions. Under optimum conditions, GPR data can resolve changes in soil horizons, bedrock fractures, water-insoluble contaminants, geological features, manmade buried objects, voids, and hydrogeologic features such as water table depths and wetting fronts. This method is generally less applicable for the delineation of contaminant plumes.

MEDIA: Digital records

PRIMARY USES: Ground penetrating radar is primarily used to obtain a high resolution cross-section of natural geologic, hydrogeologic, and anomalous conditions. Ground penetrating radar is also used for location and evaluation of manmade structures, including location of utilities, rebar in concrete nondestructive testing of manmade structures. Ground penetrating radar has some applications in detecting and mapping contaminant plumes, buried wastes and drums. Depth of mapping is less than 30 m and commonly less than 10 m.

LIMITATIONS: Depth of penetration is site specific; penetration in silts and clays and in materials having conductivity values above 15 to 20 mS/m is limited.

INTERFERENCES: main interference is from electrically conductive ground and active electromagnetic sources such as buried power lines.

DATA FORMAT: data are commonly presented in time or depth sections

COST: most surveys can be done by two people. Equipment costs from $30,000 and up.

BIBLIOGRAPHY:

ASTM D6432 - 11 Standard Guide for Using the Surface Ground Penetrating Radar Method for Subsurface Investigation; American Society for Testing and Materials: http://www.astm.org/Standards/D6432.htm. Accessed 8/23/13.

Ellefsen, K.J., Lucius, J.E., and Fitterman, D.V., 1998, An evaluation of several geophysical methods for characterizing sand and gravel deposits: U.S. Geological Survey Open-File Report 98-221, 26 p.

Electromagnetic Methods (can be conducted as airborne, ground, or borehole logging)

(Bruce D. Smith)

PRINCIPLE: Electromagnetic methods provide a means to measure subsurface electrical conductivity and to identify subsurface metal objects. Electrical conductivity is a function of soil and rock type, porosity and permeability, as well as the composition of fluids that fill the pore spaces (McNeill, 1990). Conductivity values may be related to groundwater properties such as specific conductance or total dissolved solids. Electrical

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conductivity values are given in units of milliSiemens/meter (mS/m). Electromagnetic conductivity values are not necessarily diagnostic in themselves; rather, it is the spatial variations in conductivity values that are significant.

Frequency domain electromagnetic (FDEM) methods measure the electrical conductivity of soil and rock by measuring the magnitude and phase of an induced electromagnetic current. Common frequency domain electromagnetic instruments provide bulk measurements of apparent conductivity values integrated over a volume of the subsurface from a few centimeters to a few hundred meters, depending on the instrumentation. They can also be used as a metal detector to locate ferrous and non-ferrous metallic objects.

Time domain electromagnetic (TDEM) methods measure the electrical conductivity of soil and rock by inducing pulsating currents in the ground with a transmitter coil and monitoring the decay of the induced current over time with a separate receiver coil.

Very Low Frequency (VLF) measurements are made by measuring the distortions of a VLF wave from a distant transmitter set up primarily for communications with submarines. Distortions of the VLF wave occur due to a local increase in electrical conductivity usually found within fractures. The increase in electrical conductivity is a function of the conductive material, such as water, clay or minerals within the fracture.

MEDIA: digital records

PRIMARY USES:

FDEM: Frequency domain electromagnetic measurements are primarily used for profiling to detect and map lateral changes in natural geologic and hydrogeologic conditions. Electromagnetic measurements are also applicable to detecting and mapping contaminant plumes. Electromagnetic measurements can be used for mapping buried wastes, metal drums, tanks, and metal utilities. The method can be used for azimuthally oriented measurements to determine fracture trends.

TDEM: Measurements are primarily used to determine depth and thickness of natural geologic and hydrologic conditions. TDEM measurements can also be applied to detection and mapping of landfill leachate plumes, seepage from brine pits, saltwater intrusion and mineral exploration.

VLF measurements are primarily used for location and mapping of near vertical contacts, fractures, and faults containing water, clay, or minerals.

LIMITATIONS AND INTERFERENCES:

FDEM: Limited vertical resolution and susceptible to interference from nearby metal pipes, cables, fences, vehicles and induced noise from power lines. Effectiveness of electromagnetic measurements decreases at very low conductivities (use resistivity).

TDEM: Deeper measurements require a large transmitter coil (1,000 x 1,000 feet or more) for which space may not be readily available. Susceptible to interference from nearby metal pipes, cables, fences, vehicles and induced noise from power lines. Effectiveness of electromagnetic measurements decreases at very low conductivities (use resistivity).

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VLF: Station measurements only. Method does not function well in horizontal layers of soil and rock with very few fractures or if soil is electrically conductive (e.g. clay-rich soils or water covered areas). Axis of fracture or electrical contrast must be oriented approximately toward the VLF transmitter. Susceptible to interference from nearby metal pipes, cables, fences, vehicles, induced noise from power lines, and loss of signal from the VLF transmitter.

DATA FORMAT: Presented as profiles and/or resistivity-depth sections.

COST: Ground geophysical instruments can cost from $10,000 to over $100,000. Airborne surveys cost more than $50,000 excluding mobilization.

BIBLIOGRAPHY:

Buselli, G. and K. Lu, 1999, Applications of some new techniques to detect groundwater contamination at mine tailings dams; Proceedings of the Symposium on the Application of Geophysics to Engineering and Environmental Problems, March 14-18, 1999, Oakland, California, p. 507-516.

Campbell, D.L., and Fitterman, D.V., 2000, Geoelectrical methods for investigating mine dumps: ICARD2000 Proceedings of the Fifth International Conference on Acid Rock Drainage, Littleton CO, Society for Mining, metallurgy, and Exploration, Inc., p. 1513-1523.

McNeill, J.D., 1990, Use of electromagnetic methods for groundwater studies, In Geotechnical and Environmental Geophysics v. I, ed. S.H. Ward, Society of Exploration Geophysicists, Tulsa OK., p 191-218.

Nabighian, M.N. (ed.), 1988, Electromagnetic Methods in Applied Geophysics, Volume 1 Theory; Society of Exploration Geophysicists, 510 p.

Nabighian, M.N. (ed.), 1988, Electromagnetic Methods in Applied Geophysics, Volume 2 Applications; Society of Exploration Geophysicists, 972 p.

Electrical Resistivity Measurements

(Bruce D. Smith)

PRINCIPLE: Electrical resistivity measurements are made by placing four electrodes in contact with the soil or rock. The electrodes can be placed in a variety of arrays. A current is caused to flow in the earth between one pair of electrodes while the voltage across the other pair of electrodes is measured. The depth of measurements is related to the electrode spacing. The resistivity measurement represents the apparent resistivity averaged over a volume of the earth determined by the soil, rock, and pore fluid resistivity, along with the electrode geometry and spacing. Resistivity measurements include profiling (with fixed electrode spacing, sounding (1D) by increasing electrode spacing at a fixed location and 2D and 3D imaging using multi-electrode arrays.

Electrical resistivity imaging (ERI) is a technique in which many individual resistivity measurements are combined to produce a 2D or 3D resistivity cross-section of the subsurface. A linear array of electrodes is set into the ground and a computer system controls which two electrodes act as the potential electrodes and which two electrodes act as the current electrodes. Measurements are acquired at various electrode separations and

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positions along the line to provide information at various lateral and vertical locations beneath the array.

Capacitively coupled (CC) resistivity measurements use the capacitance of an antenna to noninvasively couple an AC signal into the ground. Ohm-Mapper, manufactured by Geometrics, Inc., is a capacitively coupled resistivity system that is configured with coaxial cable in a dipole-dipole array. The conductors in the cable act as one plate of the capacitor and the earth acts as the other plate, with the insulating sheath as the capacitor's insulator. Since an AC signal can pass between the plates of a capacitor, the transmitter on the Ohm-Mapper system sends an AC signal (16.5 kHz) into the ground using the coaxial cable. The receiver measures the AC voltage at the transmitter frequency. This provides an AC equivalent to traditional DC resistivity measurements.

MEDIA: digital records

PRIMARY USES: Applications:

1. Determine the depth and thickness of geologic strata;
2. Detect lateral changes and locate anomalous geologic conditions;
3. Measure soil resistivity for estimating metal corrosion rates and designing grounding grids;
4. Map saltwater intrusion and contaminant plumes;
5. Locate buried wastes (e.g. locate landfill boundaries);
6. Azimuthal measurements to determine fracture orientation;
7. Create a detailed 3D resistivity model using data obtained with a grid of electrodes.

LIMITATIONS:

1. Susceptible to interference from nearby metal fences, buried pipes, cables, etc.;
2. Effectiveness decreases at very low resistivity values.

Traditional resistivity measurements use electrodes that are in direct contact with the ground to inject a DC current and to measure the resulting voltage difference. This method is effectively used in a wide variety of environments to provide resistivity soundings, profiles, and cross-sections of the subsurface. However, in areas where the surface resistivity is high and in areas where driving electrode stakes into the ground is not feasible (e.g. concrete, exposed rock, etc.), traditional resistivity measurements are not easily obtained. In these conditions, electromagnetic methods or capacitively coupled resistivity measurements could be a better choice.

INTERFERENCES: infrastructure that carries electrical current or metallic features such as culverts and fences.

DETECTION LIMITS/AERIAL RESOLUTION: mapping usually from depths of 1 meter to 10's of meters. Changes in electrical resistivity above 10,000 ohm meters or below .01 ohm meters are difficult to detect.

DATA FORMAT: data are presented as maps and or resistivity depth sections

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COST: Usually several thousand dollars per day of surveying. May cover 5 km in one day.

BIBLIOGRAPHY:

Ward, S.H., 1990, Resistivity and Induced Polarization methods, Geophysical Applications, Pergamon, London, 375 p.

Van Ziijl, J.S., 1985, A practical Manual on the Resistivity Method, Geophysics Division Natural Physics, Res. Lab., Council for Scientific and Industrial Research, South Africa, 265p.

Zohdy, A.A.R., Eaton, G.P., and Mabey, D.R., 1974, Application of Surface Geophysics to Ground-Water Investigations, Techniques of Water-Resources Investigations of the United States Geological Survey, Book 2, Chapter D1 116p.

Spontaneous Potential (SP)

(Bruce D. Smith)

PRINCIPLE: Spontaneous potential (SP), also known as self-potential, measures the natural voltage at the ground surface between two electrodes. Natural voltage differences are caused by geochemical effects or by the motion of ions (e.g. water flow).

PRIMARY USES: Assessing seepage from dams; locating fracture flow; locating caves and recharge zones (i.e. sinkholes, etc.).

LIMITATIONS: Requires intrusive contact with the ground; nonpolarizing electrodes must be used;

INTERFERENCES: susceptible to interference from cathodic protection, natural Earth currents, and cultural features (power lines, pipelines).

DETECTION LIMITS/AERIAL RESOLUTION: It can be used as a surface or subsurface (borehole) technique.

DATA FORMAT: data usually logged from voltmeter

COST: In the simplest form, the method requires a voltmeter, wire, and two non-polarizing electrodes which can be obtained for less than $1,000.

BIBLIOGRAPHY:

Corwin, R.F. 1990. The Self-Potential Method for Environmental and Engineering Applications. Geotechnical and Environmental Geophysics. Volume I: Review and Tutorial. S.H. Ward (ed.). Society of Exploration Geophysics, 28p.

Seismic Reflection and Refraction and Surface Wave Analysis

(Bruce D. Smith)

PRINCIPLE: Seismic measurements involve the measurement of seismic waves traveling through the subsurface. Stratigraphy, structure, and material properties can be

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assessed with seismic methods. Three of the most useful seismic methods for shallow applications include refraction, reflection, and surface wave analysis (multichannel analysis of surface waves; MASW).

Seismic reflection measures the travel time of seismic waves from the surface downward to geologic contacts where part of the seismic energy is reflected back to hydrophones at the surface. A reflection will occur from geologic strata when the reflection coefficient (derived from density and seismic velocity contrasts) between strata is sufficient. After the raw data are processed, a cross-sectional picture of subsurface strata and anomalous conditions can be developed.

Seismic refraction is a method to determine the P-wave velocity structure of the subsurface. Seismic P-waves are generated on the surface, propagate through the soil and rock, and are recorded by geophones at known distances from the source. When the seismic waves encounter interfaces separating material of different seismic velocities, the waves are refracted according to Snell's Law. At the critical angle for each interface (energy refracted 90 degrees) the seismic wave will travel along the interface with a velocity of the underlying layer. Since P-waves are the fastest portion of the seismic wave, they represent the first arriving energy at each geophone (either direct or refracted). A seismograph is used to record the travel-times of these first arrivals, after which seismic velocities can be derived. Depths to the refracting layers can also be determined. Note that the refraction method assumes that velocity of the layers increases with depth and that layers must be thick enough and have enough velocity contrast to be resolved.

Multichannel Analysis of Surface Waves (MASW) is a geophysical method that uses the dispersive characteristics of surface waves to determine the variation of shear wave (S-wave) velocity with depth (Park, et al., 1999). Data are acquired by measuring seismic surface waves generated by an impulsive source and received by an array of geophones. A dispersion curve is calculated from the data that shows the velocity of the surface waves as a function of frequency. A shear wave velocity profile (1-D profile of velocity as a function of depth) is then modeled from the dispersion curve. The resulting shear wave profiles from multiple measurement locations along a survey line are combined and contoured into a 2-D cross-section of shear wave velocity. Shear wave velocity is a function of the elastic properties of the soil and rock and is directly related to the hardness (N-values) and stiffness of the materials.

MEDIA: digital records

PRIMARY USES:

- Determining the depth and thickness of stratigraphic layers;
- Identifying low-velocity (weak) zones such as voids and sinkholes;
- Determining soil and rock elastic properties.

Advantages:

- Provides a cross-section of shear-wave velocity along a profile line;
- Can identify low-velocity layers below higher velocity layers;
- Data can be acquired with a "landstreamer" where the geophones are towed along the ground for fast acquisition;

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- Multiple geophones provide greater signal-to-noise ratio than more traditional SASW methods.
- Measurements can also be made in water covered areas

LIMITATIONS:
- Maximum depth limited to approximately 80 feet in typical conditions;
- Lateral resolution is typically 25 to 50% of the geophone spread length

INTERFERENCES: sources of acoustic noise such as heavy equipment. Velocity inversions (decrease with depth) mask acoustic propagation.

SCOPE: velocity contrasts

DETECTION LIMITS/AERIAL: mapping 10s of meters to 1000s of meters.

DATA FORMAT: amplitude cross sections

COST: widely variable from thousands to hundreds of thousands of dollars. Engineering studies are typically a few thousand dollars.

BIBLIOGRAPHY:
Park, C.B, Miller, R.D., and Xia, J., 1999. Multichannel analysis of surface waves, Geophysics, Vol. 64, p. 800-808.

Serif, R.E. and Geldart, L.P., 1983, Exploration Seismology: V. 1 History, Theory, and Data Acquisition, Cambridge University Press, 341 p.

Serif, R.E. and Geldart, L.P., 1983, Exploration Seismology: V. 2 Data Processing and Interpretation, Cambridge University Press, 341 p.

Borehole Geophysics

(Virginia T. McLemore)

PRINCIPLE: Geophysical logging techniques are intended to assist in the characterization of the geology and hydrogeology. At least five down-hole geophysical tools can be used: neutron, natural gamma, induction, density, and temperature. Logging must take place in a minimum of 2.0 inch diameter drill holes. Well logs and well construction details should be provided to the geophysical logging contractor to aid in the decision of what can be performed in the open boreholes and to aid in the interpretation of the geophysical logs after logging has been completed. Table A3.17 provides the geophysical probes and pertinent information.

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TABLE A3.17 Geophysical Tool Details.

Tool	Diameter (in)	Length (ft)	Radius of Investigation (in)	Use	Operating Environment
Neutron	1 or 1 11/16	7	16	Correlation, Saturation, Porosity, Lithology	Fluid filled or dry drill hole, open or cased
Natural Gamma	1 or 1 11/16	7	13	Correlation, Saturation, Porosity, Lithology	Fluid filled or dry drill hole, open or cased
Induction	1	6	25	Lithology	Fluid filled or dry drill hole, Open or PVC cased
density	1	5	N/A	Qualitative density for lithology and well completion material determination	Fluid filled or dry open or cased hole
Temperature	1	6	N/A	Drill hole temperature	Fluid filled open or cased drill hole to 300° F

MEDIA: subsurface

PRIMARY USES: see Table A3.17.

LIMITATIONS: For each geophysical tool, calibration data and scale parameters will be verified before logging begins and documented for each borehole.

INTERFERENCES: various

SCOPE: General requirements for borehole geophysical surveys are:

- all down hole equipment shall be decontaminated before and after being lowered into a borehole in accordance with Decontamination of Sampling Equipment procedures
- geophysical logging will be performed in accordance with the manufacturer's specifications
- borehole measurements shall be recorded both going into the hole and coming out of the hole
- paper copies of curves generated from each logging run shall show all the curves at an appropriate scale (e.g., 1 inch equals 20 feet), each paper log shall indicate the location of the well, date of log acquisition, type of survey instrument, and a list of other instruments used in that borehole, and interpretations shall be annotated on the margins of paper log records

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- all logs shall be referenced to ground level

DOCUMENTATION: Documentation should include:

- Names of personnel
- Weather conditions
- Date, time and location of logging
- Description of chosen geophysical technique(s)
- Description of survey set-up and data collection
- Times that procedures and measurements are completed
- Summary of data reduction and preliminary interpretations
- Documentation of all problems encountered
- Decontamination times
- Calibration information
- Other applicable information

DATA FORMAT: Geophysical data should be stored in hard-copy and electronic formats. After logging, a reproducible copy of the field strip-chart log with a heading specifying project, borehole number, location and depth, geophysical equipment types, and equipment settings will be maintained in the project file.

BIBLIOGRAPHY:

Buckley, D.K., 2000, Some case histories of geophysical downhole logging to examine borehole site and regional groundwater movement in Celtic regions; *in* Groundwater in the Celtic regions; studies in hard rock and Quaternary hydrogeology: Geological Society Special Publications, no. 182, p. 219-237.

Killeen, P.G. Mwenifumbo, C.J. and Elliott, B.E., 1995, The pseudo-geological log; using geophysical logs as an aid to geological logging in volcanogenic massive sulphides; *in* Current research 1995-E -- Recherches en cours 1995-E: Current Research - Geological Survey of Canada, p. 321-330.

ProSpecTIR VS

(Douglas C. Peters)

PRINCIPLE: ProSpecTIR-VS is a pushbroom airborne hyperspectral sensor that can be flown on any aircraft capable of having a bottom viewing port in its airframe and accommodating two to three personnel for piloting the aircraft and managing data acquisition. The sensor normally is flown in light aircraft such as single- and twin-engine fixed-wing aircraft.

PRIMARY USES:

- **Mineralogy and mineral exploration**–investigation of and exploration for mineralogical differences that can indicate alteration related to mineralization and mineral deposits and environmental conditions evidenced by characteristic mineralogy.
- **Vegetation characterization**–characterization and mapping of vegetation for growth and health characteristics and environmental impacts on vegetation.

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- **Geothermal exploration**–identification of mineralogy related to active and past geothermal activity.
- **Soils and outcrops**–compositional characterization and mapping of soils and outcrops for agricultural and geologic studies. The sensor can be mounted on a rotating table for on-ground, horizontal viewing applications.
- **Hydrocarbons**–mapping and identification of natural and manmade surface hydrocarbons both for oil and gas exploration and environmental impacts.

LIMITATIONS: Limited only by available airspace (e.g., heavy traffic area may require lower altitude operation), terrain limitations (flight safety considerations) versus ground resolution, and weather considerations during a mission.

INTERFERENCES: Normal considerations of atmospheric moisture, aerosols, and other factors that can be corrected for during data processing. Recent ground moisture (i.e., rain or snow) can interfere with spectral response from the ground as with all remote-sensing methods.

SCOPE: ProSpecTIR can be flown in any location in the world and at any desired resolution down to 0.5 m ground resolution, dependent only on terrain limitations for flight lines versus terrain (e.g., mountainous terrain can limit high-resolution collection due to flight-safety considerations).

DETECTION LIMITS/RESOLUTION (Weatherbee 2011)**:** All flight planning is accomplished using the program TOPOFLIGHT, which incorporates Digital Elevation Models (DEMs) to ensure topographic compensation and pixel sampling uniformity. Ground resolution is dependent only on terrain, flight altitude, and platform velocities. Typical ground resolution for most missions is 1 to 5 m.

Full specifications for this device and other airborne instruments are available at www.spectir.com (accessed 9/28/13). Operates in the 400 to 2450 nm wavelength region most commonly configured in a nominal ~5 nm center wavelength to center wavelength bandset. Spectral resolution under this configuration ranges from ~2.3 nm to ~5.8 nm full width half maximum (FWHM) from the visible-near infrared (VNIR) to short-wave infrared (SWIR) range.

DATA FORMAT: Delivered digital data are provided in ENVI™ readable format, which also is directly accessible by other image processing packages such as ERDAS Imagine™. The associated ASCII *.hdr files provide all the necessary file format information required to import the data into any other commercial or customized analysis platforms.

COST: Owned and operated by SpecTIR, LLC (www.spectir.com, accessed 9/28/13) out of Reno, Nevada. Costs include mobilization, demobilization, aircraft rental and insurance (if mission is outside of North America), personnel time, allowable downtime due to weather conditions, instrument usage, and data output. Cost is dependent on mission location and areal coverage of data acquisition, as well as type of product output, so there is no standard cost for this sensor.

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BIBLIOGRAPHY (see www.spectir.com (accessed 9/28/13) for additional documents and applications)**:**

Kratt, C., Coolbaugh, M., Peppin, W., and Sladek, C. 2009. Identification of a new blind geothermal system with hyperspectral remote sensing and shallow temperature measurements at Columbus Salt Marsh, Esmeralda County, Nevada. *GRC Transactions* 33:481-485.

Kratt, C., Coolbaugh, M., Peppin, B., and Sladek, C. 2009. Identification of a new blind geothermal system with hyperspectral remote sensing and shallow temperature measurements at Columbus Salt Marsh, Esmeralda County, Nevada. GRC Transactions 33:481-485.

Kuhn, F., Opperman, K., and Horig, B. 2004. Hydrocarbon index—An algorithm for hyperspectral detection of hydrocarbons. *Int. Jour. Remote Sensing* 25(12):2467-2473.

Naumann, J.C., Young, D.R., and Anderson, J.E. 2009. Spatial variations in salinity stress across a coastal landscape using vegetation indices derived from hyperspectral imagery. *Plant Ecology* 202(2):285-297.

Naumann, J., Anderson, J., and Young, D. 2008. Linking physiological responses, chlorophyll fluorescence and hyperspectral imagery to detect salinity stress using the physiological reflectance index in the coastal shrub, *Myricacerifera*. *Remote Sensing of Environ.* 112(10):3865-3875.

Santos, M., Greenberg, J., and Ustin, S. 2009. Using hyperspectral remote sensing to detect and quantify southeastern pine senescence effects in red-cockaded woodpecker (*Picoides borealis*) habitat. *Remote Sensing of Environ.* 114(6):1242-1250.

Ustin, S.H., and Santos, M.J. 2008. Analysis of hyperspectral remote sensing to detect and map tree decline at Ft. Benning, Georgia. Report for Cooperative Agreement W92DY-06-2-0023. U.S. Army Corps of Engineers.

Weatherbee, O. 2011. Written communication.

ASTER (Advanced Spaceborne Thermal Emission and Reflection Radiometer)

(Robert R. McDougal)

PRINCIPLE: ASTER is a cooperative effort between NASA and Japan's Ministry of Economy Trade and Industry (METI), with the collaboration of scientific and industry organizations in both countries. The ASTER space-borne instrument provides the next generation in remote sensing imaging capabilities compared with the older Landsat Thematic Mapper, and Japan's JERS-1 OPS scanner. ASTER captures high spatial resolution data in 14 bands, from the visible to the thermal infrared wavelengths, and provides stereo viewing capability for digital elevation model creation. As the "zoom lens" for Terra, ASTER data are used by other Terra and space-borne instruments for validation and calibration.

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PRIMARY USES:

- **Land surface climatology** - investigation of land surface parameters, surface temperature, etc., to understand land-surface interaction and energy and moisture fluxes.
- **Vegetation and ecosystem dynamics** - investigations of vegetation and soil distribution and their changes to estimate biological productivity, understand land-atmosphere interactions, and detect ecosystem change.
- **Volcano monitoring** - monitoring of eruptions and precursor events, such as gas emissions, eruption plumes, development of lava lakes, eruptive history and eruptive potential.
- **Hazard monitoring** - observation of the extent and effects of wildfires, flooding, coastal erosion, earthquake damage, and tsunami damage.
- **Hydrology** - understanding global energy and hydrologic processes and their relationship to global change; included is evapotranspiration from plants.
- **Geology and soils** - the detailed composition and geomorphologic mapping of surface soils and bedrocks to study land surface processes and earth's history.
- **Land surface and land cover change** - monitoring desertification, deforestation, urbanization; providing data for conservation managers to monitor protected areas, national parks, wilderness areas.

LIMITATIONS: Spatial and spectral resolution may be too coarse for some applications. Archived scenes can have unacceptable percent cloud cover. As of fall of 2008, the short wave infrared (SWIR) detector was no longer functioning due to anomalously high temperatures.

INTERFERENCES: Atmospheric aerosols, water vapor, and clouds are the primary interferences to ASTER imagery.

SCOPE: ASTER provides global coverage with a ground track repeat cycle of 16 days. Imagery is archived beginning in February 2000.

DETECTION LIMITS/AERIAL RESOLUTION: The ASTER instrument includes three separate spectrometers. The visible-near infrared (VNIR) instrument has a ground resolution of 15 m and covers a spectral range from 0.52 μm – 0.86 μm (three channels). The SWIR, as previously mentioned, is not operational, and there are no plans for further attempts at repair. The thermal infrared (TIR) has a ground resolution of 90 m and a spectral range of 8.125 μm – 11.65 μm (five channels).

DATA FORMAT: Digital imagery.

COST: Data are free to NASA-affiliated agencies. $85/scene for public FTP download.

BIBLIOGRAPHY:

Fujisada, H. 1994. Overview of ASTER instruments on EOS-AM1 Platform. Proceedings, SPIE, International Society for Optical Engineering. v. 2268, p. 14-36.

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Fujisada, H. 1998. ASTER Level-1 processing algorithm. *IEEE Transactions on Geoscience and Remote Sensing* 36:1101-1112.

Kahle, A., Palluconi, F., Hook, S., Realmuto, V.J., and Bothwell, G. 1991. The Advanced Spaceborne Thermal Emission and Reflectance Radiometer (ASTER). *Int. J. Imaging System Tech.* 3:144-156.

Yamaguchi, Y., Kahle, A., Tsu, H., Kawakami, T., and Pniel, M. 1998. Overview of ASTER. *IEEE Transactions on Geoscience and Remote Sensing* 36:1062–1071.

Yamaguchi, Y., Tsu, H., and Fujisada, H. 1993. Scientific basis of ASTER instrument design. Proceedings, SPIE, International Society for Optical Engineering, v. 1939, p. 150-160.

Yamaguchi, Y., Tsu, H., Kahle, A., and Nichols, D.A. 1994. ASTER instrument design and science objectives. *Amer. Inst. Aeron. Astron.* v. 7, paper 94-0597.

AVIRIS (Airborne Visible/Infrared Imaging Spectrometer)

(Robert R. McDougal)

PRINCIPLE: AVIRIS is a proven instrument in the realm of Earth Remote Sensing. It is a unique optical sensor that delivers calibrated images of the upwelling spectral radiance in 224 contiguous spectral channels (bands) with wavelengths from 400 to 2500 nanometers. AVIRIS has been flown on four aircraft platforms: NASA's ER-2 jet, Twin Otter International's turboprop, Scaled Composites' Proteus, and NASA's WB-57. The ER-2 flies at approximately 20 km above sea level, at about 730 km/hr. The Twin Otter aircraft flies at 4km above ground level at 130km/hr.

PRIMARY USES: The AVIRIS sensor collects data that can be used for characterization of the Earth's surface and atmosphere from geometrically coherent spectroradiometric measurements. These data can be applied to studies in the fields of oceanography, environmental science, snow hydrology, geology, volcanology, soil and land management, atmospheric and aerosol studies, agriculture, and limnology. Applications under development include the assessment and monitoring of environmental hazards such as toxic waste, oil spills, and land/air/water pollution. With proper calibration and correction for atmospheric effects, the measurements can be converted to ground reflectance data which can then be used for quantitative characterization of surface features.

LIMITATIONS: Cost of data acquisition is expensive, especially for temporal or change detection type studies. Data require expert knowledge for processing and usually require a ground calibration data collection. Georeferencing of data is very difficult.

INTERFERENCES: Atmospheric effects on spectral data must be removed; usually requiring the collection of ground calibration data. In areas of dense vegetation, geological and mineralogical studies have limited exposure.

SCOPE: AVIRIS data have been collected in North America, Europe, portions of South America, and most recently, Argentina. The main objective of the AVIRIS project is to identify, measure, and monitor constituents of the Earth's surface and atmosphere based on molecular absorption and particle scattering signatures.

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DETECTION LIMITS/AERIAL RESOLUTION: When collected by the ER-2 (20km above the ground) each pixel produced by the instrument covers an area approximately 20 meters diameter on the ground (with some overlap between pixels), thus yielding a ground swath about 11 kilometers wide. When collected by the Twin Otter (4 km above the ground), each ground pixel is 4 m square, and the swath is 2 km wide. The AVIRIS instrument contains 224 different detectors, each with a wavelength sensitive range (also known as spectral bandwidth) of approximately 10 nanometers (nm), allowing it to cover the entire range between 380 nm and 2500 nm. When the data from each detector are plotted on a graph, they yield a complete VIS-NIR-SWIR (visible-near infrared-short wave infrared) spectrum.

DATA FORMAT: Usually presented as digital or hard copy maps.

COST: AVIRIS data cost about $10 per square kilometer, but must be funded in full flights ($64k per flight + $6k per flight hour). A full flight can cover up to about 8,000 square kilometers spread over multiple sites.

BIBLIOGRAPHY:

Chrien, T.G. 1992. AVIRIS: Recent instrument maintenance, modifications and 1992 performance. *Summaries of the Third Annual JPL Airborne Geoscience Workshop,* JPL Pub. 92-14, v. 1, AVIRIS Workshop, 1992, Jet Propulsion Laboratory, Pasadena, CA, p. 78-79.

Chrien, T.G., Eastwood, M.L., Sarture, C.M., Green, R.O., and Porter, W.M. 1991. Current instrument status of the Airborne Visible/Infrared Imaging Spectrometer (AVIRIS). *Proc. of the Third AVIRIS Workshop,* JPL Pub. 91-28, 1991, Jet Propulsion Laboratory, Pasadena, CA, p. 302-313.

Chrien, T.G., Green, R.O., Sarture, C.M., Chovit, C., Eastwood, M.L., and Eng, B.T. 1993. Airborne Visible Infrared Imaging Spectrometer (AVIRIS): Recent improvements to the sensor. Summaries of the *Fourth Annual JPL Airborne Geoscience Workshop***,** JPL Pub. 93-26, 1993, v. 1, AVIRIS Workshop, Jet Propulsion Laboratory, Pasadena, CA, p. 27-30.

Porter, W.M., Chrien, T.G., Hansen, E.G., and Sarture, C.M. 1990. Evolution of the Airborne Visible/Infrared Imaging Spectrometer (AVIRIS) flight and ground data processing system. *Proc. of the Second AVIRIS Workshop,* JPL Pub. 90-54, 1990, Jet Propulsion Laboratory, Pasadena, CA, p. 271-277.

Landsat

(Robert R. McDougal)

PRINCIPLE: The mission of the Landsat Program is to provide repetitive acquisition of high resolution multispectral data of the Earth's surface on a global basis. Landsat represents the only source of global, calibrated, high spatial resolution measurements of the Earth's surface that can be compared to previous data records. The data from the Landsat spacecraft constitute the longest record of the Earth's continental surfaces as seen from space. The Landsat platforms carry multiple remote sensor systems and data relay systems along with attitude-control and orbit-adjust subsystems, power supply, and

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receivers for ground station commands and transmitters to send the data to ground receiving stations.

PRIMARY USES: Landsat images and data have been used for diverse applications for all manner of studies involving the Earth's surficial environments. Although originally designed for vegetation and geologic investigations, Landsat has proven surprisingly versatile for many different uses over land, aquatic, glacial, and marine settings.

- **Cartography:** basic cartography and new map projection, nautical charts of shallow seas.
- **Geology and geophysics:** bedrock mapping, identification of faults and lineaments, mineral prospecting, structural and tectonic geology, soil mapping, unconsolidated sediments, volcanic activity, and geomorphology.
- **Archeology:** ancient roads, lost cities, temple ruins, agricultural fields, irrigation canals, etc.
- **Hydrology:** rivers and lakes, flooding, erosion and deposition, coastal and wetland environments, irrigation, water quality, lake sediment and organic activity, snow cover, glaciers, ice sheets, and permafrost.
- **Agriculture:** cropland assessment, irrigation practices, rangeland assessment, forestry assessment; monitoring forest fires, crop disease, drought, insect infestations, etc.
- **Land use, environment, and conservation:** land-use monitoring, urban growth, mining and industrial activity, deforestation, oil-well fires, air pollution, water pollution, wetland habitats, and storm damage.
- **Oceanography:** ocean currents, ocean turbidity, coral reefs, pack ice, icebergs, and oceanic phytoplankton (algae).

LIMITATIONS: Spatial and spectral resolution may be too coarse for some applications.

INTERFERENCES: Cloud cover, particularly in tropical latitudes can be problematic. Atmospheric effects usually need to be removed.

SCOPE: Landsat has provided continuous coverage of the Earth's surface since 1972. It has a 16 day repeat coverage cycle.

DETECTION LIMITS/AERIAL RESOLUTION:

Landsat 1:

Operated from July 23, 1972 to August 5, 1972, recording only 1692 images

Return Beam Vidicon (RBV)

- 80 m resolution in the multispectral band
- Three cameras that operate in the following spectral bands:
    - Band 1 Visible blue-green (475-575 nm)
    - Band 2 Visible orange-red (580-680 nm)
    - Band 3 Visible red to Near-Infrared (690-830 nm)
- Data: 3.5 MHz FM video

Multispectral Scanner (MSS)

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- Four spectral bands:
    - Band 4 Visible green (0.5 to 0.6 μm)
    - Band 5 Visible red (0.6 to 0.7 μm)
    - Band 6 Near-Infrared (0.7 to 0.8 μm)
    - Band 7 Near-Infrared (0.8 to 1.1 μm)
- Six detectors for each spectral band provided six scan lines on each active scan
- Ground Sampling Interval (pixel size): 57 x 79 m

Landsat 2:

Operated from January 22, 1975 – February 25, 1982

The RBV system on Landsat 2 was operated primarily for engineering evaluation purposes and only occasional RBV imagery was obtained, primarily for cartographic uses in remote areas.

The rest of the configuration is the same as Landsat 1.

Landsat 3:

Operated from **March 5, 1978 – March 31, 1983**

Return Beam Vidicon (RBV)

- The RBV system on Landsat 3 used two cameras, mounted side-by-side, with panchromatic spectral response and higher spatial resolution (40 m) to complement the multispectral coverage provided by the Multispectral Scanner (MSS). Each of the cameras produced a swath of about 90 km (for a total swath of 180 km).
- 40 m resolution from 2, 80 m resolution cameras
- Two cameras with a panchromatic spectral response
- Data: 3.5 MHz video

Multispectral Scanner (MSS)

- Five spectral bands, including a thermal band:
    - Band 4 Visible (0.5 to 0.6 μm)
    - Band 5 Visible (0.6 to 0.7 μm)
    - Band 6 Near-Infrared (0.7 to 0.8 μm)
    - Band 7 Near-Infrared (0.8 to 1.1 μm)
    - Band 8 Thermal (10.4 to 12.6 μm)
- Data: 100 kHz digital
- Six detectors for each reflective band provided six scan lines on each active scan
- Ground Sampling Interval (pixel size): 57 x 79 m

Landsat 4:

Operated from **July 16, 1982 – (placed in standby in January 1986) December 14, 1993**

Multispectral Scanner (MSS)

- Four spectral bands (identical to Landsat 1 and 2):
    - Band 4 Visible (0.5 to 0.6 μm)
    - Band 5 Visible (0.6 to 0.7 μm)
    - Band 6 Near-Infrared (0.7 to 0.8 μm)
    - Band 7 Near-Infrared (0.8 to 1.1 μm)
- Data: 100 kHz digital
- Six detectors for each reflective band provided six scan lines on each active scan
- Ground Sampling Interval (pixel size): 57 x 79 m

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Thematic Mapper (TM)

- Added the mid-range infrared to the data
- Seven spectral bands, including a thermal band:
    - Band 1 Visible (0.45 – 0.52 μm) 30 m
    - Band 2 Visible (0.52 – 0.60 μm) 30 m
    - Band 3 Visible (0.63 – 0.69 μm) 30 m
    - Band 4 Near-Infrared (0.76 – 0.90 μm) 30 m
    - Band 5 Near-Infrared (1.55 – 1.75 μm) 30 m
    - Band 6 Thermal (10.40 – 12.50 μm) 120 m
    - Band 7 Mid-Infrared (IR) (2.08 – 2.35 μm) 30 m
- Ground Sampling Interval (pixel size): 30 m reflective, 120 m thermal

Landsat 5:

Operating March, 1984 – 2012 (Landsat 5 operations have been suspended indefinitely)
Specifications same as Landsat 4.

Landsat 6:

Launched October 5, 1993 (did not achieve orbit).

Landsat 7:

Operating April 15, 1999 – present (Scan Line Corrector malfunction after 2003)
Enhanced Thematic Mapper Plus (ETM+)

- Eight spectral bands, including a pan and thermal band:
    - Band 1 Visible (0.45 – 0.52 μm) 30 m
    - Band 2 Visible (0.52 – 0.60 μm) 30 m
    - Band 3 Visible (0.63 – 0.69 μm) 30 m
    - Band 4 Near-Infrared (0.77 – 0.90 μm) 30 m
    - Band 5 Near-Infrared (1.55 – 1.75 μm) 30 m
    - Band 6 Thermal (10.40 – 12.50 μm) 60 m Low Gain / High Gain
    - Band 7 Mid-Infrared (2.08 – 2.35 μm) 30 m
    - Band 8 Panchromatic (PAN) (0.52 - 0.90 μm) 15 m
- Ground Sampling Interval (pixel size): 30 m reflective, 60 m thermal
- Added the Band 6 Low and High gain 60 m thermal bands
- On-board calibration was added to Landsat 7: a Full Aperture Solar Calibrator (FASC) and a Partial Aperture Solar Calibrator (PASC), in addition to the 2 calibration lamps.

DATA FORMAT: Usually presented as digital or hard copy maps.

COST: Free to the public and government.

BIBLIOGRAPHY:

Arvidson, T., Gasch, J., and Goward, S.N. 2001. Landsat 7's long-term acquisition plan—an innovative approach to building a global imagery archive. *Remote Sensing of Environment* 78:13-26.

Earth Observation Satellite Company. 1985. *User's Guide for Landsat Thematic Mapper Computer-Compatible Tapes*. Lanham, MD: Earth Observation Satellite Company [variously paged].

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Earth Observation Satellite Company. 1994. *Landsat System Status Report—September 1994*. Lanham, MD: Earth Observation Satellite Company, p. 1-11.

Goward, S.N., Haskett, J., Williams, D., Arvidson, T., Gasch, J., Lonigro, R., Reeley, M., Irons, J., Dubayah, R., Turner, S., Campana, K., and Bindschadler, R. 1999. Enhanced Landsat capturing all the Earth's land areas. *EOS* 80(26): 289-293.

NASA (National Aeronautics and Space Administration). 1981. *Draft Landsat-D Worldwide Reference System (WRS) User's Guide*. Greenbelt, MD: National Aeronautics and Space Administration [variously paged].

NASA (National Aeronautics and Space Administration). 1983. *Landsat Data Users Notes.* Sioux Falls, SD: National Oceanic and Atmospheric Administration [variously paged].

U.S. Geological Survey and National Oceanic and Atmospheric Administration. 1984. *Landsat 4 Data User's Handbook*. Washington, D.C.: U.S. Geological Survey and National Oceanic and Atmospheric Administration [variously paged].

QuickBird

Robert R. McDougal)

PRINCIPLE: QuickBird is a high-resolution commercial earth observation satellite, owned by DigitalGlobe and launched in 2001 as the first satellite in a constellation of three scheduled to be in orbit by 2008. QuickBird uses Ball Aerospace's Global Imaging System 2000 (BGIS 2000) that collects the second highest resolution commercial imagery of Earth after WorldView-1, and boasts the largest image size and the greatest on-board storage capacity of any satellite. The satellite collects panchromatic (black & white) imagery at 60-70 centimeter resolution and multispectral imagery at 2.4- and 2.8-meter resolutions. At this resolution, detail such as buildings and other infrastructure are easily visible. The imagery can be imported into remote sensing image processing software, as well as into GIS packages for analysis. The imagery can also be used as a backdrop for mapping applications, such as Google Earth and Google Maps.

PRIMARY USES: QuickBird is unique because of its ultra-fine resolution, which makes it suitable for local land cover assessment as well as for validation of more coarse resolution collections. Additionally, QuickBird is valuable because the four multispectral bands approximate the first four bands of Landsat imagery. Although they are not immediately interoperable, they can be and still retain scientific integrity. QuickBird's ultra-fine resolution imagery is familiar to users of GoogleEarth, and anyone watching many news broadcasts involving that technology.

LIMITATIONS: Having one panchromatic and four multispectral bands can be limiting for some studies.

INTERFERENCES: Cloud cover, particularly in tropical latitudes can be problematic. Atmospheric effects usually need to be removed.

SCOPE: Quickbird has provided high resolution global coverage since 2001. By fall of 2009, the DigitalGlobe constellation will include Quickbird, WorldView-1, and WorldView-2.

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DETECTION LIMITS/AERIAL RESOLUTION:
Panchromatic – 450-900 nm, 0.6 m spatial resolution
Blue – 450-900 nm, 2.4 m spatial resolution
Green – 520-600 nm, 2.4 m spatial resolution
Red – 630-690 nm, 2.4 m spatial resolution
Near infrared – 760-900 nm, 2.4 m spatial resolution

DATA FORMAT: Usually presented as digital or hard copy maps.

COST:
Bundle imagery (Panchromatic + multispectral) (Table A3.18).

TABLE A3.18 Costs associated with QuickBird.

	Image Library	Standard Tasking
Standard	$24.00/km^2	$28.00/km^2
Ortho Ready Standard	$24.00/km^2	$28.00/km^2
1:50,000 Orthorectified	$36.00/km^2	$36.00/km^2
1:12,000 Orthorectified	$38.00/km^2	$38.00/km^2
1:5,000 / 1:4,800 Orthorectified	$40.00/km^2	$40.00/km^2
Custom Orthorectified	$40.00/km^2	$40.00/km^2

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APPENDIX 4—EXAMPLES OF SAMPLING PLANS AND QUALITY ASSURANCE PROJECT PLANS

Compiled by Carol C. Russell and Virginia T. McLemore

Reviewed by Thomas C. Moyer and Mary P. Goldade

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Volume 6: Sampling and Monitoring for the Mine Life Cycle

Editors: Virginia T. McLemore, Kathleen S. Smith, and Carol C. Russell

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DISCLAIMER:

The views expressed in this appendix are those of the authors and editors and do not necessarily represent the views of the U.S. Environmental Protection Agency or of the U.S. Government, nor does the selection and inclusion of example Quality Assurance Project Plans/Sampling and Analysis Plans (QAPP/SAP) necessarily represent current U.S. Environmental Protection Agency (USEPA) or other federal agency QAPP/SAP requirements. This handbook has been peer reviewed and approved for publication consistent with U.S. Geological Survey Fundamental Science Practices (http://pubs.usgs.gov/circ/1367, accessed August 2013). This appendix of example QAPP/SAPs is not intended to provide all of the information needed to conduct sampling and monitoring at mining influenced sites. The example plans do not contain all the elements required by USEPA QA/R-5 (USEPA 2001, 2006). Requirements for sampling plans are discussed in the main text of this handbook. Please also note that regulatory requirements may be more specific than the information included in this handbook. To obtain current USEPA requirements and guidance on quality assurance project plans, please visit www.epa.gov/QUALITY/qs-docs/r5-final.pdf (*EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5,* EPA/240/B-01/003, March 2001, accessed May 2013), which was reissued May 2006 (see www.epa.gov/QUALITY/qs-docs/reissue.pdf, accessed May 2013), and *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5*, EPA/240/R-02/009 (www.epa.gov/quality/qs-docs/g5-final.pdf, accessed June 2013). The descriptions 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. Any use of trade, firm, or product names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

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Appendix 4 consists of the following documents. The three Field Sampling Plans were used in three separate projects. These plans are unedited and reflect the plan "as is" during the projects.

1. EFFECT OF WEATHERING ON THE STABILITY OF THE QUESTA ROCK PILES (NEW MEXICO)—FIELD SAMPLING PLAN was used in the Questa, New Mexico project (QRPWASP 2009).
2. CINNAMON GULCH AND PENNSYLVANIA MINE SITE, SUMMIT COUNTY, COLORADO, SITE INSPECTION—SAMPLE AND ANALYSIS PLAN (Rudolph and Schroeder 2007, for the Colorado Department of Public Health and Environment).
3. QUALITY ASSURANCE PROJECT PLAN FOR THE DATA COLLECTION ACTIVITIES OF THE SACRAMENTO RIVER METALS TRANSPORT STUDY (U.S. Geological Survey and Sacramento Regional County Sanitation District 1996).
4. Also included in this appendix is the USEPA REGION 8 QUALITY MANAGEMENT PLAN CROSSWALK (www.epa.gov/region8/qa/R8-QMP-crosswalk%207-12-2011.docx, accessed May 2013). This crosswalk is used to review the Quality Management Plans (QMPs) that are submitted to the Region 8 Quality Assurance Office for review under ANSI/ASQC E4-1994, Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs and EPA Order 2105-0.

References:

QRPWASP (Questa Rock Pile Weathering and Stability Project). 2009. *Effect of Weathering on the Stability of the Questa Rock Piles (New Mexico)—Field Sampling Plan,* 2005, updated 2009, Characterization Team. http://geoinfo.nmt.edu/staff/mclemore/projects/environment/documents/samplingplan_6.pdf. Accessed May 2013.

Rudolph, M. and Schroeder, W. 2007. Site Inspection—Sample and Analysis Plan, Cinnamon Gulch and Pennsylvania Mine Site, Summit County, Colorado. Colorado Department of Public Health and Environment, Hazardous Materials and Waste Management Division.

USEPA (U.S. Environmental Protection Agency). 2001. *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5*. EPA/240/B-01/003. Washington, DC: Office of Environmental Information. www.epa.gov/quality/qs-docs/r5-final.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2002. *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5*. EPA/240/R-02/009. Washington, DC: Office of Environmental Information. http://www.epa.gov/quality/qs-docs/g5-final.pdf. Accessed June 2013.

USEPA (U.S. Environmental Protection Agency). 2006. Memorandum: *Reissue of Agency-wide Quality System Documents (QA/R2, QA/R-5, QA/G-7, and QA/G-10)*. Reggie Cheatham, Director, Quality Staff, Office of Environmental Information. www.epa.gov/quality/qs-docs/reissue.pdf. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2013. *EPA Region 8 Quality Management Plan Crosswalk* download. www.epa.gov/region8/qa/R8-QMP-crosswalk%207-12-2011.docx. Accessed May 2013.

USEPA (U.S. Environmental Protection Agency). 2013. A*gency-wide Quality System Documents* website. www.epa.gov/quality/qa_docs.html. Accessed May 2013.

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USGS-SRCSD (U.S. Geological Survey and Sacramento Regional County Sanitation District). 1996. *Quality Assurance Project Plan for the Data Collection Activities of the Sacramento River Metals Transport Study, Appendix 1*. Prepared by the U. S. Geological Survey for the Sacramento Regional County Sanitation District. http://pubs.usgs.gov/wri/wrir_994286/appendix_1.pdf. Accessed May 2013.

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EFFECT OF WEATHERING ON THE STABILITY OF THE QUESTA ROCK PILES (NEW MEXICO)--FIELD SAMPLING PLAN

Prepared by the QRPWASP Characterization Team

January 2005

REVISION LOG

Revision Number	Description	Date
SP.1	Original Field Sampling Plan	1/19/04
SP.2	Comments from Peters, Paul-Jackson, Robinson, Adams	2/16/04
SP.3	Revisions based on changes in scope and schedule	3/25/04
SP.4	Revisions by KMD	9/8/04
SP.5	Revisions by VTM incorporating comments from George Robinson	1/25/05
SP.6	Edits by VTM, including updates from the January 2005 version	1/28/09, 2/28/09

1.0 INTRODUCTION

In order to evaluate the stability of the Questa rock piles, a variety of field sampling methods of solid, water, and biological media are required. This Field Sampling Plan describes the field sampling and analyses that will be conducted as part of studies for the Rock Pile Weathering Stability study. The field sampling program will be carefully planned to meet the Data Quality Objectives (DQOs) outlined in the Quality Assurance Project Plan (QAPP), minimize cost, and minimize the potential conflicts between the numerous investigations. Appendix 1 is a list of SOPs. Appendix 2 is a list of personnel and emergency phone numbers. The QRPWASP database includes forms used during the sampling program. All of this information is in the QRPWASP database. The specific fields and data collected have been determined by various PIs to be important to characterize the samples.

2.0 DATA QUALITY OBJECTIVES

The proposed investigation will focus on the following interrelated critical issues:

1. Understanding weathering processes, both at the surface and within the mine-rock piles
2. Determine the rate at which such weathering processes occur within the rock piles over time

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3. Determining the effect of these processes on the long-term physical stability of the rock piles.

The key question to be addressed is, "Will the rock piles become gravitationally unstable over time?"

The characterization portion of this research project has identified ten Data Quality Objectives, described in the QAPP that must be addressed in order to solve this problem. The field sampling plan specifically addresses nine of the DQOs:

1. Determine how the hydrogeochemistry and water balance dynamics influences rock pile weathering and stability.
2. Determine how mineralogy, stratigraphy, and internal structure of the Questa rock piles contribute to weathering and stability.
3. Determine if the sequence of alteration (hypogene and supergene) and weathering within alteration scars and outcrops in the Questa area provides a basis to predict the effects weathering can have on mine rock material.
4. Determine how weathering of the rock piles affects the geotechnical properties of the rock pile material.
5. Determine if cementation forms in the rock piles and alteration scars and how does the cementation contributes to the stability of the rock piles.
6. Determine how reactive are pyrite and carbonate minerals so representative samples are used in the weathering cells.
7. Determine how much and where the pyrite is in the rock piles and how the pyrite concentration affects the weathering process.
8. Determine if pyrite oxidation, moisture content, and microbe populations affects rock pile weathering and stability.
9. Determine if the geotechnical and geochemical characteristics of the bedrock and alteration scars (foundation) underlying the rock piles influences the rock pile stability.

To meet these DQOs, the Characterization Team will implement this field sampling program in the Goathill North rock piles, other Questa rock piles, alteration scar areas, drill cuttings, and additional adjacent areas. This sampling plan provides the detail sampling procedures to accomplish the work plan.

3.0 STUDY AREA

The Questa molybdenum mine is located on the western slope of the Taos Range of the Sangre de Cristo Mountains, Taos County, northern New Mexico. The current mine is an underground, molybdenum disulfide ("moly") mine that currently uses a block caving mining method to extract ore. Pit production began in 1969 and ended in 1981, when 81 million tons of ore with an average grade of 0.185 % MoS_2 had been processed. During the open pit period of mining ~350 million tons of overburden rock were stripped and deposited onto mountain slopes and into tributary valleys forming the rock piles in this study.

4.0 INVESTIGATION APPROACH

The purpose of the Questa Rock Pile Weathering and Stability Project (QRPWASP) is to develop a model to assess and identify the future risk of physical failure of the existing rock piles based on the physical, chemical and mineralogical composition and weathering of the rock at Chevron's (formerly Molycorp) Questa mine, in Taos County, New Mexico. **The key question to be addressed is, "Will the rock piles become gravitationally unstable**

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over time?" Chevron further indicated that the "proposed investigation should focus on 1) understanding weathering processes both at the surface of and within the rock piles, 2) the rate at which such processes occur over time, and 3) the effect or influence of these processes on the overall mass stability of the rock piles."

Weathering can have at least two counter influences on slope stability. First, dissolution of feldspars, biotite and other silicate minerals results in reduction of the silicate mineral grain size and generation of clay minerals, both of which can adversely affect rock-pile stability. Second, iron oxyhydroxides generated by pyrite oxidation, as well as other reaction products, can aid in the cementation of soil grains. Formation of such aggregates will tend to increase rock-pile stability. Generation of water-soluble cements from the oxidation of pyrite also can adversely affect rock-pile stability. Both of these processes appear to be operational at the Questa site and have been observed at many mining sites.

Thus the first step in assessing the stability of the Questa rock piles is a complete physical, mineralogical, and chemical characterization of approximately 350 million tons of rock present in nine rock piles at the site utilizing new and existing data. This report addresses the field sampling plan (FSP) required to characterize the rock piles and other materials in the Questa area.

The field characterization investigation has been designed to collect data to:

- Characterize the overburden bedrock lithologies, including drill core from open pit deposits and surface outcrops
- Characterize the drill core through rock piles
- Characterize the rock piles in trenches
- Characterize the rock pile samples before and after accelerated humidity cells testing
- Characterize the alteration scars, debris aprons, colluvium, and weathering profiles through the bedrock
- Characterize the underlying country rocks
- Characterize material to be used as cover on the rock piles
- Characterize soils and colluvium on the adjacent slopes.

The following tasks will be performed:

- TASK 1 Literature review (Phase I)
- TASK 2 Characterization of overburden bedrock lithologies (Phase I)
- TASK 3 Characterization of drill core through rock piles (Phase I)
- TASK 4 Characterization of rock piles in test pits/trenches (Phase I)
- TASK 5 Characterization of rock pile samples before humidity cells testing (Phase I, II)
- TASK 6 Characterization of alteration scars, debris aprons, colluvium, and weathering profiles in the bedrock (Phase I, II)
- TASK 7 Characterization of underlying country rocks (Phase I)
- TASK 13 Stable isotopes of alteration materials(Phase I, II)
- TASK 14 3-D Characterization of sulfides using heavy liquid separations, XRD and geostatistics (Phase I, II)
- TASK 16 Drilling program (Phase I, II) (this task was eliminated)
- TASK B1.5 Characterization of rock-pile samples after humidity cells testing (Phase II)
- Task B1.1 Characterization of weathering & dev of weathering index and conceptual model (Phase II)

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- Task B1.2 Isotope/age dating (Phase II)
- Task B1.3 Develop the conceptual and descriptive rock pile model (Phase II)
- Task B1.4 Desription of natural analogs, alteration scars, debris flows, river ferricrestes, other rock piles (Phase II)
- Task B 5.1 Geotech-in situ testing (Phase II)
- Task McLemore48 Boulder weathering study (Phase II)
- Task McLemore49 Hot spots characterization study (Phase II)
- Task McLemore Crust study (Phase II)
- Task McLemore Megasample geotech testing study (Phase II)

Sampling of surface water will occur where encountered.

- TASK 11 Hydrological characterization (Phase I - III)
- TASK 12 Water stable isotopes (Phase I)

Supporting tasks

- TASK 8 What will the future climate be? (Phase I - II)
- TASK 10, B1.8.1 Spokesperson/Field manager duties (Phase I, II)
- TASK 17, B1.8.4 Database design and implementation (Phase I, II)

These tasks and supporting activities are described in this field sampling plan (FSP), drilling plans, and supporting SOPs.

5.0 RESPONSIBILITIES AND QUALIFICATIONS

The Characterization Team members will supervise the sampling activities. The Team Leader and Characterization Team will have the overall responsibility for implementing this sampling field plan. They will be responsible for assigning appropriate staff to implement this sampling field plan and for ensuring that the procedures are followed.

All personnel performing these procedures are required to have the appropriate health and safety training. In addition, all personnel are required to have a complete understanding of the procedures described within this sampling field plan, and receive specific training regarding these procedures, if necessary.

All environmental staff and assay laboratory staff are responsible for reporting deviations from this sampling field plan to the Team Leader.

6.0 PERMIT REQUIREMENTS AND NOTIFICATION

Chevron and its drilling contractor(s) are responsible for securing all necessary permits and meeting all local, state, and federal regulations applying to all sampling and drilling activities, including, but not limited to, site access, waste disposal, operator health and safety, etc.

7.0 LIST OF EQUIPMENT

See SOPs for list of equipment.

8.0 RELATED STANDARD OPERATING PROCEDURES

The procedures set forth in this plan are intended for use with the SOPs listed in Appendix 1.

9.0 FIELD ACTIVITIES

This section describes field activities that will be performed for the Chevron Weathering Project and consist of the following activities:

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- Premobilization and site reconnaissance
- Surveying
- Geophysical surveying
- Geological mapping of the rock piles
- Characterization sampling
- Drilling
- Ground water sampling
- Ground water monitoring well installation and development
- Microbe sampling
- Decontamination
- Quality control and quality assurance procedures

Instructions for implementing these tasks are given in standard operating procedures (SOPs), which are included with this FSP. Field audits, as described in the quality assurance project plan (QAPP), will be performed to verify that approved methods and procedures are followed during the field investigation. Any identified deviations from this FSP or SOPs will be documented and the Team Leader and Principle Investigators will be informed. Procedures for corrective action are described in the QAPP.

Chevron or their contractors will be responsible for containing and disposal of all wastes incurred during the field investigation as well as procedures for utility clearance.

Premobilization and site reconnaissance

One or more reconnaissance trips may be needed to identify sampling sites and address any potential safety issues. All trips to Questa must be scheduled with Virginia McLemore and Bruce Walker.

Sample Site Selection and Marking

Selected sample locations will be marked with flagging tape or paint. Sample sites will be selected on the basis of location of specific lithologic sample needed and safe access. Photographs will be taken (SOP 4).

Surveying

Conventional land and GPS survey techniques will be used to survey drill hole locations and elevations; surface water and sediment sampling locations; surface soil and biological sample locations; geophysical and sampling grid locations, elevations, and orientations; utility clearance (as applicable); and other surface and subsurface features. Chevron Mining Inc. will be responsible for all surveying other than GPS locations of samples. SOP 3 describes the procedures for GPS surveying.

Geophysical surveying

SOP 23 describes the procedures for surface geophysical surveys.

Geological mapping and modeling of the rock piles

The first step in geological mapping of the rock piles will identify existing data and maps. The maps will be incorporated into GIS as separate layers and other graphic programs as needed. The following is a partial list of GIS layers and models needed:

- Geology (Geologic Mapping Plan)
- Current topography

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- Original topography
- Remote sensing (Peters, Hauff)
- AVRIS
- Underground workings
- Drill holes, test pits/trenches locations
- Sample locations (proposed and already collected)
- Photograph locations
- Surface and borehole geophysical profiles
- Pyrite reserve model will provide a view of the pyrite distribution within the rock piles (SOP 34) (by Brimhall)
- Stratigraphic columns of drill holes and test pits/trenches (by Characterization team)
- Cross sections and longitudinal sections of the rock piles (by Characterization team)
- Geologic models of the rock pile (mineralogy, internal structure, stratigraphy) (by Characterization team)
- Hydrologic and hydrostratigraphic models to investigate hydrologic processes within the framework of our geologic models (by Characterization team)
- Heat flow/temperature/two-phase water flow models will help determine the degree to which heat can drive liquid and vapor-phase water flow within the piles and identify hot spots within the piles (by Reiter)
- Infiltration models will aid in estimating the range of liquid-phase water inflows to the rock piles using estimated, measured, or simulated climatologic inputs.
- A water balance model for each rock pile is critical to determining rock pile stability because it will predict how water content and matric potential are distributed within the pile as well as how they will evolve dynamically in time. Applying different future climate conditions to the water balance model will produce the matric potential distributions needed by the geotechnical models to predict long-term stability.
- Climatic models (by Characterization team)

The geologic mapping plan is developed and available

SAMPLING

Different sampling strategies will be employed based upon the purpose of each sampling task. Field geologists with experience in the regional stratigraphy and in the recognition of altered and weathered rocks perform the sampling. The types of samples to be collected include:

- Characterization sampling (rock outcrops, soil, other) (SOP 5)
    - **Criteria for selection:** weathering cells tests, alteration assemblages, different cementation, lithologies, mineral zonation, different stages of weathering
- Alteration scars (SOP 5)
    - **Criteria for selection:** lithologies, degree of alteration, specific minerals for stable isotopes and dating
- Soil samples of areas that represent what we want the final slopes to look like in terms of vegetation cover
- Samples to go into the humidity cells (SOP 5) **(samples to Univ of Utah)**
    - **Criteria for selection:** lithology, different stages of weathering, pyrite content, sulfur content, location is accessible; samples of the proposed cover material for the rock piles

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- Samples after humidity cells testing **(samples from Univ of Utah)**
- Drill core and cuttings of the deposit before being placed into the rock piles (SOP 5)
- Drill core and cuttings of the rock piles (SOP 5)
- Test pits, trenches, and high wall select and composite samples (SOP 5)
- Rock pile surface, channel, and transect sampling (SOP 5)
- Drill core and cuttings of the rocks underlying the rock piles (SOP 5)
- Sampling for the Remaining Pyrite Model (SOP 34) **(samples to Brimhall)**
- Surface water and seep sampling (SOP 15)
    - **Criteria for selection:** as available
- Samples for pore water measurements (SOP 39)
- Snow and rain (SOP 47)
    - **Criteria for selection:** as available
- Water vapor (SOP 46) and other gases
- Microbe sampling (SOP 37)
    - **Criteria for collecting from test pits/trenches:** increased temperature as determined during drilling using a standard thermometer
    - **Criteria for collecting:** include selected samples of all types of samples for microbe examination
- Samples for geotechnical testing

Characterization sampling

Characterization sampling is required to identify samples that go into the humidity cells as well as characterize the overburden, rock piles, underlying rocks, and other country rocks. All of the major lithologic units using outcrop localities, drill core, rock piles, and test pits/trenches will be sampled and will include different degrees of alteration and weathering, alteration assemblages and mineral zonation. SOP 5 describes the procedures for sampling outcrops, rock piles, and drill core (solid samples). SOP 6 describes the procedures for sampling during the drilling program. SOP 9 describes the procedures for sampling test pits, trenches, and high walls.

The exploratory stage of characterization will occur first, primarily to determine which samples are selected for humidity cell tests. This stage also will provide samples for characterization of rocks that went into the rock piles, rocks that underlie the rock piles, alteration scars, and debris flows. Samples of all of the major lithologic units as well as alteration assemblages and different stages of weathering will be collected from outcrop localities, open pit, drill core and cuttings, rock piles, test pits, and high walls. Additional samples will be collected from throughout the rock piles as needed.

A standardized protocol will be followed after each sample is taken (SOP 2). At each site, a select, grab, or bulk sample of rock or other material is collected for petrographic study and geochemical analyses that is representative of the DQO being addressed. A hand specimen also is collected for thin section and archived. Each sample is collected in a separate bag, assigned a unique number (field_id), and logged on a field description form (Appendix 3). Selected sample sites are marked in the field and a digital photograph (SOP 4) is taken at most localities. Photographs provide visual record of sample site; the photograph form identifies site specifics, provides basic location and other data about the photograph (SOP 4). Location information by GPS, type of sample, and field and laboratory petrographic descriptions will be collected. Each sample will be clearly identified. Geologic observations

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are recorded on the field description form and each site is located on a map (SOP 5). A global positioning system (GPS) reading is recorded as well (SOP 3). Hand specimen description provides a record of what was collected, aids in petrographic description, and provides information on sample for the labs (high S may be treated differently then low S). The hand specimen description is the preliminary data required to determine what samples need specific detailed analyses to meet the DQOs. NMBGMR will archive all samples for potential future studies.

Reflectance spectroscopy analyses in the field (SOP 41) provide on site mineral identification and helps in selecting samples for specific purposes to meet the DQOs (jarosite is needed for dating, field identify clay minerals and cements for easier sampling). Samples are selected in the field by visual examination in field that require immediate mineral identification.

The samples are transported from the field to NMBGMR (SOP 36), where each sample is prepared for analyses (SOP 8). Samples will be cut and chips will be sent for preparation of polished thin sections. The prepared samples are then sent to a laboratory for analyses. NMBGMR standards are submitted blind to the commercial laboratory with each sample batch to assure analytical quality.

Petrographic analysis and mineral identification are important in differentiating various rock units, determining rank and intensity of alteration, determining chemistry of alternating fluids, describing cementation, and determination of paragenesis of mineralization, alteration, and cementation. Alteration rank is based upon the mineral assemblages, which infers temperature, pressure, and permeability conditions at the time of formation. A weathering index will be defined and used to define the different stages of weathering. Petrography will be performed using standard petrographic and reflected ore microscopy techniques. Mineral concentrations will be estimated using standard charts and data. Estimates of both primary and alteration minerals will be determined, cementation described, and the alteration intensity will be determined from the concentration of alteration minerals. Forms are in Appendix 3. Digital photographs will be taken (SOP 4). Simple shear box tests will be performed on many samples to determine the strength of the material.

Characterization Sampling Procedure

1. Collect samples of all of the major lithologic units using outcrop localities, drill core, rock piles, test pits/trenches following the guidelines outlined for each task or specific sampling protocol, depending on the purpose of collecting the sample (SOP 5, 6, 9, 37). The numbering system we will use will be 3 letters that represent the mine feature (SSW)-3 initials of sampler-0001 sequentially. For example SSW-HRS-0001 is the sample #1 collected by Heather R. Shannon from the Sugar Shack West rock pile. If at all possible, collect a sample of the adjacent soil derived from the rock outcrop so that shear stength and other geotech tests can be performed. Samples of weathering profiles should be collected where ever possible to provide differences in weathering of the same material.
2. Record geologic observations on the **sample description form**. Fill out a chain of custody and request for analysis form (SOP 2).
3. Record site description on the **sample description form**.
4. Locate each site on a topographic map and take a GPS reading (NAD 27, UTM) and enter on the **sample description form (SOP 3).**

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5. Photograph sample site (information recorded on **photograph form,** SOP 4). The numbering system for the photograph will be the field identification number 0001 sequentially. For example SSW-HRS-0001-F001 is the photograph #1 that HRS took at sample site #1. Photographs will be taken at highest resolution as jpeg or tif files and stored in separate folders corresponding to their image type.
6. Archive hand sample or split of unconsolidated sample prior to sample preparation.
7. All data are entered into the project Access database

Procedure for collecting samples for humidity cells

1. Samples will be selected on the basis of representative lithology, variable pyrite concentrations, different alteration assemblages and intensity, and different stages of weathering.
2. Twenty to 400 pounds of rock are collected, including some fine material.
3. Samples are shipped to Univ of Utah for processing and weathering cells testing and to Univ of Utah for microbial analyses.
4. Univ of Utah will ship a split of the processed material to New Mexico Tech for characterization.

Procedure for collecting undisturbed samples

See appropriate SOPs for sampling of undisturbed materials for hydrologic and geotechnical tests.

Drilling and trenching

Subsurface data provides the third dimension to this study. Diamond drill-core and cuttings will be examined and logged. Trenches, test pits, and high walls will be examined. A binocular microscope and/or hand lens will be used in examining the samples. Samples will be obtained for thin section preparation and geochemical analyses. A summary of pertinent drill core data will be provided. Drill holes, trenches, test pits, and highwalls are located on Chevron maps. Digital photographs will be taken. SOP 5 describes the procedures for sampling outcrops, rock piles, and drill core (solid samples). SOP 6 describes the procedures for sampling during the drilling program (also see drilling plan). SOP 9 describes the procedures for sampling trenches, test pits, and highwalls. Selected samples sent to Univ of Utah for microbial analyses.

Surface water and seep sampling

Should any surface water or seeps be encountered in or near the rock piles, sampling could occur according to SOP 15. Selected samples sent to Univ of Utah for microbial analyses.

Microbe sampling

SOP 18, 37 describes the procedures for microbe sampling. **All samples collected for microbe studies must have a split sent for full chemical and mineralogical analyses.**

Decontamination

SOP 7 describes the procedures for decontamination of field equipment.

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10.0 ANALYTICAL ACTIVITIES

This section describes analytical activities that will be performed for the Chevron Weathering Project and consist of the following activities:

- Sample preparation
- Bulk mineral identification
- Clay mineral analyses
- Particle size determination
- Leaching analysis
- Geochemical and statistical analyses
- Shear box tests
- Decontamination
- Analytical measurements
- Quality control and quality assurance procedures

Sample preparation and analysis (solid)

- Solid samples crushed for chemical analyses and cut for thin sections (information recorded on **sample preparation form,** SOP 8).
- Polished thin sections of selected samples will be prepared by NMIMT lab and examined for mineralogy (**petrographic description form,** SOP 24).
- Photograph taken of thin sections (information recorded on **photograph form,** SOP 4).
- Selected thin sections and other samples for electron microprobe study (SOP 26).
- Samples analyzed for bulk mineralogy using XRD and recorded in **mineralogy table** (SOP 27).
- Selected samples analyzed for clay minerals and recorded in **clay mineralogy table** (SOP 29).
- Selected samples leached for DI leach for soluble ions (soluble mineralogy, SOP 38).
- Selected samples broken into size fractions and chemistry determined on individual size fractions (SOP 33).

Bulk mineral identification by XRD

Bulk mineral identification can be used to identify minerals present in quantities greater than approximately 3%. Altered, unaltered, and mineralized samples, including select samples of cement, will be powdered and analyzed by X-ray diffraction (XRD) at NMSU using pressed powder slides, as described in SOP 27. XRD analyses will be determined using a Rigaku XRD unit with CuKa at 50 kV and 25 mA. Weathered mine rock piles typically contain some amount of amorphous material that can not be identified by XRD (Smith et al., 2000), but by integrating electron microprobe and geochemical data this material can be characterized. This amorphous material typically contains metals that can be released into the water and also may form clay minerals. This amorphous material in some cases acts as a cement.

A random sample mount is used for a sample split after it has been ground to pass a 60-mesh sieve. With greater care, the sensitivity can be lowered to identify minerals in the ±2 wt.% range. However, this depends on the character of the x-ray reflections. Well-crystallized material is more easily identified than poorly crystalline material. Quartz is commonly the dominant mineral present in New Mexico sediments because these sediments

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consist of mostly of structural or detrital mineral grains. Cementing agents (clay minerals, dolomite, and calcite) are present in smaller amounts.

The identification is by principal reflections. In most samples from a given area or deposit, standard mineralogy is quickly established. After an initial period of mineral identification using all available reflections, only the principal reflections are used to compare ratios for semi-quantitative analyses. However, all diffractograms are scanned to see if minerals other than the standard suite are present. If unknowns other than very minor ones are identified, a computer program called JADE is available for mineral identification. If more accuracy that our normal semi-quantitative method is necessary, "spikes" of known quantities of the monomineralic materials are used with samples to establish the amount present.

Clay mineral analyses

The identification of clay minerals is commonly done with oriented <2 μm fraction of samples, as described in SOP 29. A split of the sample is placed in distilled water, stirred and allowed to settle. After a specified time interval, a portion of the <2μm fraction is removed from the top of the suspension, placed on a glass slide, and dried. The resulting coat consists of the oriented clay-size fraction of the sample. The slide is examined with x-ray diffraction from 2 to 40° 2θ, soaked in an ethylene glycol atmosphere and examined again with x-ray diffraction, and heated to 350°C for 30 minutes and examined again with diffraction. The results are reported as parts in ten of the following clay mineral groups: kaolinite, illite, chlorite, vermiculite, smectite, and I/S (mixed-layer illite-smectite). The mineralogy of the small amounts of non-clay minerals is determined as well. The common non-clay minerals are calcite, dolomite, gypsum, orthoclase, plagioclase, and quartz. When only small amounts of the feldspars are present, it may not be possible to distinguish between orthoclase and plagioclase.

Particle size determination

A split (preferred 500 gr, but can use 250 gr minimum) is taken of the original sample for particle size analysis, as described in SOP 33. It is then dried and weighed. The sand-and-larger-size material is first separated from the split by washing the split through a 230-mesh screen. The sand+ fraction remains on the screen. It is dried and weighed. The silt-and-clay mixture passing through the screen is separated into clay- and silt-clay suspensions by standard settle techniques. The clay suspension is weighed and the weights of the sand+ and clay fractions are used to calculate the silt fraction. Results accurate to +2%. If requested, the sand fraction can be separated from coarser material. If a greater number of size splits is needed, other labs at the Bureau can be used to determine these values.

Selected samples will be submitted for particle size analyses in order for bulk mineral analyses (XRD) and chemical analyses (XRF, ICP) to determine the mineralogy of the size fractions. Several 1000 gr of material will be required in order to provide enough material for analyses on each size fraction.

Chemical analyses

Chemical analyses of solids (ICP, XRF, other, SOP 30, 31, 28) provide detailed concentrations of abundance of specific elements that directly relate to mineralogy and water quality and are required for selecting samples for humidity cells **(data goes to Trujillo, Lapakko),** and for developing geotechnical models **(data goes to Wilson**), weathering

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models **(data goes to Trujillo**), and geologic (mineralogy, stratigraphy, internal structure) models of the rock piles.

Chemical analyses of water samples provide, in formation, on pore water chemistry and relates to the sequence of alteration SOP 30, 31).

1. Solid samples analyzed for major and trace elements using XRF, ICP, and other methods (**XRF**— SiO_2, TiO_2, Al_2O_3, Fe_2O_3, MnO, CaO, NaO, K_2O, P_2O_5, LOI, As, Ba, Cu, Pb, Zn, Mo, Cr, Co, Ni, V, Ga, Nb, Sc, Rb, Cs, Zr, Hf, Y, Ta, Th, U; **ICP**—Au, Ag, Sb, As, Se, Te, Ba, Sn, Bi, Tl, Cu, Pb, Zn, Cd, Mo, Cr, Co, Ni, V, Be, Sc, Rb, Cs, Zr, Th, U, La, Ce, Sm, Eu, Tb, Yb, Lu; **other methods**—SO4, S, CO_3, F, Hg) (SOP 11, 28, 30, 31, 35)
2. Water samples analyzed for major and trace elements using ICP and other methods— CO_3^{-2} , HCO_3^{-1}, Cl^{-1}, SO_4^{-2}, NO_3^{-1}, PO_4^{-3}, Na^{+1}, K^{+1}, Mg^{+2}, Ca^{+2}, Al, As, Ba, Cd, Cr, Co, B, Fe, Pb, Li, Mn, Hg, Mo, Ni, Se, Si, Ag, Sr, Th, U, pH, Conductivity, Total Dissolved Solids (TDS), Total Suspended Solids (TSS) (SOP 30, 31)

Paste pH and conductivity

Paste pH and paste conductivity (SOP 11) are used to evaluate the geochemical behavior of mine rock materials subject to weathering under field conditions and to estimate the pH and conductivity of the pore water resulting from dissolution of secondary mineral phases on the surfaces of oxidized rock particles.

Leaching analysis

This test, described in SOP 38, is designed to extract soluble carbonates and sulfates from minerals without attacking insoluble minerals such as silicates, and in particular, clay minerals. A simple washing or leaching of the sample is performed using deionized water. The leachate is analyzed for major and trace elements using the ICP. The carbonates and sulfates act principally as cementing agents as in New Mexico sediments, but also can be present as structural particles as well.

Electron microbe

Electron microprobe analyses (SOP 26) provide mineral identification, specific mineral chemistry, relationships between the minerals, provide information on how minerals are altering, and what new minerals forming at a microscopic level. Probe analysis can identify rims around sulfide minerals that could inhibit alteration of the mineral.

Criteria for selection: after petrographic analysis, clay mineralogy, DI leach, required to answer specific questions concerning microscopic characteristics

Stable isotopes

Stable isotopes of solid minerals (SOP 25) are important in understanding the origin of alteration and sequence of alteration and weathering with time. Analyzing the chemistry (SOP 30, 31) and stable isotope concentrations (SOP 25) of precipitation, snow, pore water, and exfluent (seepage) will constrain water flow models and provide calibration data.

Criteria for selection: occurrence of specific minerals

Stable isotope analyses of liquid and vapor-phase water provide a reality check on models of water and heat transport through the rock piles by revealing the amount of evaporation.

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Argon/argon age determination

Argon/argon age determinations (SOP 44) are important in understanding the origin of alteration and sequence of alteration and weathering with time.

Criteria for selection: occurrence of specific datable minerals

Porosity and bulk density

Porosity (SOP 42) and bulk density (SOP 32, 65, 70) are needed to estimate the critical hydraulic relationships (i.e., moisture retention relationship, SOP 45, and the hydraulic conductivity – moisture content relationship), and to model water transport and rock pile stability.

Moisture content

Mapping *in situ* moisture content describes a point-in-time distribution of water within the pile. It also identifies which hydrostratigraphic units are more likely to reach the near-saturation water contents necessary to induce movement. *In situ* moisture content measurements are inputs and/or calibration data for models of water flow **(data to Wilson**), reactive transport **(data to Trujillo**), and mechanical stability **(data to Wilson, Fredlund**). Determining the moisture retention (SOP 45) and hydraulic conductivity – moisture content relationships for selected hydrostratigraphic units are essential inputs for modeling water flow, reactive transport **(data to Trujillo**), and mechanical stability **(data to Wilson, Fredlund).**

Matric potential

Monitoring *in situ* matric potential values with tensiometers and thermal conductivity sensors describes the capillary forces driving water through the piles. The time evolution of matric potentials at selected locations will belp determine the overall dynamic water balance within the rock pile. Knowledge of matric potential distributions is essential for predicting water flow through the pile and therefore the pile's mechanical stability. Measured matric potentials are critical inputs and/or calibration data for modeling water flow, reactive transport **(data to Trujillo**), and mechanical stability **(data to Wilson, Fredlund).**

Climatic data

Collecting historic and on-site climate data such as precipitation, temperature, evaporation, snow accumulation, and wind speed are necessary inputs for all models involving water flow and understanding historic climatic patterns needed to predict future patterns.

Geochemical and statistical analyses

Statistical determinations of chemical analyses of samples collected for this study and of data sets provided by Chevron and their consultants are important in summarizing the data and ultimately in interpreting the significance of each data set. Each data set will be statistically analyzed separately using appropriate computer programs, such as WinStat. Means, standard deviations, and correlation coefficients will be determined where appropriate and factor analyses and discriminate analyses will be employed. Histograms, scatter plots, and other plots of the data will be performed to determine trends. Petrologic calculations of major-element analyses of unaltered and altered rock samples will be obtained.

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Shear box tests

Simple shear box tests will be conducted on fine grain size material to determine the strength of the material (SOP 50).

Decontamination

SOP 7 describes the procedures for decontamination of field equipment.

11.0 QUALITY CONTROL AND QUALITY ASSURANCE PROCEDURES

Procedure—A standardized protocol will be followed after each sample is taken and chain of custody and request for analyses will be completed. Location data by GPS, type of sample, and field and laboratory petrographic descriptions will be collected. Each sample will be clearly identified and a chain of custody process will be followed to assure that all samples were transported to the laboratory, analyzed, and results sent back to NMBGMR. The samples will be transported from the field to NMBMMR, where each sample will be crushed in jaw crushers and pulverized by tungsten-carbide disc grinders to a grain size of <35 μm. Each sample will be homogenized at each crushing step by cone and quarter method. The samples will be sent to a laboratory for analyses.

For each batch of 10 samples, 1 set of duplicate samples with different sample_id numbers will be run. For each batch of 25 samples, 1 set of triplicate samples with different sample_id numbers will be run. NMBGMR internal (Capulin, rhyolite, basalt, sand, Capulin rock pile) and commercial certified standards will be submitted blind to the laboratory with each sample batch of 25 samples to assure analytical quality. NMBGMR will archive a split of all samples for future studies.

Accuracy and precision of data—The accuracy of the data is how close the measured value is to the true value. Analyzing certified standards as unknown samples and comparing with known certified values monitors accuracy. The precision of an analysis is the repeatability of a measurement. Precision is monitored by multiple analyses of many sample duplicates and internal standards. Estimates of accuracy and precision will be determined. Errors due to accuracy will be determined by multiple analyses of commercial standards for which accepted values are known. Errors due to precision will be determined by multiple analyses of selected sample splits, as well as multiple analyses of standard samples. There are numerous reasons why duplicate samples and/standards do not always agree. Some samples, such as rhyolite and andesite, grind into powder more easily than other samples, such as stream-sediment and rock pile samples. Fusion techniques required for XRF analyses vary from lab to lab and may also differ between different personnel that could result in variations between sample pairs. Analytical error is higher for analyses with concentrations close to the detection limit. In addition, mine samples and alluvium, such as the Capulin standard (CAP-MLJ-0001), are very heterogeneous and difficult to completely homogenize.

Another problem encountered with mine samples, is the variability of sample collection. Only trained geologists will collect the Chevron samples using the exact procedures to avoid variations between sample collectors. In addition, three samples will be collected at two separate sites by one sampler to estimate sample heterogeneity and assigned different field_id numbers. At one site, two or three different people will collect a sample and assign a different field_id numbers to estimate sample collection variability.

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12.0 DATA MANAGEMENT

Data for the Rock Pile Weathering study will be obtained from a combination of sources, including field and laboratory measurements. The process of collecting and managing data is a coordinated effort (described in SOP 1) and will be conducted by project staff and laboratories working closely together. Laboratory data will be provided, when appropriate, in electronic form, in addition to the required hard-copy analytical data package. Data quality will be examined (SOP 1) before results are presented or used in subsequent activities. The laboratory will confirm sample receipt, sample condition, and required analyses. All pertinent information about each sample will be recorded.

13.0 SAMPLING SCHEDULE

August 12-13, 2003 Examination of Sugar Shack West and collection of samples to calibrate thermal camera (McLemore, Shannon)

September 12, 2003 Examination of rock piles, preliminary geophysical survey (McLemore, Dunbar, Gomez, Heizler, Hendickx, Sigda, Shannon)

October 22, 2003 Examination and sampling of alteration scars (Campbell, Lueth)

December 8-9, 2003 Logging and sampling of Sonic drill holes S-1, S-2 (McLemore, Gomez, Raugust, Tachie-Menson, Guiterez, Brimhall, Hoffman, Vargas)

January 7-12, 2004 Logging and sampling of SI holes, holes of pit deposit before mining, sonic holes, and collection of reports (McLemore, Gomez, Raugust, Tachie-Menson, Guiterez, Shannon, Sigda, Campbell, Lueth, Mullen, Krueger)

January 29-February 2, 2004 determination of characterization sample location, installing precipitation collectors (McLemore)

January-June 2004 Characterization sampling for determining which samples are submitted for weathering cells tests

January-June 2004 Logging and sampling of pre-existing drill core of the overburden before it was mined

February-June 2004 Sampling alteration scars for stable isotope analysis

February-June 2004 Geophysical surveying

May-October 2004 Characterize Goat Hill North rock pile

January-Febuary 2005 Characterize Goat Hill North rock pile

March–September 2005 characterization and drill core sampling

ANALYSES SCHEDULE

As samples are collected, sample preparation and analyses will occur as indicated on the chain of custody and request for analyses. The ICP-OES laboratory will not be available until February 2005.

14.0 TYPES, NUMBERS, AND LOCATIONS OF SAMPLES TO BE COLLECTED

All four media will be collected for this project: solid, water, air, and biological material. Gas analyses were added as a new task at the December meeting, therefore SOPs and a sampling plan are required and not included here. Table 1 describes the estimated number of samples proposed for collection for each proposed task. In addition, samples for microbe analysis could be collected from drill holes and test pits.

TABLE 1. Estimated number of characterization samples by tasks.

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Task	Title	Solid samples	Probe samples	Clay samples	Water samples
Task 1	Literature review	0	0	0	0
Task 2	Characterization of overburden bedrock lithologies, including drill core from open pit deposit and surface outcrops	50	20	30	0
Task 3	Characterization of drill core through rock piles	75	20	50	0
Task 4	Characterization of rock piles in test pits	110	20	50	0
Task 5 (phase 2)	Characterization of rock pile samples before weathering cells testing	110	40	40	0
Task 6	Characterization of alteration scars and debris aprons	70	20	20	0
Task 7 (phase 2)	Characterization of underlying country rocks	50	20	60	0
Task 8 (phase 2)	What will the future climate be	0	0	0	0
Task 9 (phase 3)	Characterization of rock pile samples after weathering cells testing	100	80	100	0
Task 10	Spokesperson duties	0	0	0	0
Task 11.1	Hydrostratigraphy and hydraulic properties	20	0	10	20
Task 11.2	Map and quantify liquid-phase inputs (infiltration)	10	0	5	10
Task 11.3 (phase 2)	Model heat and multi-phase water flow	0	0	0	0
Task 12	Constraining water fluxes by tracer analysis	0	0	0	40
Task 13	Stable Isotope and Age Characterization of Rock Piles and Alteration Scars	80	20	10	20
Task 14A	Remaining Pyrite Model using 5 foot samples of all new drill holes. Pulps subjected to heavy liquid separation methods, Quantitative XRD and modal reflected light analysis on check samples	Up to 3500 depending on drilling	0	0	0
Task 14B (phase 2)	Digital Elevation Model and Rock Pile Surface Dynamics and Monitoring with InSAR	0	0	0	0
Task 15 (phase 2)	Characterization and Geochemical modeling of water well data to define pyrite oxidation hotspots	0	0	0	Most of the existing water quality samples
Task 16	Drilling program	100	0	100	0
Task 17	Characterization Database				
Total		775 plus 3500	240	475	90

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TABLE 2. Type of sample

Type of sample	Description
drill cuttings	cuttings from reverse circulation
Becker cuttings	drill cuttings from Becker drill rig
sonic core	core obtained from sonic drilling
split spoon	sample collected from split spoon, indicate size in comments
drill core	diamond drill core
bucket	sample collected from backhoe bucket
grab	randomly collected sample by hand
hand auger	sample collected by hand using an auger
channel sample	continuous sample over a specified length and width
composite	sample collected from different locations and combined to form one sample
composite rock pile	sample collected from different locations and combined to form one sample in a rock pile
select	sample selcted for specific analysis
screened	solid sample screened to specified size fraction, specify size in comments
pump sample	
field blank	field blank, typically used for only water
field replicate	Field replicate sample
quality control sample	As required by SOP, generally 1 duplicate per 25 samples collected
biological	samples selected for biological purposes
Gas analyses	TBD

TABLE 3. Sample description

Sample_description	Description
well	water from well or drill hole
subsurface unsaturated/vadose zone	selected subsurface sample
waste water	waste water
mine drainage	water sample from mine drainage
rock	unaltered or slightly altered rock
mineralized rock	rock is altered or mineralized
ore	mined or extractable ore material
rock pile	rock material
soil	soil sample
sediment	stream or lake sediment
select	select sample for specific analysis
microbe	sample collected for microbe analyses
core	drill core
cuttings	drill cuttings

The following are the samples to be collected in this project and sampling procedures are described in the various SOPs.

- Characterization sampling (rock outcrops) (SOP No. 5)
- Alteration scars (SOP No. 5)
- Samples to go into the weathering cells (SOP No. 5)
- Samples after weathering cells testing
- Drill core and cutting of the deposit before being placed into the rock piles (SOP No. 5)
- Drill core and cuttings of the rock piles (SOP No. 5)

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- Test pits, trenches, and high wall select and composite samples (SOP No. 9)
- Rock pile surface, channel, and transect sampling (SOP No. 5)
- Drill core and cuttings of the rocks underlying the rock piles (SOP No. 5)
- Sampling for the Remaining Pyrite Model (SOP No. 34)
- Surface water and seep sampling (SOP No. 15)
- Ground-water sampling (SOP No. 16)
- Microbe sampling (SOP No. 37)
- Samples for Pore water measurements (SOP No. 39)
- Samples for geotechnical testing
- Air

15.0 SAMPLE MANAGEMENT

Samples are collected, prepared, and submitted to analytical laboratories for analyses. Specific analyses, procedures, and preservation methods are in the QAPP. Procedures for collection, analyses, handling, shipping, documentation, and chain of custody are in specific SOPs (Appendix 1). SOP 2 describes the procedures for sample management. Each sample, regardless of media, is assigned a unique field identification number.

The field identification (ID) number for samples will be comprised of three components, separated by dashes, for example SSW-HRS-0001, shown in Table 4.

TABLE 4. Field identification number (Field id).

Component 1	Component 2	Component 3
Three letter abbreviation for the mine feature, for example SSW for Sugar Shack West.	Three letter initials of the sample collector, for example HRS for Heather R. Shannon.	Sequential four number designation, for example 0001.

Each sample is then assigned a separate sample identification number during sample preparation. The first part is identical to the field identification number and is followed by a sequential two numbers pertaining to the type of analysis to be performed, for example SSW-HRS-001-01, shown in Table 5.

TABLE 5. Sample identification number (Sample id).

Component 1	Component 2	Component 3	Component 4
Three letter abbreviation for the mine feature, for example SSW for Sugar Shack West.	Three letter initials of the sample collector, for example HRS for Heather R. Shannon.	Sequential four number designation, for example 0001.	Sequential two number designation to indicate the type of analysis, for example 01.

Any deviations from this sampling numbering system will be documented and reported to the Team Leader and Principle Investigators and recorded in the appropriate forms.

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16.0 FIELD DOCUMENTATION

Forms will be used to record field and laboratory data (Appendix 3). These forms include:

- Sample description form
- Photograph form
- Drill log
- Test pit log
- Chain of custody and request for analysis

All forms that are completed by hand and all field forms will be archived in a binder in Socorro for future reference. Data will be immediately entered into the database. Copies of the database forms after completion also will be stored in binders.

Geologic observations and site description are recorded on the **sample description form**. Each site is located on a topographic map and a GPS reading taken (NAD 27, UTM) (SOP 3) and entered on the **sample description form.**

Photographs are taken of sample site (SOP 4; information recorded on **photograph form**). The numbering system for the photograph will be the field identification number (three components) or sample number (four components) followed by a letter representing type of image (F-field, T-thin section, P-probe, H-test pit, D-drill core/cuttings, G-general, O-historic) 001 sequentially (Table 6). For example SSW-HRS-001-F001 is the photograph #1 that HRS took at sample site #1. Photographs will be taken at the highest resolution as jpeg or tif and stored in separate folders corresponding to their image type.

TABLE 6. Photograph Number (Photo no).

Component 1	Component 2	Component 3	Component 4	Component 5
Three letter abbreviation for the mine feature, for example SSW for Sugar Shack West.	Three letter initials of the sample collector, for example HRS for Heather R. Shannon.	Sequential four number designation, for example 0001.	Sequential two number designation, for example 01.	Letter of image type (F, T, P, H, D, G, O, M), followed by a sequential three number designation, 001.

17.0 SAMPLE PRESERVATION, STORAGE, SHIPPING AND CUSTODY

Preservation and Storage

SOP 36 describes the procedures for sample preservation, storage, and shipment. Table 7 summarizes the parameters, preservation methods, and holding times acceptable for water samples. Sample filtration, if required, is explained in SOP 15 and 16.

TABLE 7. Sample preservation and holding times for water samples.

Parameter	Field Analysis	Glass sample container	Polyethylene sample container	Preservation	Maximum storage/holding time
pH	X			unpreserved	6 hours
Electrical conductance	X			unpreserved	24 hours
Temperature	X				

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Parameter	Field Analysis	Glass sample container	Polyethylene sample container	Preservation	Maximum storage/holding time
Dissolved oxygen	X				
TOC					
redox					
TDS		X	X	unpreserved	7 days
Acidity		X	X	unpreserved	24 hours
Alkalinity		X	X	unpreserved	24 hours
Calcium (Ca)			X	HNO3	6 months
Magnesium (Mg)			X	HNO3	6 months
Sodium (Na)			X	HNO3	6 months
Potassium (K)			X	HNO3	6 months
Chloride (Cl)		X	X	unpreserved	7 days
Sulphate(SO4)		X	X	unpreserved	7 days
Nitrate (NO3)		X	X	unpreserved	24 hours
Total iron (Fe)			X	HNO3	6 months
Ferrous Iron			X	HNO3	24 hours
Aluminium (Al)			X	HNO3	6 months
Arsenic (As)			X	HNO3	6 months
Cadmium (Cd)			X	HNO3	6 months
Copper (Cu)			X	HNO3	6 months
Lead (Pb)			X	HNO3	6 months
Nickel (Ni)			X	HNO3	6 months
Zinc (Zn)			X	HNO3	6 months
Mercury (Hg)		X		H2SO4+K2 Cr2O	1 month

Shipping (SOP 36)

Samples collected during this investigation will be either shipped to the laboratory via a carrier or will be hand delivered to Socorro for preparation and analysis. If the samples are sent by common carrier, an air bill will be used. Receipts of air bills will be retained as part of the permanent documentation. Commercial carriers are not required to sign off on the chain-of-custody forms as long as the chain-of-custody forms are sealed inside the sample cooler and the custody seals remain intact.

If, at any time after samples have been secured, custody seals on the container are identified as having been tampered with, the following procedures will be conducted:

- Check with personnel having access to sample coolers to evaluate whether inadvertent tampering can be documented.
- Document findings of the incident in a logbook.

If it cannot be documented that inadvertent breaking of the custody seal that did not affect the integrity of samples has occurred, the samples will be re-collected and the Project Manager and QA Officer will be notified.

Sample Archival and Disposal

As described in the QAPP and SOPs, all solid samples will be collected and archived at NMBGMR or the mine for potential future evaluation. Groundwater, surface water, and

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microbe sample aliquots will not be archived as a part of the study for future analysis or consideration. Any water sample volume not consumed during sample analysis will be disposed of by the laboratories as described in the laboratory QAM, and in accordance with all applicable rules and regulations.

18.0 HEALTH AND SAFETY

The site health and safety plan (HASP) will be followed by all contractors and subcontractors working on the site. It is the policy of New Mexico Tech to provide a safe and healthful work place for all employees, subcontractors, and clients in compliance with Chevron and MSHA requirements. Telephone contacts are in Appendix 2.

Safety shall take precedence over expediency or short cuts. It is a condition of employment that all employees and subcontractors work safely and follow established safety rules and procedures. All injuries, vehicle accidents, and incidents with potential for injury or loss will be investigated. Appropriate corrective measures will be taken to prevent recurrence, and to continually improve the safety of the work place. Chevron has a mandatory drug testing program that all personnel working on the property must follow.

Chevron and its drilling contractor(s) will identify and rectify all subsurface and overhead hazards at each drilling or other construction site in accordance with the site Health and Safety Plan (HASP). They also will identify other hazards associated with mining activities including, but not limited to, ore haulage, slope stability, high walls, open pits, blasting areas and any other potential mining and/or drilling hazards. The characterization team will notify Chevron of movements about the site and personnel locations. Communication will be conducted using two-way radios and other signals specified by Chevron.

The characterization team will comply with all of Chevron's notification, communication, and specific permit requirements at all times while working on site in accordance with the QAPP. Drill hole, test pit excavation, and other excavations will be cleared of utilities by Chevron or their designee prior to drilling or excavation.

19.0 REFERENCES

APPENDIX 1. List of SOPs and plans

Number	Name	Description
MP	Project Management Plan	Overall project management plan
WP	Work plan	Work plan for the project
HASP	Health and Safety Plan	Health and safety plan for field and laboratory work
QAPP	Quality Assurance Project Plan	Quality assurance and quality control plan
FSP	Field Sampling Plan	Field sampling plan
DP1	Drilling plan, phase 1	Drilling plan
DP2	Drilling plan, phase 2	Drilling plan
GMP	Geologic mapping plan	Geologic mapping plan
SOP 1	Data management	entering, reporting, verification, and validation of data to the database
SOP 2	Sample management	procedures of handling samples from field to laboratory to archive
SOP 3	Surveying (GPS)	field procedures using GPS and other surveying methods
SOP 4	Photography	procedures taking photographs in the field and laboratory
SOP 5	Sampling outcrops, rock piles, and drill core	field procedures for taking surface solid samples
SOP 6	Drill logging and sampling of	field procedures for drilling, logging, and sampling of

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Number	Name	Description
	subsurface	subsurface samples (solids)
SOP 7	Sample equipment Decontamination	field procedures for decontamination of sampling equipment
SOP 8	Sample preparation	laboratory procedures for sample preparation (solids)
SOP 9	Test pit excavation, logging, and sampling (solid)	field procedures for test pit excavation, logging, and sampling (solid)
SOP 10	Met station maintenance	field procedures for maintaining meteorological station
SOP 11	Paste pH and paste conductivity	laboratory procedures for paste pH and paste conductivity
SOP 12	Field measurements of water	field procedures for measuring water flow, pH, conductivity, alkalinity, temperature when collecting water samples
SOP 13	Water elevation measurements	field procedures for measuring water elevations in drill holes
SOP 14	Field filtration of water samples	procedures for filtering water samples in the field
SOP 15	Surface water and seep sampling	field procedures for collecting samples of surface and seep water samples
SOP 16	Groundwater sampling	field procedures for collecting ground-water samples
SOP 17	Borehole logging	field procedures for borehole logging
SOP 18	Pump testing	field procedures for collecting information during pump testing
SOP 19	Geophysical logging	field procedures for borehole geophysical logging
SOP 20	Well development	field procedures for development of wells
SOP 21	Monitoring well installation	field procedures for installing monitoring wells and instrumentation
SOP 22	Analytical data validation	procedures for data validation
SOP 23	Geophysics with electromagnetic induction	procedures for geophysical surveys
SOP 24	Petrographic analysis	laboratory procedures for describing petrographic samples
SOP 25	Stable isotope analysis	laboratory procedures for stable isotope analyses
SOP 26	Electron microprobe analyses	laboratory procedures use for analyses using the electron microprobe
SOP 27	X-ray diffraction (XRD) analyses	laboratory procedures for mineralogical analyses by x-ray diffraction (XRD)
SOP 28	X-ray fluorescence (XRF) analyses	laboratory procedures for chemical analyses by x-ray fluorescence (XRF)
SOP 29	Clay mineralogy analyses	laboratory procedures for sample preparation and XRD analyses of clay minerals
SOP 30	ICP-OES analyses	laboratory procedures for chemical analyses using ICP-OES
SOP 31	ICP-MS analyses	laboratory procedures for chemical analyses using ICP-MS
SOP 32	Bulk density	laboratory procedures for determining bulk density
SOP 33	Particle size analysis	laboratory procedures for determining particle size analyses
SOP 34	Sampling for the Remaining Pyrite Model	approach, collection of samples and laboratory procedures required for sampling for remaining pyrite model
SOP 35	Volumetric moisture content	collection of samples and laboratory procedures for determining volumetric moisture content
SOP 36	Sample preservation, storage, custody, shipping	procedures for sample preservation, storage, and shipment
SOP 38	DI leach	laboratory procedures for leaching solid samples by deionized water to provide for soluble material
SOP 39	Samples for Pore water measurements	laboratory procedures for collecting samples for pore water measurements
SOP 40	Gravimetric moisture content	collection of samples and laboratory procedures for gravimetric moisture content
SOP 41	Reflectance spectroscopy	field procedures for mineralogical analyses using

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Number	Name	Description
		reflectance spectrography
SOP 42	Porosity	laboratory procedures for determining porosity
SOP 43	Tensiometer and thermal conductivity thermal conductivity sensor installation	field procedures for installing tensiometers and thermal conductivity sensors procedures for argon/argon dating
SOP 44	Argon/argon geochronology	laboratory procedures for argon/argon dating
SOP 45	Moisture retention relation by hanging column	procedures for determining moisture relations
SOP 47	Rain and snow collection for isotope	field procedures for collecting rain and snow
SOP 48	Dye tracer studies	Tracer studies using dyes
SOP 49	Chip tray preparation	How to prepare chip trays of drill cuttings for examination
SOP 50	Direct Shear tests	How to do simple shear box tests
SOP 51	Collecting Thermal images	How to collect thermal images using thermal camera
SOP 52	Static Net Acid Generation (NAG) Test	NAG test for laboratory
SOP 53	Tension Infiltrometer	procedures for tension infiltrometer measurements
SOP 54	Atterberg Limits	procedures for Atterberg Limits
SOP 55	General Microbial Sampling – Solids	General Microbial Sampling - Solids
SOP 56	Classical microbial analysis – solids	Classical Microbial Analysis - Solids
SOP 57	Microbial laboratory safety	Microbial Laboratory Safety
SOP 58	Microbial metabolic profiles – biology	Microbial Metabolic Profiles - Biology
SOP 59	Microbial nucleic acid analysis	Microbial Nucleic Acid Analysis
SOP 60	Slurry pH-redox-conductivity-temperature	Slurry Ph – Redox – Conductivity - Temperature
SOP 61	Neutron density gauge	Describes measurements taken with the nuclear density gauge (density, water content)
SOP 62	Acid-base accounting (ABA)	Procedures for acid base accounting in laboratory
SOP 63	Kelway soil acidity and moisture measurements	Kelway Soil Acidity and Moisture Tester for field measurements
SOP 64	Portable tensiometer	procedures for using field portable tensiometers as opposed to in place monitoring
SOP 65	Sandcone	procedures for sand cone
SOP 66	Gas analyzer	procedures for gas analyzer
SOP 67	Solid sample collection and compound analysis	procedures for solid sample collection and compound analysis
SOP 68	Water analyses	water analyses in lab
SOP 69	Other chemical analyses on solids	other chemical analyses on solids (ammonia, nitrate, fluorine, etc)
SOP 70	Sand replacement	calculates volumetric moisture content and bulk density
SOP 71	Guelph permeameter	procedures for guelph permeameter measurements
SOP 72	SWCC	Soil water characteristic curve (UBC)
SOP 73	Falling head Permeability	Permeability by falling head method
SOP 75	Specific gravity	procedures for determining specific gravity
SOP 76	Slake durability	procedures for slake durability tests
SOP 77	Point load	procedures for point load tests
SOP 78	Humidity cell testing	procedures for weathering cells tests
SOP 79	Sample preparation for humidity cell testing	procedures for weathering cells sample selection and preparation
SOP 90	XRD sample preparation for pyrite reserve model	XRD sample preparation for pyrite reserve model

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Number	Name	Description
SOP 91	Color	procedures for obtaining the color of a soil sample

APPENDIX 2. Telephone Contacts

Team Project Manager: Dr. Terry Chatwin Team Leader

Characterization Team Leader Dr. Virginia T. McLemore 505-835-5521 (office)
505-835-3823 (home)
505-838-6040 (cell)

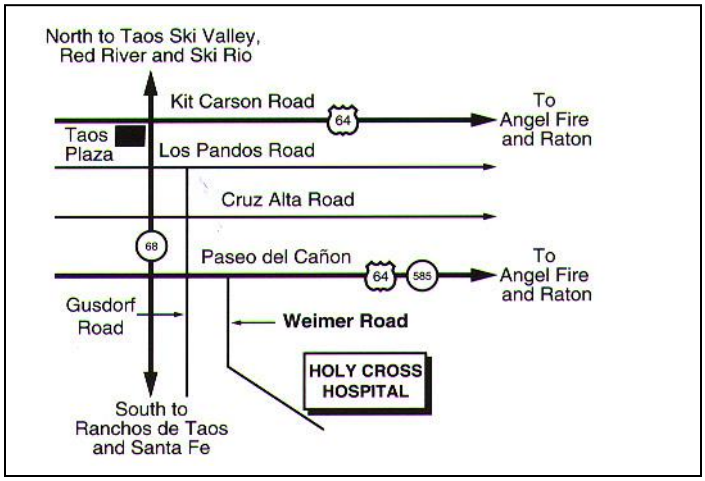
Chevron Guard Station: (505) 586-7640

Chevron Geologist: Bruce Walker (505) 586-7628

FIRE, POLICE, SHERIFF, AND AMBULANCE EMERGENCIES ONLY: 911
Ambulance: Taos County Non-Emergency: (505) 758-9591
Animal Control: Taos Animal Control: (505) 758-2981

Health Care:

Holy Cross Hospital, Taos: (505) 758-8883
Hospital Address:
1397 Weimer Road
Taos, NM, 87571



(see map at right)

Questa Health Center: (505) 586-0315
Red River Medical Office: (505) 754-2379

Poison And Drug Information Center: 1-800-432-6866

Police - Non Emergency:

Questa: (505) 586-1196
Red River: (505) 754-6166
Taos: (505) 758-4656
Sheriff - Non Emergency: (505) 758-4709
State Police - Non Emergency: (505) 758-8878

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Note: Cellular telephones often do not work at the field site. If necessary, use telephones located at the Red River Waste Water Treatment Plant, the Town of Red River, or the Chevron mine offices.

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COLORADO DEPARTMENT OF PUBLIC HEALTH
AND ENVIRONMENT
HAZARDOUS MATERIALS AND WASTE MANAGEMENT DIVISION

SITE INSPECTION - SAMPLE AND ANALYSIS PLAN

CINNAMON GULCH AND PENNSYLVANIA MINE SITE
SUMMIT COUNTY COLORADO

Prepared by:

Mark Rudolph
William Schroeder

Approved: _____ **Date:** 6/18/07
Daniel Scheppers, Superfund Unit Leader

Approved: _____ **Date:** 6/22/07
Jean Mackenzie, EPA Project Manager

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SITE INSPECTION - SAMPLING AND ANALYSIS PLAN
CINNAMON GULCH AND PENNSYLVANIA MINE SITE
SUMMIT COUNTY COLORADO

1.0 INTRODUCTION

The Hazardous Materials and Waste Management Division (HMWMD) of the Colorado Department of Public Health and Environment (CDPHE) will conduct a Site inspection (SI) of the Cinnamon Gulch Watershed and Pennsylvania Mine Site, located in Summit County Colorado (Figure 1). This SI is being conducted and is consistent with all requirements set forth under 40 CFR Part 312, *Standards and Practices for All Appropriate Inquiries.* The US Environmental Protection Agency (EPA), Region 8, is coordinating with, and providing assistance on the surface water, sediment, and macroinvertebrate sampling and analysis component of this investigation, as well as providing aquatic toxicity testing in the Region.

Fieldwork for this SI is tentatively scheduled to begin during the early summer of 2007 for a high-flow surface water sampling and in late summer 2007 for all mine waste, seep sampling and low-flow surface water sampling. This Sampling and Analysis Plan (SAP) is prepared to guide field operations and to outline the analytical objectives for the SI. This SI will evaluate potential site source areas by evaluating existing data and by collecting surficial composite soil samples and depth soil samples to characterize contamination. The proposed use of the Cinnamon Gulch Watershed and Pennsylvania Mine Site is to be preserved as Open Space.

This SAP calls for the collection of composite mine waste samples from Cinnamon Gulch and the adjacent portion of Peru Creek, and samples of water discharge from identified mining features in the watershed that have been identified as wet or saturated or discharging water from an historic mining feature.

Composite mine waste samples will be collected from areas in the watershed that show an environmental degradation ability that constitutes a potential threat to surface water quality in the watershed. Criteria used to determine if a specific mine waste pile will be sampled are as follows and based on Environmental Risk Ranking Criteria (2 through 5) from the BLM publication, *Risk Management Criteria for Metals at BLM Mining Sites*:

1. If a property has an associated water discharge or contaminated ground water concern associated with it. An example of such a site would be a wetland adjacent to, or on any suspect mine waste or a discharging adit.

2. Properties that have suspect surface water erosion based on visual observations of erosion gullies or rills made during the fieldwork portion of the AGS report (American Geological Services 2003a; American Geological Services 2003b). These sites have the potential for surface water run-on and run-off controls to limit further contact with site related drainage.

3. Properties located within 300 feet of surface water.

All mine waste soil samples will be tested for the following list of total metals: aluminum, arsenic, cadmium, chromium, copper, iron, lead, manganese, magnesium, mercury, nickel, selenium, silver, and zinc. Additionally, composite mine waste samples will be analyzed for Acid Base Accounting (ABA) and the USGS

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Modified Leach Test Method which uses a de-ionized water solution instead of a dilute nitric acid solution as in the SPLP. The modified USGS method more accurately represents the natural state of leaching by natural precipitation of historical mine waste. This method quantifies toxic inputs and the soluble constituents available to mobilization. All mine waste and drainage sample locations are shown in Figures 1, 2, and 3 and are described in Tables 1 and 2.

All surface water samples collected during the high flow event in early summer 2007will be analyzed for sulfate, chloride, alkalinity, dissolved organic carbon, and the following list of dissolved and total recoverable metals: aluminum, arsenic, cadmium, chromium, copper, iron, lead, manganese, magnesium, nickel, mercury, selenium, silver, and zinc. Various cations, including sodium, potassium, calcium, and magnesium, will also be analyzed from the dissolved metals fraction. Certain locations within the Peru Creek watershed where chemistry samples are collected will also be sampled for macroinvertebrates, sediment metals, and surface water toxicity to fish. Whenever a water chemistry or toxicity sample is collected, field parameters, including temperature, conductivity, pH, dissolved oxygen, and flow will be taken using EPA protocols. Table 3 describes all of the locations to be sampled during the high flow event and the samples to be collected at each location. Tables 4 through 8 describe all parameters to be analyzed from the water and sediment samples and the required container types, preservatives, detection limits, and analytical holding times.

Sampling and analysis will include appropriate laboratory and field quality assurance/quality control protocols to ensure data are of known quality and can be used for the intended purposes. QA/QC protocols are described in Section 7.0 of this SAP.

2.0 OBJECTIVES

This SAP calls for the collection of sampling and other information to characterize and quantify the presence and level of contaminants in site area mine waste piles and discharges to local surface water. These data will identify and document the potential threat to human health or the environment posed by these possible sources of metals contamination to Cinnamon Gulch and the adjacent portion of Peru Creek. CDPHE and EPA will collect these samples with the intent of fulfilling the following objectives:

- Identify, compile and inventory all Recognized Environmental Conditions as per American Society for Testing and Materials Standard ASTM E 1527-00 in the Cinnamon Gulch Watershed and the Pennsylvania Mine Site;
- Collect composite soil samples and water samples from mine drainage features in the Cinnamon Gulch Watershed to identify locations in the watershed to determine major contributors of metals loading to Cinnamon Gulch;
- Quantify potential site remedial strategies to reduce overall metals loading from Cinnamon Gulch and the section of Peru Creek along the Pennsylvania Mine and Decatur Townsite;
- Assist the Peru Creek Watershed Group in a Voluntary Cleanup and Redevelopment Act (VCRA) application that provides for the protection of public health and the environment.
- Determine the impact that degraded water quality in the watershed is having on aquatic life and the amount of metals reduction that will be required in order to have a sustainable trout population downstream of Peru Creek;
- Characterize the toxicity of various tributaries in Peru Creek and the Snake River and determine the toxic effects that these tributaries are having on these watersheds;

3.0 BACKGROUND INFORMATION

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3.1 LOCATION AND SITE DESCRIPTION

The Peru Creek and Cinnamon Gulch drainages are located in Summit County, Colorado, in the Montezuma Mining District east-northeast of the town of Montezuma (Figure 1). The headwaters of Peru Creek and Cinnamon Gulch originate from the highly mineralized and glaciated terrain found along the Continental Divide (Wood et al, 2005).

Cinnamon Gulch and the Peru Creek drainage are located approximately 14 miles east of Dillon and 4 miles northeast of Montezuma, Colorado. The site area is reached by traveling east on US 6 from Interstate 70 in Dillon to the town of Keystone. Continue on US 6 for approximately 1.2 miles past Keystone to Montezuma Road. Turn south at Montezuma Road and then turn left at the first fork in the road and proceed east, past the ski area parking lots, for 4.6 miles to where the road makes a sharp bend and crosses the Snake River. Immediately after crossing the Snake River turn left at the unpaved 4-wheel drive road and proceed first north then west up Peru Creek on a road that becomes increasingly rugged (URS 2002). During winter and spring months, the forest service road along Peru Creek is locked and access must be acquired from the USFS.

Elevations range from about 10,800 feet above sea level at the confluence between the lower branch of Cinnamon Creek and Peru Creek, to 11,800 feet at the Silver Spoon Mine (apparent headwaters of Cinnamon Creek). The highest point in the Cinnamon Gulch watershed is 12,889 feet on Revenue Mountain. Cinnamon Gulch flows between Silver and Brittle Silver Mountains on the west, and Revenue and Decatur Mountains on the east (Wood et al, 2005).

The Snake River watershed is located in central Colorado's Rocky Mountains immediately west of the Continental Divide. As a result of both historic mining activity and natural biogeochemical processes, a large percentage of the basin is impacted by acid rock drainage. Peru Creek is the major tributary that drains the study area and then flows into the Snake River, which in turn flows into Dillon Reservoir, a municipal source of drinking water for the City of Denver. The drainage basin of Peru Creek is enclosed by the continental divide on the north, east, and south and encompasses approximately 20 square miles (URS 2002).

The Snake River and Peru Creek are identified on Colorado's 2000 303(d) list of impaired water bodies due to high concentrations of zinc, cadmium, copper, lead, and manganese. The headwaters of the Snake River receive inflow from acidic and metal-enriched tributaries and groundwater on the eastern side of the watershed, where disseminated pyrite is abundant in the country rock (Theobald 1963). This headwater reach also runs through a naturally occurring bog iron ore deposit. The inflow of Deer Creek, a pristine tributary of approximately equal flow, raises the pH and causes precipitation of aluminum and iron hydroxides (Wood et al, 2005).

Cinnamon Gulch receives inflow from numerous abandoned mines. The Pennsylvania Mine, located immediately upgradient of the Cinnamon Gulch and Peru Creek confluence, has been found to be one of largest sources of metals and acidity within the Peru Creek watershed (McKnight and Bencala, 1990). Large sections of the Snake River and Peru Creek are coated in metal hydroxides primarily iron and aluminum hydroxides—which precipitate out of solution as flow from pristine tributaries raises the pH of the stream water. The Snake River joined by the

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tributaries Keystone Gulch and the North Fork of the Snake River, flows into Dillon Reservoir, which is a major water supply for the City of Denver. Keystone and Arapaho Basin ski resorts are located within the basin and use water from these streams for snowmaking.

The Pennsylvania Mine Site, a single location and one of the major contributors to metals loading in the Peru Creek Watershed is located at 39.6003191138959° North latitude and -105.809631386592° West longitude.

3.2 SITE HISTORY AND PREVIOUS WORK

The Snake River watershed, which drains a portion of the Continental Divide from Loveland Pass south to Webster Pass in east central Summit County, Colorado, has been the site of extensive environmental research in the past two decades. Water quality issues within the Snake River basin impact numerous users and present complex policy decisions. Much of the research has been focused on the upper tributary basin of Peru Creek (Middle Fork Snake River) which has been identified as a major contributor of metal solutes and acidity to the Snake River watershed through the processes of acid-rock-drainage (ARD). The Peru Creek basin contains several moderately sized historic narrow-vein silver/base metal mines that have been identified as discrete sources of metal loading. All of these mines are currently inactive. Water quality in Peru Creek is also affected by zones of diffuse and unidentified naturally occurring ARD related to extensive alteration of country rock directly associated with the ore deposits.

Initial environmental research was centered on the Pennsylvania Mine, the largest producing silver-lead-zinc mine in the Peru Creek area. Investigations were later expanded to include basin-wide and watershed scale sampling initiatives and abandoned mine inventories. The initial efforts led to more aggressive and area targeted assessments utilizing seasonal and synoptic constituent water sampling, streambed sediment analysis, and increased site mapping. In recent years more focused topical research including the innovative use of chemical tracers were initiated to reveal the complex interplay between anthropogenic and naturally occurring ARD on Peru Creek water quality. All of these studies have shed important additional information on seasonal fluctuation of metal loading dynamics that are central to understanding the Snake River basin as a whole. In addition, chemical tracer research has recently begun to address the possible effects of broader multi-year climatic fluctuations on discharge and metal loading from Peru Creek into the greater Snake River watershed greatly aiding the advent of sound basin-wide policy.

3.2.1 Initial Investigations/Mine Inventories/Site Assessments

The influences of the Pennsylvania Mine on the water quality of Peru Creek were investigated in 1984 through the collection of chemical data for select anion and cations. This study showed that effluent coming from the mine contained high concentrations of Ca, Mg, Al, Mn, Fe, and Zn. The study also suggested that dissolution of Al and Fe oxide in the streambed were pH dependant. This study provided an initial baseline for subsequent research (McKnight and Bencala, 1990).

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The Colorado Division of Mines & Geology (CDMG) sampled the Pennsylvania Mine effluent and briefly operated a small water treatment facility that attempted to improve Peru Creek water quality by raising pH through the addition of powdered limestone. This operation was unsuccessful due to adverse winter operating conditions, excessive metal precipitation in the plant and settling pond, and Clean Water Act liability fears.

Synoptic water sampling was performed in 1996 and 1997 at Peru Creek to investigate spatial and temporal variability in stream chemistry. Over this two-year study, significant changes in all measured solute concentrations were observed due to tributary inflow. The study documented summer increases in dissolved concentrations of metals by factors of 2 to 12 with decreasing flow and reduced dilution by snowmelt. These authors reported that dissolved metal concentrations in Peru Creek increase by a factor of 10 immediately at and below the inflow from the Pennsylvania Mine (Sullivan and Drever 2001).

Initial mine site inventory work and reconnaissance level water testing in Peru Creek was performed by the Colorado Geological Survey (CGS) in 1993 as part of an eight-year study of abandoned mined lands on United States Forest Service (USFS) administered property. Although this work was focused on public lands, some of the privately held mining properties impacting adjacent USFS lands and ultimately affecting Peru Creek were also investigated. Mine site inventory work included site mapping and on-site measurement of field parameters (pH & conductivity) with limited constituent water sampling. This study identified two areas of greatly increased environmental degradation: the entire Cinnamon Gulch tributary of Peru Creek and the abandoned Pennsylvania Mine (USFS 1994). In September 2000, the USFS requested CGS to perform a watershed characterization for Cinnamon Gulch and a more detailed investigation of the smaller mines found there; as well as a full site evaluation for the Pennsylvania Mine/Mill inventory area (Wood 2005). Results of this study will be discussed in the following section on Seasonal Investigations: High Flow/Low Flow.

In September of 2001, The US Environmental Protection Agency (EPA) commissioned URS Operating Services of Denver, Colorado to perform a Phase I Site Assessment (SA) for the Snake River Technical Support Project under START2-Region VIII. The objectives of this study included: 1) evaluation, characterization, and documentation of background water quality in the Peru Creek drainage; 2) evaluation, characterization, and documentation of potential release of metals to the environment from mine workings and mining wastes in Cinnamon Gulch and in Shoe Basin (Upper Peru Creek basin); and 3) evaluation of an existing water diversion design for Cinnamon Gulch (URS 2001).

A watershed scale study of the entire Snake River basin was conducted in 2001 and 2002 (Todd 2005). This study implicates the Pennsylvania Mine and Cinnamon Gulch as the major contributors to metal loading in Peru Creek and downstream in the Snake River basin. These authors were able to show an increase in dissolved metal concentrations at 2002 sample sites during low flow, indicating the importance of flow variation related to drought conditions in assessing remediation alternatives.

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3.2.2 Colorado Geological Survey Study – 2001

The Colorado Geological Survey (CGS) revisited selected sites in the Peru Creek drainage both in July and October of 2001 in an attempt to document changes in discharge and water quality during high and low flow regimes. A total of 39 water samples were collected during this study (19 high-flow samples in July, and 20 low-flow samples in October), including some in stream samples collected to bracket selected mines or groups of mines. These samples were all collected from within Cinnamon Gulch and from Peru Creek both above and below the Cinnamon Gulch outflow. No new samples were collected from the Pennsylvania Mine, as it was believed that pre-existing data from State of Colorado Division of Mines & Geology (CDMG) were available and sufficient (Wood 2005).

Direct measurements of surface water discharge from Cinnamon Gulch into Peru Creek are complicated by numerous alluvial fan distributaries at the mouth of this reach. For this study discharge was thus calculated indirectly by measurement in Peru Creek both immediately above and below the confluence of the various outputs from Cinnamon Gulch. Total discharge estimated in this manor for Cinnamon Gulch ranged between 460 gallons per minute (gpm) at low flow to 1,920 gpm at high flow. In Cinnamon Gulch itself this study recorded only 8.4-gpm discharge of surface water during low flow from discrete anthropogenic sources (9 mine adits), and 3-gpm discharge of surface water during low flow from identified natural sources (seeps & springs). This suggests that the overwhelming majority (97% to 98%) of total discharge from Cinnamon Gulch into Peru Creek is gained from ground water inflow (Wood 2005). Discharge from the Pennsylvania Mine ranged from 148 gpm at 162 gpm when measured by the CDMG in June and July of 1978 (Holm 1978). This is believed to have been a high flow event.

Throughout the Cinnamon Gulch watershed, pH ranged from a low of 2.91 to a high of 5.42, and 18 of 33 samples had a pH below 4. Due to the combined inflows from Cinnamon Gulch, Peru Creek was observed to suffer a decrease in pH and an increase in dissolved concentrations of Al, Cu, F, Fe, Mg, Mn, Si, Na, SO_4, and Zn at both high and low flow regimes. At many sample sites there were generally higher concentrations of most ions at low flow than at high flow, reflecting the expected dilution from runoff at high flow. A few sites displayed the opposite (higher concentrations during higher flow), possibly reflective of dampened recharge in the unsaturated zone. As with discharge, total metal loading from Cinnamon Gulch was also determined by subtracting values of samples from above and below the inflow zone into Peru Creek, both at low and high flow. Cinnamon Gulch gains close to 400 gpm discharge during low flow in its lower portion that is not attributable to anthropogenic sources, suggesting that this reach is a groundwater discharge zone affected by hydrothermally altered bedrock. Metal loading calculations for both anthropogenic and natural sources indicate that discrete sources throughout Cinnamon Gulch account for only about 4% of total metal loading at base flow and 5-8% of total metal loading at high flow (Bird 2003). The small percentage of groundwater capture by the abandoned surface mine workings in Cinnamon Gulch presents numerous remediation challenges (Wood 2005).

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High flow water samples from the Pennsylvania Mine portal in 1978 had a pH of 3.0 with constituent dissolved concentrations of the following metals: 12,000 parts per billion (ppb) Al, 10,600 ppb Cu, 27,000 ppb Mn, 76,000 ppb Zn, 83 ppb Pb, and .011 ppb Cd. Peru Creek above the Pennsylvania Mine and Cinnamon Gulch is relatively pristine with a recorded pH of 6.5 and dissolved constituent concentrations of 13 ppb Cu, 6 ppb Pb, 240 ppb Mn, and 150 ppb Zn as of June/July 1978 (Holm 1978).

3.2.3 URS/EPA Site Assessment (SA) – 2001/2002

A Phase I Site Assessment performed by URS Operating Services consisted of a number of synoptic sampling events spanning Peru Creek from its upper basin (Shoe Basin) to its confluence with the Snake River. The initial sampling event occurred on September 24-25, 2001 and was originally designed to establish a baseline prior to the removal of water from the North Fork Snake River for snowmaking at Arapahoe Basin ski area. Calculated low flow daily loading rates at the mouth of Peru Creek were about 119 Pounds Per Day (ppd), Al, 0.3 ppd Cd, 5.3 ppd Cu, 15 ppd Fe, 0.5 ppd Pb, 59 ppd Mn, 0.5 ppd Ni, and 70.6 ppd Zn. This study also concluded that the majority of metal loading to Peru Creek occurs in the middle reach in the area of the Pennsylvania Mine, Cinnamon Gulch, and from seeps along the creek downstream, including Warden Gulch. As in the CGS study, this first sampling event recognized a large contribution from non-discrete sources in the Cinnamon Gulch area to total metal loading in Peru Creek. The study also determined that although there is some degradation in upper Shoe Basin, winter season flows are so small that impact on Peru Creek is minimal to non-existent (URS 2001).

Phase II synoptic sampling was conducted on May 21, 2002 and was designed to capture a snapshot of high flow conditions. Discharge in Peru Creek at its confluence with Snake River was double the winter flow, however; both the 2001 and 2002 sampling were conducted during the longest duration drought period in central Colorado in 100 years. Results confirm the initial findings that the majority of metal loading occurs in the Pennsylvania Mine/Cinnamon Gulch area and that Peru Creek above this area is only slightly impacted. Metal loading for most constituents at the confluence of Peru Creek with the Snake River was significantly reduced during the high flow-sampling event, except for Fe and Pb. This study also removes Chihuahua and Thurman Gulches from suspicion of environmental degradation. Indeed, water quality in Peru Creek consistently improves at and below the mouth of Chihuahua Gulch (Wood 2005).

3.2.5 Tracer Experiments

A number of thought-provoking chemical tracer experiments have been conducted in recent years by graduate students of Diane McKnight, PhD, Institute of Arctic and Alpine Research (INSTAAR), University of Colorado-Boulder. These studies have reaffirmed some existing convention regarding environmental degradation and have added new areas of concern for metal loading into Peru Creek. The tracer experiments have also helped to clarify seasonal and climatic effects on metal loading through modeling of metal exchange between the in stream and hyporheic zones of Peru Creek. These

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experiments have also further clarified the relationship between metal transport and loading during years of drought conditions. The technique utilizes lithium injection in conjunction with synoptic sampling. Test were conducted in 2002, during severe drought conditions, and then again in 2004 and 2005.

2002 Tracer Experiment:
A lithium tracer experiment coupled with synoptic sampling was conducted along a 7 km stretch of Peru Creek on August 5-6, 2002. The purpose of this test was to determine the main discrete and diffuse sources of metal loading along the reach from just above the Pennsylvania Mine to the confluence with Snake River. A 4.5M lithium chloride (LiCl) injectate solution was introduced into Peru Creek at a rate of 80 mL/min for 22.5 hours prior to synoptic sampling to ensure plateau concentrations (Duren 2004). Tracer experiments determine discharge and enable a mass balance of water to be calculated enabling identification of gaining reaches, effluent inflows, pristine tributary inflows, and groundwater contributions. The conclusions of this study add to the list of suspect areas for degradation in Peru Creek. At the time of this study the predominant source of metal/sulfate loading into Peru Creek was from diffuse groundwater inflow in the vicinity of Warden Gulch. Through analyses of percent total results, the study suggests that complete removal of degradation from the Pennsylvania Mine would only reduce loading into Peru Creek for Al, Cd, Cu, Fe, Mn, Pb, Zn, and SO_4 by 11, 16, 27,7,18,7, 16, and 10%, respectively. By contrast, complete removal of the groundwater contribution from Warden Gulch would reduce loading of all metals and sulfate by 40%-80% into Peru Creek. It should be emphasized that this study was conducted in a severe drought year and that results must be viewed in light of this depressed flow regime. In addition, an intense rain event occurring the morning of the test is suspected of impacting results.

2004/2005 Tracer Experiments:
In order to document changes from the drought year of 2002, additional tracer experiments were conducted during 2004 & 2005. During the summer of 2004, a lithium tracer injection experiment focusing on the Warden Gulch area was executed to document effects on Peru Creek stream water chemistry during higher flows than in 2002. In the summer of 2005, another tracer experiment was conducted in the middle reaches of Peru Creek to document effects of the Pennsylvania Mine during a non-drought year. This second study included the application of the OTIS-P transient storage model with parameter optimization to quantify hydrologic changes between the 2002 drought year data and those collected during the higher-flow years of 2004-2005. Results indicated that compared to drought years, a return to more normal flow regimes may cause discrete tributaries to have a larger influence on metal loading and in stream metal concentrations in Peru Creek. In other words, higher flows lead to diminished loading and in stream concentration of metals from diffuse, generally non-anthropogenic sources. This was true for both Warden Gulch and for the Pennsylvania Mine. This study also discusses relationship between the metal storage capabilities of the hyporheic zone in Peru Creek during high and low flow years. During periods of more normal discharge, both the main channel and the hyporheic storage zone of Peru Creek are increased in area, allowing for diminished solute transport through dissolved metals retention in wetlands and beaver ponds, conspicuously absent in 2002. However, in both normal and

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low flow years, EPA toxicity threshold limits for aquatic life were exceeded for all dissolved metals at all sampled locations (Wong 2006).

3.2.6 Water and Sediment Study – Snake River

Between October 9 and 12, 2001 the USGS collected 36 surface water samples and 38 streambed samples from areas within the entire Snake River watershed. This study found low flow concentrations of several metals in both water and sediment to exceed aquatic life standards in various parts of the watershed. Zinc concentrations in water, corrected for the ameliorating effect of hardness, exceed the acute toxicity criteria for rainbow trout, and the chronic criteria for brown trout in the main stem of the Snake River, Peru Creek, and many upper tributaries affected by historic mining. Dissolved concentrations of copper and cadmium, corrected for the ameliorating effect of hardness, exceeded both the chronic and acute toxicity criteria for all species of trout in Peru Creek between the confluence of the Snake River and Cinnamon Gulch, as well as Cinnamon Gulch itself. Loading calculations from this study indict the entire reach of Peru Creek between the confluence with Snake River upstream to the Pennsylvania mine as the primary area where metal loads increase and are added to Peru Creek (Fey 2002).

3.3 SITE CHARACTERISTICS

3.3.1 Physical Geography

Cinnamon Gulch and the Pennsylvania Mine Site are located in the Rocky Mountains of central Colorado in alpine terrain characterized by steep-sided glaciated valleys and unvegetated rocky upland ridges and peaks. The site is located in a semiarid climate zone with the majority of annual precipitation falling as snow during the winter and spring.

Elevations range from about 10,800 feet above sea level at the confluence between the lower branch of Cinnamon Creek and Peru Creek, to 11,800 feet at the Silver Spoon Mine (apparent headwaters of Cinnamon Creek). The highest point in the Cinnamon Gulch watershed is 12,889 feet on Revenue Mountain. Cinnamon Gulch flows between Silver and Brittle Silver Mountains on the west, and Revenue and Decatur Mountains on the east.

3.3.2 Geology and Hydrogeology

The Pennsylvania Mine is developed along a vein hosted primarily in schistose rocks near the contact with the Montezuma Stock. Quartz schist, quartz-biotite schist, injection gneiss, granite gneiss, quartz monzonite, and granite are common. Galena is the most abundant mineral in the veins, with pyrite and chalcopyrite also common (Wood 2005).

Cinnamon Gulch lies on the northeastern margin of the Montezuma Stock, a 40 million-year-old (Oligocene) quartz monzonite porphyry. The Montezuma stock and its associated intrusions are part of a voluminous suite of porphyries that was emplaced 45-35 million years ago along the north-central Colorado Mineral Belt from Empire to Climax. The copper, zinc, lead, silver, gold and molybdenum-bearing hydrothermal

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systems of the area are related to intrusions of this suite of late Eocene-early Oligocene granitic intrusions. The stock intruded Precambrian hornblende gneiss and schist (Wood 2005).

The Montezuma Shear Zone, a band of argillized, sericitized, and pyritized rocks, passes through Cinnamon Gulch, and is presumed to have localized mineralizing fluids in the area. Zones of the most intensely altered rock show a strong spatial correlation with the Montezuma Shear Zone. Hydrothermally altered rock and sulfide veins are common throughout the district. Sericitic and propylitic alteration are common, and argillic alteration is present locally. The ore deposits within the district are dominantly silver-lead-zinc veins. Pyrite, galena, and sphalerite are the dominant sulfides, generally with tetrahedrite and chalcopyrite and less commonly with sulfosalts of silver and bismuth. Minor amounts of other sulfides are present, including chalcopyrite, bismuthinite, molybdenite, and sphalerite. Gold is mostly insignificant. Ferricrete and ferrosinter deposits are common in the areas underlain and flanked by altered rocks (Wood 2005).

Ground water is present in the fractured bedrock and the valley alluvium. Valley alluvial water ranges from being saturated at the ground surface to deeper depths, all depending on time of year and surficial topography. Water in the fractured bedrock ranges from less than 10 feet to hundreds of feet bgs, depending on the location of the fracture containing ground water (Wood 2005).

3.3.3 Hydrology

Peru Creek is the major tributary that drains the study area and then flows into the Snake River. Contributing drainages that flow into Peru Creek include but are not limited to the following: Chihuahua Gulch; Warden Gulch; Cinnamon Gulch; Falls Gulch; Horseshoe Basin and various other unnamed intermittent drainages. Peru Creek flows into the Snake River, which in turn flows into Dillon Reservoir, a municipal source of drinking water for the City of Denver. The drainage basin of Peru Creek is enclosed by the continental divide on the north, east, and south and encompasses approximately 20 square miles (URS 2002).

Stream gradients in the site area are very steep and stream flow rates are rapid. There is considerable variation in flow volumes between low-flow and high-flow regimes. Flow in Peru Creek between April and July 2006 ranged from a low of 37 cfs to a high of 128 cfs. Flow in Cinnamon Gulch between June and July 2006 ranged from a low of 1.8 cfs to a high of 9.1 cfs. Peru Creek and Cinnamon Gulch are major tributaries to the Snake River, the major stream in the area that flows into Dillon Reservoir. The Cinnamon Gulch watershed encompasses approximately 780 acres (EPA 2006; USGS 2007).

The closest stream gauge to the site is located on the Snake River near the town of Montezuma. The Snake River at Montezuma has an average annual stream flow of 61.8 cubic feet per second (CFS). Upstream of the site area, the Snake River has a drainage area of approximately 57.7 square miles (USGS 2007).

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4.0 PATHWAY ANALYSIS

4.1 WASTE CHARACTERISTICS

Approximately 45 American Geology Service (AGS) sites were identified in the Cinnamon Gulch watershed and the area around and including the Pennsylvania Mine (Figure 2). These sites have been identified as potential sources of metals loading to Cinnamon Gulch and the adjacent portion of Peru Creek. At these 45 AGS locations, 15 have been identified as sources where water drainage is ongoing (Figure 3). Two major sources of mine water discharge are located at the former Silver Spoon Mine site and at an adit on the east side of the gulch approximately 250 feet below the Silver Spoon Mine (USFS 1994). Additionally, the Pennsylvania Mine site has a significant draining adit discharging into a waste rock feature and tailings further down gradient of the waste rock.

Limited waste rock and tailings sample data exist for the greater portion of Cinnamon Gulch watershed. This data consists of various reports and characterizations, which are limited to observations and in some cases, limited analytical data on particular and not all encompassing waste piles. It has been reported that between 20 and 50 percent of the waste rock material in the Cinnamon Gulch watershed are sulfide minerals containing varying concentrations of aluminum, cadmium, chromium, cobalt, copper, lead, manganese, nickel, and zinc (Munroe 2000). These contaminants are prevalent as shown in surface water samples collected in Cinnamon Gulch and Peru Creek. Previous sampling by the USFS collected two samples of the mine waste rock from the 0 to 6-inch horizon and sample and two from the 6- to 12-inch horizon. At this particular pile, no evidence of chemical zoning was noted between these sample sets (Munroe 2000).

The AGS sites in the Cinnamon Gulch watershed occur within six mining areas as described below:

4.1.1 Decatur Town Site

The Decatur Town Site is comprised of one mining claim owned by May Louise Coors of San Luis Obispo, California. This claim is approximately 58 acres in size along the alluvial plain of Peru Creek. Mining features are limited to three small prospect pits and one waste rock dump. Waste rock mineralization is quartz, sericite and pyrite with iron oxides on the rock dumps. The waste dump is located 300 feet above Peru creek but no water discharge was observed. Several collapsed and erect wooden structures are found in this area (Wood 2005).

There is a high probability that mill tailings are located beneath the wetlands along Peru Creek on the southeast portion of the claim and in direct contact with alluvial ground water associated with Peru Creek. These alluvial tailings are not associated with any AGS identified site. AGS features present at or near the Decatur Town Site consist of AGS-63.

4.1.2 Brittle Silver Mill Site

The Brittle Silver Mill Site is located along Cinnamon Gulch approximately 200 meters upstream from the confluence of Cinnamon Gulch and Peru Creek. AGS features present

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at or near the Brittle Silver Mill Site consist of AGS-111 and AGS-113.

Effluent from the caved adit at the Brittle Silver Mill Site emerges from a rocky area believed to be on UFS land above the Brittle Silver Mill site and flows through the old timbered ruins of the collapsed portal and infiltrates into Brittle Silver Mill Site mine waste dumps. The effluent is clear and supports grass and moss below the portal ruins. Minor amounts of reddish precipitate were observed on rocks in the channel. Mostly gravel to finer size material cover the surface of the waste dump estimated to contain approximately 500 to 2,500 cubic yards. Abundant pyrite, minor to moderate sphalerite, and galena are present in these waste dumps. A retaining wall partially retains waste material at the mine waste dump from eroding to the area below (Wood 2005).

Water samples (high-flow sample CG-01-4 and the low-flow counterpart CG-01-35) collected in Cinnamon Gulch Creek below the tailings, and just before the confluence with Peru Creek, had pH, aluminum, cadmium, copper, lead manganese, and zinc exceeding State standards (Wood 2005).

4.1.3 Delaware Mine Area

The Delaware Tunnel is located near the north end of the Silver Spoon area. Located on the western slope of Decatur Mountain about ½ mile south of Peru Creek, the Delaware Mine was one of the earlier producers in the area (AGS 2003; Wood 2005). AGS features present at or near the Delaware Mine Site consist of the following source groupings: AGS-33, AGS-35, and AGS-51.

The adit at the Delaware Tunnel is on the east side of FR #262 about ¾ of a mile south of the Peru Creek crossing. A mine waste dump is present and has effluent emerging from the portal area of a completely caved adit. The effluent is relatively clear, but has deposited an orange precipitate and supports the growth of moss. Low dissolved oxygen at the portal (1 ppm) indicates that minimal oxygen is able to migrate from the surface into the caved adit. From the portal area, the effluent flows down the adit trench and ditch into a settling pond constructed on the western side of FR #262. Water from the settling pond appears clear and flows over a steep, 15- ft-high embankment and enters Cinnamon Creek. The combined length of the adit trench and ditch is about 280 feet (AGS 2003; Wood 2005).

A portion of the effluent overflows the ditch and infiltrates into the bulldozed portion of the dump and road. This dump contains approximately 250 to 450 cubic yards of mostly gravel size material and has been extensively bulldozed and mixed with other material (AGS 2003; Wood 2005).

Two adits, one of which appears to be on private land and one on USFS, discharge water. In July of 2001, the flows from the two adits were measured at 4.6 and 7.6 gpm, respectively. In October of 2001, the flows were 1.5 and 3.4 gpm. Water samples were collected from both adits during both sampling events (CG-7 and CG-9, and their low-

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flow counterparts CG-30 and -33). The pH of the samples ranged from 3.62 to 4.92, with conductivity ranging from 177 to 629 µS/cm (Wood 2005).

Constituents exceeding State water quality standards included dissolved aluminum, cadmium, copper, iron, manganese, and zinc in all four samples; and dissolved lead in three samples (Wood 2005).

Effluent from adit on USFS land contained higher metal concentrations than the adit on private land with the exception of aluminum and lead. The low flow sample at adit on private land (CG-9) had the highest lead concentration of all Cinnamon Gulch samples at 560 µg/L. High-flow samples CG-7 and CG-9 have overall higher metal concentrations than low-flow samples CG-3- and CG-33 (Wood 2005).

Waste rock dumps associated with the two adits discussed above were the only dumps inventoried in the Rich Ore Lode and Silver Spoon Mine areas during the CGS field work. The volume of material on the dumps was estimated in 1993 at 770 and 450 yards, respectively. The inventory reports that water was draining across the surface of both dumps. The toe of the dump on private property is in direct contact with the surface water of Cinnamon Gulch, so it is reasonable to assume that weathering of the dump could be releasing contaminants to the creek (Wood 2005).

4.1.4 Brittle Silver Group

The Brittle Silver group includes five patented lode claims (Brittle Silver, Little Nell, White Sparrow, Boston And Peavy) on the eastern side and near the top of Brittle Silver Mountain; and the patented Brittle Silver Mill Site, located between the Brittle Silver lode claims and Peru Creek (Wood 2005). AGS features present at the Brittle Silver Group consist of the following source groupings: AGS-128, AGS-130 through AGS-138, AGS-174 and AGS-178. It should be noted that the Brittle Silver Mill Site referenced in the Delaware Site area is not included in the Brittle Silver Group area, but is further downgradient near the confluence of Cinnamon Gulch and Peru Creek.

The adit at the Psiupsilon Tunnel and the adit at the Little Nell claim both discharge water. In July of 2001, the flows from the two adits were measured at 2.5 and 2.4 gpm, respectively. In October of 2001, the flows were 1.5 and 0.85 gpm. Water samples were collected from both adits during both sampling events (CG-6 and -8, and their low-flow counterparts CG-37 and -31). The pH of the four samples ranged from 4.15 to 5.42 with conductivity ranging from 75 to 130 µS/cm. Constituents exceeding State water quality standards included dissolved zinc in all four samples, dissolved cadmium in three of the four samples, dissolved aluminum and lead in two of the four samples, and dissolved copper and manganese in one of the four samples. Effluent from the Psiupsilon Tunnel had higher concentrations of metals during low-flow, whereas effluent from the Little Nell had higher concentrations during high-flow (Wood 2005).

Samples of the tailings and waste rock at the Brittle Silver Mountain site contained detectable gold, silver, and mercury, and relatively high concentrations of copper (723

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ppm and 937 ppm), lead (1.6% and 2.6%), and zinc (0.4% and 0.7%) and detectable levels of gold, silver, and mercury. Samples had net acid generating potentials ranging from –6.7 tons CaCO3 to –14.2 tons CaCO3 per 1000 tons. Acidic paste pH ranged from 3.83 to 5.05. These results indicate that the tailings are capable of releasing ARD in the present and in the long term (Wood 2005).

Due to the remote location of this grouping on the mountainside, these areas will be grouped together in three composite areas as shown in Table 2.

4.1.5 Silver Spoon Mine Area

The Silver Spoon Mine area includes the Rich Ore Load area. This area is located high in the rugged terrain of Cinnamon Gulch, which requires considerable effort to reach. There are no fences or signage I this region of Cinnamon Gulch. AGS features present at the Silver Spoon Mine Area consist of the following source groupings: AGS-15 through AGS-20, AGS-22, AGS-23, AGS-44, AGS-45, AGS-148, AGS-149, AGS-151, AGS-152, AGS-211, and AGS-213

At the Silver Spoon Mine, mine waste rock covers an area of approximately 50 feet by 50 feet and reaches a height of approximately 20 feet. It is estimated that the volume of mine waste rock at the location is approximately 1,000 cubic yards. The material at the Silver Spoon Mine waste rock pile was an unsorted mixture of angular mining rubble that is in size and coloring similar to the surrounding material mantling the hillside. Coarse angular rocks cover the mine waste rock pile and the finer grained material appears to have infiltrated into the depths of the waste rock pile. Mine waste water discharge from the Silver Spoon Mine flows along the north flank of the waste rock pile and is eroding and transporting some of the waste rock material. The majority of waste rock piles in the Silver Spoon area similar to this site.

Mine waste piles contain abundant amounts of quartz vein pyrite and moderate amounts of sphalerite, and less common galena. Galena from mines in this area contains high silver concentrations (Wood 2005).

Samples of mine waste water discharge collected in Cinnamon Gulch from the Silver Spoon Mine and approximately 250 feet downgradient of the Silver Spoon Mine had an estimated low flow of 2 to 3 gallons per minute, a pH of 3.33 and a conductivity of 471 µS/cm. Metals concentrations of aluminum, cadmium, chromium, cobalt, copper, lead, manganese, nickel, and zinc contamination were potentially elevated with respect to Cinnamon Gulch and Peru Creek (Wood 2005).

Four separate adits in this area discharge water along the Rich Ore Lode and have a combined flow of approximately 4.5 gpm. Red orange precipitate is prevalent where water is draining from a mine feature. The pH ranged from 3.16 to 3.58 with a conductivity ranging from 287 to 611 µS/cm. In October of 2001, only one of the four adits was flowing at a rate of about 1 gpm. Standing water was present at the other three

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adits. The pH ranged from 2.91 to 3.21 with the conductivity ranging from 464 to 593 µS/cm (Wood 2005).

In 2004, water samples were collected from all adits with flowing or standing water. Numerous constituents exceeded State water quality standards in the effluents from the adits along the Rich Ore Lode. Dissolved aluminum, cadmium, copper, iron, lead magnesium and zinc-exceeded standards in all eight samples. In addition, dissolved nickel-exceeded standard in seven samples and dissolved silver and sulfate in two samples (Wood 2005).

Water collected at low flow from Cinnamon Gulch Creek downstream from the influence of the Rich Ore Lode workings. The stream at this sample site includes discharge from both the Rich Ore Lode and the upgradient Silver Spoon Mine, as well as natural sources in the area. Stream flow was 56 gpm with a pH of 3.35 and a conductivity of 258 µS/cm. This sample exceeded State water quality standards for dissolved aluminum, cadmium, copper, iron, lead, manganese, nickel, and zinc. Red-orange precipitate was visible in the channel (Wood 2005).

Samples CG-01-11 (high flow) and CG-29 (low-flow), CG-12, CG-26, and CG-28 were collected from natural springs or streams in the Silver Spoon Inventory Area. The combined flow from these sources was 17 gpm at low flow with a pH ranging from 3.39 to 4.47 and conductivity ranging from 84 to 318 µS/cm. Constituents exceeding State water quality standards included dissolved aluminum, cadmium, copper, and zinc in all samples, dissolved manganese in four samples, and dissolved lead in three samples (Wood 2005).

Rock chemistry data from composite soils collected from the Rich Ore Lode area shows relatively large concentration and zinc, plus detectable gold, mercury, and silver. The negative net acid base potential and the acidic paste pH suggest that the dump will be acid generating over the long term (Wood 2005).

4.1.6 Pennsylvania Mine Site

AGS features present at the Pennsylvania Mine Site Area consist of the following source groupings: AGS-107 through AGS-110, and AGS-114 through AGS-120.

The Pennsylvania Mine Site is largely owned by claims held by Transpacific Tourism. There are numerous adits, waste rock dumps, mill sites and tailings ponds and impoundments. Characteristic mineralization for this mine is pyrite, chalcopyrite, galena, minor sphalerite, manganese and iron oxides on rock dumps. Metallic mining equipment (boilers, pipes) is found throughout the area. Historic wood mining facilities remain on site. Two uncontained and untreated water sources are found uphill of the largest mine facility and drain into and through nearby waste dumps. A water treatment facility with passive wetlands captures heavy water flow from the lowest adit in the area. This facility is not operational.

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Waste rock dumps in the Pennsylvania Mine area have a potential for significant environmental degradation. Spring and mine drainage runs through the tailings and continues down the watershed at a rate of 1.5 gpm. A test of the drainage showed a pH of 4.1 and a conductivity of 800 µS/cm (Wood 2005).

The Pennsylvania mine portal is a source of perennial contaminated drainage that discharges to Peru Creek. The discharge ranged from 0.33 to 0.36 cfs (148 to 162 gpm) measured by CDMG in June and July of 1978. The pH ranged from 3.0 to 4.85, and conductivity ranged from 1,400 to 1,525 µS/cm. An additional potential major contaminant source is the infiltration through waste-rock dumps and tailings in the alluvial wetlands along Peru Creek. Combining the surface areas of all the waste rock dumps and tailings piles in the Pennsylvania Mine area gives a cumulative surface area of almost 50,000 sq ft. Assuming 36 inches of annual precipitation, it is possible that over 2 gpm is infiltrating through the dumps and tailings and leaching contaminants to the alluvial groundwater and Peru Creek (Wood 2005).

In 1985, the Colorado Division of Mined Land Reclamation constructed a diversion system for the Pennsylvania Mine effluent. The following year, the Colorado School of Mines, diverted the effluent from the Pennsylvania Mine into a natural wetland below the Pennsylvania Mill. The project was aimed at evaluating the capability of the wetland in removing metal concentrations. A plumbing system was built in 1990 to handle effluent from the Pennsylvania Mine. Effluent from the caved portal enters a grated sewer inlet. Most of the effluent was piped about 1,000 feet to a cinder-block building and into a settling pond. The building houses an inactive lime/limestone dosing system designed to add alkalinity to the mine effluent. The effluent was then directed into the settling pond to allow metal oxy-hydroxide floc to settle out of solution. Water from the settling pond flows into a wetland area next to Peru Creek. The grassy wetland area contains mill tailings from the adjacent Pennsylvania Mill. Two lined ponds northeast of the settling pond, not present in 1993, did not appear to be in use. One was dry; the other was filled with water. Iron deposits in the pipeline have apparently constricted the pipe and reduced its capacity to remove all of the effluent collected at the portal. The system apparently was draining all of the effluent in 1993, however the capacity was exceeded in 2001. A pond was formed over the inlet and some of the effluent overflowed onto the dump. This dump contains an estimated 17,700 cubic yards waste rock or tailings (Wood 2005).

The treatment system constructed at the Penn Mine was a “passive hydro powered” chemical treatment system capable of removing heavy metals to near in stream standard levels. This project was funded on a demonstration basis through the Non-Point Source Program of the Clean Water Act (§ 319). The initial design specified the use of powdered limestone as a neutralizing agent delivered using a hydro-powered (mechanical drive) combination hopper and ribbon blender/chemical feeder. Limestone addition rate was initially designed be normally set to be linearly related to mine water flow, however the turbine speed could be adjusted by a nozzle adjustment. The limestone-mine water mixture was designed to run out of the treatment building which housed the turbine and ribbon blender-feeder, through a sluice in the floor, pass down a 75-100 foot long rip-rap

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channel) to provide mixing and limited aeration), and then enter the settling pond for sludge removal (Trout Unlimited 2007).

The system was constructed in 1990, but only operated for a short time due to chemical inefficiency, mechanical problems and materials of construction failures.

Boulder Innovative Technologies conducted the next study. A water quality baseline and design basis lab and field piloting program began in March 1993 and ran through early 1994. The overall program envisioned two subsequent phases. Phase II was to be a design and construction of the revised full-scale chemical pre-treatment and a polishing biological system. Phase III was to be the operational demonstration of the installed revised system. During calendar years 1993 and 1994 a detailed investigation was conducted to fully characterize the chemistry and metal loading from this drainage and to conduct a comprehensive series of laboratory and field piloting treatability studies. A very comprehensive water quality sampling, flow sampling and data interpretation of the Pennsylvania Mine drainage was conducted. Year round data collection provided a very good understanding of the seasonal variation in both flow and chemistry from the drainage. It was learned that the drainage is very responsive to the snowmelt, with both high flows (over 150 gpm) and the most degraded water quality occurring during June and July. Annual metal loadings are the highest during this period. Flows in the fall and winter diminish to 20-40 gpm or less. This major swing in seasonal loading has significant impact on the design and selection of treatment approaches and on forecasting of treatment costs (Trout Unlimited 2007).

Laboratory work during the late winter and spring of 1993 was conducted using samples of mine water. A series of screening chemical neutralization tests were conducted to define dose requirement to chemically precipitate the toxic metals from the acidic water. Characterization of the produced chemical sludge from these tests was also performed. Using both raw mine water and partially chemically neutralized mine water, a series of ion exchange polishing tests were performed using natural zeolites and chabcarb, as combination natural zeolite-activated carbon ion exchanger (Trout Unlimited 2007).

Based on the laboratory screening studies, a field pilot treatability study was designed and equipment was procured, fabricated and installed at the mine site during June 1993. The pilot operation consisted of a field camp, field laboratory and pilot plant sized for approximately 1-gpm flows. The field-piloting program consisted of a series of chemical dosing and sludge characterization studies with selected neutralizing agents. Agents tested included lime, limestone, trona, calcined trona, soda ash, magnesium oxide, calcium polysulfides and Trapzene a proprietary calcium peroxide chemical. Several pieces of equipment were also tested for operational characteristics including the water powered Aquafix dry chemical feeder, and an Accurate volumetric dry chemical feeder (Trout Unlimited 2007).

A set of ion exchange experiments with the natural zeolite, clinoptilolite and Chabcarb was conducted using mine water, which had been partially treated with various chemical neutralizers. The ion exchange tests were conducted in 1.5” diameter columns, with a

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single test conducted in 8" diameter columns (Trout Unlimited 2007).

Engineering interpretations of the data derived from the laboratory and field piloting resulted in recommendation for a treatment system to minimize the consumption of neutralizing chemicals and the resultant sludge. The recommended chemical for partial neutralization for this site was determined to be reactive grade magnesium oxide (MgO). While more expensive, MgO was found to produce a rapid settling dense sludge with only 25% settled volume compared to more traditionally used chemicals such as lime and limestone. This was determined to be a major factor in the secondary costs of sludge disposal and on constraints on the sludge-settling pond (Trout Unlimited 2007).

Ultimately, the treatment system which was recommended for Pennsylvania Mine as a result of this study and a follow up conceptual sludge disposal feasibility study involves a partial neutralization with reactive grade magnesium oxide, settling of the sludge's in a settling pond, followed by final treatment in sulfate reducing bacteria (SRB) beds. The partial chemical neutralization is projected to extend the operating life of the SRB system and improve its performance by a reduction in the influent loading of iron and aluminum, plus providing an influent with a pH more suitable for the optimal performance of the sulfate reducing bacteria. The selected chemical sludge disposal system for such a combined treatment system is pumping to the bullheaded portions of the underground workings of the Pennsylvania Mine (Trout Unlimited 2007).

At the conclusion of this study, work commenced on the construction of the wetlands associated with the pre-treatment. A major project was established with Volunteers for Outdoor Colorado for the weekend of August 6-7, 1994. Meanwhile, on December 22, 1993 the EPA sent a letter to the state of Montana, which outlined the EPA policy on discharges from abandoned mines. Notwithstanding the fact that the Pennsylvania Mine discharge was dumping metal laced water into Peru Creek with no interest from EPA, this letter was interpreted to mean that if the state of Colorado began to treat the mine discharge, they would need to obtain an NPDES permit and could be responsible for the treatment of the waste stream in perpetuity. As a result of this potential liability, work on the treatment system was stopped (Trout Unlimited 2007).

Further work was then put on hold in early summer 1998 due to lack of progress on the liability issue. With no resolution in sight for the liability issues, DMG closed out the Pennsylvania Mine project on November 16, 1998 (Trout Unlimited 2007).

4.2 GROUND WATER PATHWAY

A review of groundwater well records maintained by the State of Colorado Division of Water Resources, State Engineers Office, indicates that there are no groundwater wells for any beneficial use permitted in the Peru Creek Drainage. State records indicate that 79 groundwater wells have been permitted in the town site of Montezuma, approximately 2.75 miles west, southwest of the Peru Creek Mine sites (Colorado Division of Water Resources 2001). The town site of Montezuma is located in the valley of the Snake River, upgradient of Peru Creek and there is no known mechanism to permit mixing of groundwater from the Peru Creek Valley with that from the Snake River Valley

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upgradient of the confluence of the two streams. Four other groundwater wells are permitted along the Snake River downstream of the confluence of Peru Creek with the Snake River within four miles of the site (Colorado Division of Water Resources 2007).

To date, no known ground water wells have been installed or sampled as part of any site characterization in Cinnamon Gulch or the Pennsylvania Mine. There are beliefs that the ground water plays a potential significant role in contaminant contribution to surface water. Characterization of ground water in the vicinity of Peru Creek will aid in the understanding of what the substrate consists of as well as what contaminants are present in the shallow alluvial ground water.

4.3 SURFACE WATER PATHWAY

Peru Creek is the major tributary that drains the study area and then flows into the Snake River. Contributing drainages that flow into Peru Creek include but are not limited to the following: Chihuahua Gulch; Warden Gulch; Cinnamon Gulch; Falls Gulch; Horseshoe Basin and various other unnamed intermittent drainages. Peru Creek flows into the Snake River, which in turn flows into Dillon Reservoir, a municipal source of drinking water for the City of Denver. The drainage basin of Peru Creek is enclosed by the continental divide on the north, east, and south and encompasses approximately 20 square miles (URS 2002).

There are extensive palustrine scrub-shrub and emergent wetlands located along Peru Creek. Multiple small isolated semi-permanent palustrine open water wetlands are located along Peru Creek downstream of the site to Peru Creek's confluence with the Snake River. Approximately six acres of streamside wetlands are located along the Snake River from the confluence of Peru Creek to the inlet of Dillon Reservoir. There are small upland wetland areas located in upper Cinnamon Gulch near the Silver Spoon Mine and associated adits (URS 2002).

There are no recorded surface water intakes along Peru Creek. While Peru Creek and the Snake River are not stocked by the Colorado Department of Fish and Wildlife, Keystone resort stocks portions of the Snake River with trout species in and around the Keystone area. Trout are present in Chihuahua Gulch (URS 2002).

The rock chemistry data from waste rock dumps and tailings in the watershed consistently show negative net acid-base potential and acidic paste pH, as well as enrichment of copper, silver, lead, and zinc, plus detectable gold and mercury. The data suggest that these features will be acid generating over the long term (Wood 2005).

Due to the inflow from Cinnamon Gulch, Peru Creek experiences a drop in pH and increases in concentrations of Al, Cu, F, Fe, Mg, Mn, Si, Na, SO4, and Zn. This indicates that Cinnamon Gulch perennially has a lower pH than Peru Creek, and higher concentrations of these constituents. Water from Cinnamon Creek, after mixing with Peru Creek below the lowest tailings pile produces reddish-orange precipitate coating on the streambed of Peru Creek. Upstream from this mixing zone the Peru Creek streambed has a white precipitate coating (Wood 2005).

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Discharge in the Cinnamon Gulch watershed ranged from just over 1 cfs (460 gpm) at low flow to 4.3 cfs (1,920 gpm) at high flow. Numerous ions are consistently elevated in samples from around the watershed. Average dissolved concentrations of selected metals at low flow were 5.5 mg/L aluminum, 5.5 mg/L manganese, 2.8 mg/L zinc, 0.014 mg/L cadmium, 0.26 mg/L copper, and 0.082 mg/L lead. Numerous ions exceeded State of Colorado Water Quality Standards for chronic aquatic life exposure. At low flow, zinc exceeded the standard in all samples; copper and lead exceeded the standard in all but two samples; cadmium and manganese exceeded the standard in all samples; iron and nickel exceeded the standard in about half of the samples; and aluminum exceeded the standard in all but one sample. Sulfate failed the secondary drinking water standard in two samples (Wood 2005).

Comparison of water quality at low-flow versus high-flow shows some peculiarities in the data. At some sample sites, the data reveal the expected, i.e., higher concentrations of most ions at low flow than at high flow, due to dilution from runoff at high-flow. However, other sites show the opposite, i.e., higher concentrations at high flow. The ability of the unsaturated zone to dampen recharge pulses is postulated as a possible explanation. Loading calculations indicate that anthropogenic contributions are about 4% during low flow and 5-8% at high flow. Iron and aluminum precipitate out of solution and onto the streambed in the Cinnamon Gulch watershed and in Peru Creek along the reach where Cinnamon Gulch enters Peru Creek. Lead is attenuated as well, likely by adsorption onto precipitated iron and/or aluminum phases along these reaches (Wood 2005).

The lower reach of Cinnamon Gulch is apparently a ground-water discharge zone, because the creek gains almost 400 gpm between sample site CG-27 and its confluence with Peru Creek, but discrete measurable sources along this reach total less than 8 gpm. Minimal dilution occurs along this reach, suggesting that contaminants are being transported in the subsurface to the ground-water discharge zone. The source of these contaminants could be mostly natural, with the conclusion that remediation of surface features, and removal of sources of surface water contamination, might not remove the major sources of contamination from the watershed. The relatively low proportion of ground-water flow that is intercepted by underground workings is problematic from a remediation perspective. It is encouraging in that a very small proportion of the total water budget is discharged from adits, which are difficult to remediate, but discouraging in that removing the adit discharges would remove only a small percentage of the total contamination in the watershed, and would likely have minimal impact (Wood 2005).

The discharge from Cinnamon Gulch at its mouth cannot be directly measured due to the numerous alluvial fan tributaries into which the creek splits before its confluence with Peru Creek. However, the total discharge from Cinnamon Gulch was calculated by measuring the discharge in Peru Creek above and below the various outputs from Cinnamon Gulch. Thus, the discharge from Cinnamon Gulch ranges from 460gpm at low flow to 1,920 gpm at high flow. At low flow, approximately 8.4 gpm is from discrete anthropogenic sources (9 mine adits), and 3 gpm was measured from discrete natural sources in the watershed. The balance of approximately 449 gpm represents the amount of flow that Cinnamon Gulch gains from ground-water discharge, which amounts to 157 gpm above sample site CG-32, and 292 gpm below CG-32 (Wood 2005).

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4.4 SOIL EXPOSURE PATHWAY

All locations where mine waste occurs are not fenced and are accessible by recreational hiking. A four-wheel drive trail up Cinnamon Gulch provides easy access to areas up the drainage. Persons accessing the site area are exposed to potential site contaminants on a transient basis.

URS Operating Services conducted a Site Inspection of the Upper Peru Creek (Shoe Basin Mine) and Upper Cinnamon Gulch (Silver Spoon Mine). As part of the soil sampling that was conducted, a background soil sample (SR-MWR-01) was collected in Cinnamon Gulch from a grassy hillside location topographically above the mining activity associated with the Silver Spoon Mine. A review of the analytical data from the background sample and from samples collected from mine waste rock piles in Cinnamon Gulch indicate that metals concentrations in the mine waste rock piles are above background levels (URS 2002).

5.0 DATA QUALITY OBJECTIVES

The EPA Data Quality Objectives (DQO) Process is a seven-step systematic planning approach to develop acceptance or performance criteria for EPA-funded projects. These DQO's were co-developed by CDPHE and EPA based on a Data Gap Analysis on data that are currently available and data that are needed in this evaluation of the Cinnamon Gulch and Peru Creek watersheds. The mine waste characterization portion will focus on the contribution of metals and acidic conditions to adjacent water bodies. The water quality portion of this sampling event will focus on identifying the chemical and biological health of Peru Creek and its tributaries.

Based upon the potential risks associated with the potential hazardous substances, the soil exposure and surface water pathway with a ground water-to-surface water component are of the highest concern. Limited to no analytical data are available to characterize specific problem areas for potential removal or remedial actions.

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5.1 Step 1 – The Problem Statement

Water quality issues within the Snake River basin impact numerous users and present complex policy decisions. Much of the research has been focused on the upper tributary basin of Peru Creek (Middle Fork Snake River) which has been identified as a major contributor of metal solutes and acidity to the Snake River watershed through the processes of acid-rock-drainage (ARD). All mines in the Cinnamon Gulch and Peru Creek drainages are currently inactive. While the site area contains several moderately sized historic narrow-vein silver/base metal mines that have been identified as discrete sources of metal loading, it also has numerous potential areas where diffuse and unidentified naturally occurring ARD occurs. In order to determine the waste piles that may be loading sources to Peru Creek, to evaluate potential risks to human health and ecological receptors, and to further evaluate the nature and extent of contamination associated with Cinnamon Gulch and the Penn Mine, additional hydrological, analytical, and biological data are needed. These data will also be used in evaluating potential remedies associated with reduction of metal loading into Peru Creek that ultimately effect water quality in the Snake River.

5.2 Step 2 – Identifying the Decision

The purpose of this step is to define the decision statements that this study will attempt to resolve. There are two primary study questions for this investigation including Principle Study Question (PSQ) 1: factors associated with loading into Peru Creek/Snake River waterbodies: Which mine waste piles in the Cinnamon Gulch watershed are in direct contact with surface water and are eroding or transporting contaminants to adjacent surface water via erosion or leaching from mine water features or precipitation? Are there specific mine waste piles in the Cinnamon Gulch watershed that are contributing contamination significantly more than others. Are there remedial options to limit this contaminant migration? Is alluvial ground water in direct contact with mine waste along Peru Creek and if so, what are the extent and volume and location of these alluvial tailings; What is the loading of metals into Peru Creek and the Upper portion of Snake River from Pennsylvania Mine, Warden Gulch and Cinnamon Gulch during high flow and low flow conditions? and; 2) Are contaminant concentrations in Pennsylvania Mine and Peru Creek resulting in measurable human health and /or ecological impacts?

5.3 Step 3 – Identifying the Decision Inputs

The information that is required to arrive at a decision for PSQ 1 includes:

Identification of specific mine waste piles in the Cinnamon Gulch watershed are in direct contact with surface water and are eroding or transporting contaminants to adjacent surface water via erosion or leaching from mine water features or precipitation;

- Analytical data from soil and waste for identification of specific mine waste piles in the Cinnamon Gulch watershed that are contributing to surface water and or ground water contamination significantly more than others;
- Field and analytical data from surface water and sediment collected from Cinnamon Gulch and Peru Creek as well as up-gradient and down-gradient locations;

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- Adit and stream flow measurements of surface water in Peru Creek, Cinnamon Gulch, Warden Gulch, and Snake River
- Analytical data from ground water wells located adjacent to or within the alluvial tailings along Peru Creek;
- Analytical data from background surface soil samples;

The information that is required to arrive at a decision for PSQ 2 includes:

Identification of human health and ecological risks associated with impacts from the Penn Mine, Cinnamon Gulch, and Peru Creek via direct contact with surface soil/mine waste, surface water and sediment.

- Analytical data from surface soil;
- Field parameters (pH, conductivity, temperature, dissolved oxygen) and analytical chemistry data for surface water and sediment from upstream areas unaffected by Penn Mine and Cinnamon Gulch
- Surface water and sediment analytical data adjacent and downstream from mining inputs
- Macroinvertebrate populations/assemblages in downstream areas of Peru Creek and Snake River and upstream from unimpacted tributaries;
- Toxicity testing to fish species likely to reside in the watershed;
- Current applicable soil, surface water, and sediment human health and ecological criteria or benchmarks

5.4 Step 4 – Defining the Study Boundaries

Spatial Boundaries: PSQ 1: The study area for mine waste sample collection is shown on Figures 1 through 3 and incorporates the entire watershed of Cinnamon Gulch and the area surrounding the Pennsylvania Mine and its associated alluvial tailings located along Peru Creek immediately up gradient of Cinnamon Gulch.

PSQ 2: The study area for water, sediment, toxicity, and macroinvertebrate sample collection is shown in Figure 4 and includes locations on Peru Creek and on the Snake River, and several tributaries to these watersheds, including Cinnamon Gulch.

Temporal Boundaries: Field activities are expected to occur during or immediately following runoff (high-flow) and low-flow conditions of the summer of 2007. The low-flow field-sampling event will incorporate a limited subset of sampling locations as compared to the high-flow runoff-sampling event. This limited subset will be based on data interpretation of the high-flow runoff data.

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5.5 Step 5 – Developing A Decision Rule

The Decision Rule that is required for PSQ 1 is:

If surface water samples from Cinnamon Gulch identify increases of contaminants incrementally in Cinnamon Gulch, potential sources are likely present and will be further explored for remedial alternatives. Mine waste piles with water features (erosional or draining features) and waste characteristics that indicate they are readily available for contaminant leaching to surface or ground water will be assessed for potential remedial alternatives.

The Decision Rule that is required for PSQ 2 is:

Historical field and analytical data indicate that contaminants are detected in surface water above AWQC, therefore toxicity testing and macroinvertebrate population metrics will be conducted to determine the level of effects to aquatic life in Peru Creek and the upper reaches of the Snake River.

If toxicity results indicate that water in the study area is toxic to trout species, the degree of cleanup needed to establish and maintain a healthy trout and macroinvertebrate population will be determined. Options to reach this cleanup goal will then be explored to see if it can be realistically achieved given naturally occurring metals and pH contributions to the watershed and budgetary constraints.

Inputs Needed for Decision Rules

- Analytical and physical characteristics of mine waste piles;
- Comparison of soils analytical data to open space exposure standards for evaluation of potential risks to human health and to ecological soil screening levels for ecological receptors;
- Comparison of field parameters (pH, conductivity, temperature, dissolved oxygen) and analytical chemistry data (including metals, hardness, alkalinity, and DOC) for surface water and sediment, to upstream areas unaffected by Penn Mine and Cinnamon Gulch;
- Comparison of surface water and sediment analytical data to applicable MCL's/AWQC/State WQS for human health and AWQC/State WQS/surface water and sediment screening level ecological benchmarks;
- Evaluation of macroinvertebrate assemblages in the watershed and unimpacted tributaries;
- Determination of toxicity levels to fish species likely to reside in the watershed;

5.6 Step 6 – Defining Tolerance Limits On Decision Errors

Since limited analytical data are available to document the presence or absence of discreet sources of contamination, judgmental mine waste sampling will be used to bias samples toward more potentially contaminated areas. Statistical sampling will not be used and a tolerance limit on decision errors will not be calculated. All sampling activities will be conducted according to

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CDPHE and EPA Standard Operating Procedures (CDPHE 2000, EPA 2002). Composite soil samples will be collected to more accurately reflect the average concentration over the sampling areas. All analytical data will be reviewed, verified, and validated to ensure data are acceptable for the intended use following CDPHE and EPA protocols (CDPHE 2000, EPA 2002).

5.7 Step 7 – Optimizing the Sample Design

The purpose of this step is to identify the most resource-effective data collection design for generating data that are expected to satisfy the DQO's specified in the preceding six steps. A comprehensive sampling plan including both abiotic and biotic media has been designed to identify waste, estimate waste loading into adjacent water bodies, determine associated water quality and toxicological effects that are representative during both high-flow and low-flow events. The sample design includes several aspects, such as synoptic sampling and collecting biotic and abiotic parameters during the same time frame to ensure efficient and effective data are being collected for achieving the remedial goals as follows:

- Waste Piles: Aerial photographs and site specific knowledge in the initial identification of waste piles; All piles will be located with GPS to allow for accurate identification of sources; Using established prioritization criteria, only those waste piles expected to be impacting water quality will be sampled;
- Waste Tailing Delineation: Estimates of volumes and extent of tailings deposited in and along Peru Creek will be made to allow for potential future remedial actions in this location;
- Groundwater: Groundwater quality will be assessed along Peru Creek and in the upper reaches of Cinnamon Gulch for potential assessment of the ground water to surface water contribution;
- Surface Soil: Mine waste piles will be sampled for contaminant characteristics and potential contaminant attribution to regional surface water quality;
- Surface Water: Existing sample locations were selected to assist in maintaining data comparability and assess trends during both high flow and low flow conditions; locations established to enable the ability to assess surface water quality contributions from tributaries,
- Sediment: further determine nature and extent of contamination;
- Field Parameters: measured insitu at all locations during sampling to assist in accurate evaluation of water quality and potential impacts
- Toxicity Tests: evaluate the toxicological impacts of contamination; locations selected to measure effects downstream of potentially contributing tributaries; dilution series based on assessing load reductions; test species used represent end-use objectives; testing conducted using water most representative of prolonged exposure conditions expected at the site (low flow conditions)
- Macroinvertebrates: Locations are selected based on representative habitats and data will enable an evaluation of the biological condition of the study area.
- Fish Survey: Survey locations are areas where historical surveys indicate healthy populations. Surveys will be limited only to those areas where populations are not known.
- Quality Assurance: both field and laboratory protocols are being implemented for this

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study to ensure data usability.

6.0 FIELD PROCEDURES

6.1 CONCEPT OF OPERATIONS

6.1.1 Schedule

Fieldwork for this SI is tentatively scheduled to begin the week of July 9, 2007 for surface water, sediment, and biota sampling (including fish and benthic macroinvertebrate surveys, and aquatic toxicity testing). This sampling event is expected to take 5 to 7 days to complete. All aqueous samples will be collected prior to any sediment or macroinvertebrate samples as the process may stir up contaminants that are not reflected during the normal flow regime. All aqueous samples will be collected synoptically Monday July 9 and July 10 using several sampling teams. Field parameters and stream flow will also be collected at this time. All sites will be revisited after all aqueous samples have been collected for completion of macroinvertebrate and sediment sampling using one to two teams. At the drafting of this SAP, EPA received notification and approval from the CDOW for a fish survey of several locations in the Peru Creek watershed. It is unclear at this time when this event will occur, but it is tentatively scheduled for the same timeframe, July 2007.

A second sampling event including sampling of mine waste, mine water features (draining adits and seeps), soil borings, and ground water is scheduled for the week of September 24, 2007. Certain locations from the July 2007 sampling event will be sampled again for comparability purposes as well as for contaminant and toxicity testing during low-flow conditions. An abbreviated amendment tables providing the details for the low-flow sampling event will be prepared and attached to this SAP.

6.1.2 Safety

The mine waste site investigation will be conducted by a CDPHE team, which will consist of a minimum of two persons. It is expected that most site-related activities can be accomplished in Level D personal protective equipment (PPE). The water quality-sampling event will be lead by an EPA field team and will consist of volunteers from several different agencies. EPA personnel will conduct a safety briefing before sampling begins.

All EPA and CDPHE personnel involved in this study have current health and safety training certifications and are participating in the EPA medical monitoring program. All personnel have been trained in field safety, first aid, CPR, and laboratory safety. In case of emergency, field personnel can seek help from the following entities:

Fire/Police/Sheriff/Ambulance: 911
Provenant Summit Medical Center: 970-668-3300
(24-hour emergency health care facility located on Hwy. 9 at School Road in Frisco)

6.1.3 Site Access and Logistics

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Access to the site and all sample locations will need to be acquired from the property owner if on private property. Because this is a joint effort between the US EPA, USFS, CDPHE and Summit County, properties owned by Summit County or the USFS have open access to collect samples.

6.2 SAMPLE LOCATIONS

This SAP calls for the collection of the following samples:

- 33 surface water quality sample locations (2 events - High and Low flow);
- 31 sediment quality sample locations (2 events - High and Low flow);
- 15 surface water samples for toxicity testing (2 events - High and Low flow);
- 19 macroinvertebrate samples (2 events - High and Low flow);
- 100 surficial (0 to 6 inches bgs) composite soil samples (1 event Aug/Sept 2007);
- 18 mine feature water sample (GW seeps or adit discharge) (1 event Aug/Sept 2007); and
- 22 ground water samples.

All mine waste soil samples will be tested for the following list of total metals: aluminum, arsenic, cadmium, chromium, copper, iron, lead, manganese, magnesium, mercury, nickel, selenium, silver, and zinc. Additionally, composite mine waste samples will be analyzed for Acid Base Accounting (ABA) and the USGS Modified Leach Test Method which uses a de-ionized water solution instead of a dilute nitric acid solution as in the SPLP. The modified USGS method more accurately represents the natural state of leaching by natural precipitation of historical mine waste. This method quantifies toxic inputs and the soluble constituents available to mobilization. All mine waste and drainage sample locations are shown in Figures 1, 2, and 3 and are described in Tables 1 and 2.

All surface water samples collected during the high flow event in early summer 2007will be analyzed for sulfate, chloride, alkalinity, dissolved organic carbon, and the following list of dissolved and total recoverable metals: aluminum, arsenic, cadmium, chromium, copper, iron, lead, manganese, magnesium, mercury, nickel, selenium, silver, and zinc. Various cations, including sodium, potassium, calcium, and magnesium, will also be analyzed from the dissolved metals fraction.

Ground water and soil core samples will be collected from the alluvial area adjacent to Peru Creek to delineate the extent of tailing in contact with alluvial water. Additionally, ground water and core samples will be collected from the upper Cinnamon Gulch area to better define and understand depth to bedrock and if ground water contamination flowing in the substrate is contaminated, if a reactive barrier type passive treatment system would be an applicable affordable alternative in treating groundwater in the upper reaches of Cinnamon Gulch.

Certain locations within the Peru Creek watershed where chemistry samples are collected will also be sampled for macroinvertebrates, sediment metals, and surface water toxicity to fish.

Whenever a water chemistry or toxicity sample is collected, field parameters, including temperature, conductivity, pH, dissolved oxygen, and flow will be taken using EPA protocols.

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Table 3 describes all of the locations to be sampled during the high flow event and the samples to be collected at each location. Surface water sample locations and corresponding toxicity test locations for low-flow sampling will be determined based on the results of the high-flow testing.

Table 3 and Figure 4 describe the surface water sampling locations and the types of samples to be collected at each location. Tables 4 through 8 describe all parameters to be analyzed from the water and sediment samples and the required container types, preservatives, detection limits, and analytical holding times.

6.3 SAMPLING PROCEDURES

6.3.1 Surface and Subsurface Soil Sampling

Surface soil composite samples will be collected in accordance with procedures described in CDPHE SOP 16, "Surface and Shallow Depth Soil Sampling." Samples will be placed in appropriate sample containers and documented in accordance with the procedures outlined in CDPHE SOP 5, "Sample Location Documentation" (CDPHE 2000). All soil sample locations will be documented with a Global Positioning System (GPS) for incorporation into the CDPHE GIS database.

Collection of composite soil samples from waste rock and tailings piles will involve the collection of 30 individual soil aliquots or increments from a single mine waste pile that has a uniform appearance. A uniform appearance reflects colors or significant changes to the appearance of the mine waste pile. The aliquots or increments are collected from the upper 4 inches of the mine waste pile using dedicated disposable sampling equipment. Coarse material (>4-mm) is discarded during collection. All increments are placed into a 2-gallon plastic bucket or zip-lock bag and mixed. The sample should be air dried and well homogenized. The complete 30-point soil composite will be sieved to pass a 2-mm stainless steel screen. The <2-mm fraction used for the USGS leaching procedure and metals analysis. The >2-mm fraction will be discarded.

Measures will be taken to minimize the amount of in-field equipment decontamination required for the sampling event. Sampling equipment will be decontaminated prior to the sample event. Equipment that will be reused will be decontaminated in the field in accordance with procedures described in CDPHE SOP 16, "Equipment Decontamination" (CDPHE 2000).

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6.3.2 Surface Water Quality Samples

Water from surface sampling locations will be collected synoptically over a four hour time period on Monday, July 9, 2007 from the Peru Creek study area. Surface water will also be sampled in September 2007 to evaluate low-flow conditions. A SAP addendum will be prepared and attached to this SAP prior to the September sampling event. Several teams will be needed to collect all samples during the limited four-hour time frame. Surface water flow measurements and field parameters will be taken at the same approximate time that water samples are collected. All water quality-sampling tasks will follow procedures outlined in the EPA Field Sampling Protocols SOP (EPA 2002).

Field measurements of physicochemical parameter, total and dissolved metal samples will be collected at specified locations as indicated in Table 3. Water quality sampling station locations for field parameters, chemical analyses, and toxicity are presented in Table 3. Field parameters, including flow, temperature, conductivity, pH, and dissolved oxygen will be collected in-situ at each sampling location. Methods and detection limits for field parameters are described in Table 4. All water quality metals samples will be collected into HDPE or LDPE containers and preserved with 0.5 ml nitric acid in the field as described in Tables 5 and 6.

Total and dissolved samples for metal analysis will be collected at each location. A minimum of 125 ml of sample will be collected for both dissolved and total metals analysis. Samples will be filtered in the field in accordance with EPA SOP 720 and CDPHE SOP 16, “Surface Water Sampling” (CDPHE 2000; EPA 2002). The filter size for these samples will be 0.45 micron. Total carbonate hardness will be estimated from the ICP measurements of dissolved calcium and magnesium. Holding times will follow EPA recommendations found in "Methods for Chemical Analysis of Water and Waste," 1983 as shown in Table 5. Sample containers and sample preservation for chemical samples will follow the procedures outlined in Table II of 40 CFR 136. Samples for dissolved organic carbon (DOC) will be collected in 250 ml HDPE containers and preserved to pH < 2 with 0.5 ml of phosphoric acid and chilled to 4 C using ice. Samples for alkalinity, sulfate, and chloride analysis will be collected in 250 ml HDPE containers and also chilled at 4 C for preservation as described in Table 7.

6.3.3 Sediment Samples

Sediment samples will be collected from the locations listed in Table 3. Sediment grab samples will be collected from the surface of the stream base. Sampling will begin after all water quality samples have been obtained. Pre-cleaned Teflon scoops will be used to collect the samples into 60 ml HDPE sample containers. Sediment samples will be cooled using ice to 4 C as described in Table 9.

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6.3.4 Toxicity Testing

Surface water to be used in the aquatic toxicity tests, as described in Table 8, will also be collected synoptically over a four-hour time period into pre-cleaned plastic containers and chilled during transport to the laboratory. Water from the site will be transported to the Region 8 Laboratory and toxicity tests will commence the morning following sample collection in the EPA Region 8 Laboratory. For the July 9, 2007 toxicity test event, up to 11 locations will be tested using aquatic static-renewal toxicity tests methods in accordance with EPA-821-R-02-012. *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms*. Fifth Edition, October 2002. The total number of tests will depend on laboratory capacity. A minimum of nine tests are likely to be conducted and a total of 15 tests are expected. Most testing will be conducted using rainbow trout (30-day post egg-sac absorption), however, several of the test locations may include testing of brown trout (depending on availability of test species). As indicated in Table 3, toxicity testing will either include profile testing (i.e., exposure using 100% site water), or serial dilutions (using .75 or .50 dilution series), to determine LC50s. Ammonia will be measured during all toxicity tests to be conducted at the Region 8 Laboratory. A second set of toxicity testing will be conducted during representative low flow conditions scheduled for the week of September 24, 2007.

6.3.5 Macroinvertebrate Samples

Macroinvertebrates will be collected after all water quality-sampling procedures have been completed. Protocols outlined in the EPA document "Standard Operating Procedure for Field Sampling of Aquatic Macroinvertebrates" (EPA 2002), will be followed. In general, macroinvertebrates will be sampled using a one-square foot Surber sampler. A qualitative habitat description will be performed and documented at each sample location. Three surber sample locations will be collected from riffle habitats or other representative habitats at each sampling location and will be consolidated into a 500 ml or 1-liter plastic container as described in Table 8. Samples will be preserved in the field using 70 percent ethyl alcohol and will be relinquished to the US Forest Service for identification to the lowest possible taxonomic level.

Sample locations with a high density of macroinvertebrates indicate that these locations may be used as a food resource for fish and other wildlife. Therefore, a split sample will be obtained and submitted for metals laboratory analysis in addition to taxonomic identification at these locations.

6.3.6 Groundwater Samples

Ground water sampling will be conducted according to those procedures outlined in CDPHE SOP 12, "Ground Water Sampling" (CDPHE 2000). All ground water sampling locations will be documented in accordance with the procedures outlined in CDPHE SOP 5, "Sample Location Documentation" (CDPHE 2000; EPA 2002). Additionally, all well locations will be surveyed for elevations to aid in the completion of a ground water contour map.

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All ground water wells will be installed using a hollow stem auger drill rig, which will allow for the collection and observation of discreet soil samples from cores collected from hole. This will aid in the determination of the presence of tailings or waste rock and the extent of it. Soil cores and Split spoon sampling is further discussed in Section 6.3.7, "Soil Core Samples."

Following are required procedures

- Prior to well installation, a survey to locate all underground utilities at the site must be completed.
- The outer diameter of the hollow stem auger shall be at least six (6) inches in diameter if a 4-inch casing is to be installed, and at least four (4) inches in diameter if a 2- inch casing is to be installed. A minimum 2-inch filter pack annulus is required for both cases.
- The monitoring well(s) shall be two (2) inch or four (4) inch in diameter with threaded joints. The monitoring well casing shall be constructed of stainless steel, P.V.C. schedule 40 minimum) or approved equal. The installation of 1-inch diameter, pre-packed monitoring wells (micro wells) will be considered on a case-by-case basis.
- Shallow monitoring wells shall have a well screen that is two (2) inch or four (4) inch diameter with a preferred slot size of 0.010-inch. If a slot size of 0.020-inch or higher is installed, a coarser sand pack must be used. The American Society for Testing and Materials (ASTM) D 5092-90 recommends that the slot size should retain at least 90% (and preferably 99%) of the sand pack. The well screen should be set at least two (2) feet above the normal annual high water table and at least three (3) feet below the annual low water level.
- The monitoring well casing shall be installed true vertical in the drilled bore hole.
- A clean washed sand pack shall occupy the annular space from the bottom of the borehole to one (1) foot above the well screen. The ASTM D 5092-90 recommends the use of sand with a grain size that is about four to ten times greater than the d-30 (30% finer) grain size of the hydrologic unit being filtered. Use of a filter sock in lieu of a sand pack is not acceptable.
- The use of fine clean washed sand, instead of clay (hydrated bentonite), which tends to bridge the annulus, is preferred to create a seal (6-inch thick) above the annular space pack material to avoid grout migration into the pack. The remaining annular space between the casing and the borehole shall be grouted with neat cement or with a cement/bentonite mix (up to 6% bentonite by weight).
- Permanent monitoring wells shall be constructed in such a way that they are not prone to surface water infiltration or located in low areas that experience repeated flooding.
- All wellheads of the permanent monitoring wells should be constructed to be in a best-protected manner, with a two-foot steel well stickup and set in a concrete pad.
- The top of the permanent monitoring wells shall be sealed with a watertight locking cap. The well shall be kept locked at all times to prevent tampering.

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- All permanent-monitoring wells shall be adequately labeled to distinguish them as monitoring wells and shall be properly secured to prevent vandalism and/or the accidental introduction of contaminants.

6.3.7 Soil Core Samples and Procedures

All ground water wells and soil cores will be installed using a hollow stem auger drill rig, which will allow for the collection and observation of discreet soil samples from cores collected from a split spoon from the drilled hole. This will aid in the determination of the presence of tailings or waste rock and the extent of it. Split spoon sampling is generally used to collect undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be extracted with a split spoon sampler to give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom of the augured hole and the core extracted.

The following procedures are used for collecting soil samples with a split spoon:

- Assemble the sampler by aligning both sides of barrel and then screwing the drive shoe on the bottom and the headpiece on top.
- Place the sampler in a perpendicular position on the sample material.
- Using the appropriate auger-drilling rig, drive the tube to the desired depth. Do not drive past the bottom of the headpiece or compression of the sample will result.
- Record in the site logbook or on field data sheets the length of the tube used to penetrate the material being sampled, and the number of blows required obtaining this depth.
- Withdraw the sampler, and open by unscrewing the bit and head and splitting the barrel. The amount of recovery and soil type should be recorded on the boring log. If a split sample is desired, a cleaned, stainless steel knife should be used to divide the tube contents in half, longitudinally. This sampler is typically available in 2 and 3 1/2 inch diameters. A larger barrel may be necessary to obtain the required sample volume.
- With minimal disturbance to the core, transfer it to appropriate labeled sample container(s) and seal tightly.

7.0 CHAIN OF CUSTODY

Volunteers from various State and Federal governmental agencies will conduct field measurements and collect water, sediment, and macroinvertebrates for laboratory analyses of those analytes identified in Tables 4 thru 9 of this sampling plan. All parties involved in this sampling effort will be responsible for the collection and preservation of all samples and their appropriate chain-of-custody requirements. Chain-of-custody procedures will follow EPA Region VIII Field Protocols SOP for sample collection (EPA 2002). Bill Schroeder will be the sample custodian for the aqueous sampling event in July 2007 and will keep records of all samples collected for analysis at the designated laboratory. Mark Rudolph will be the sample custodian for the mine waste and seep ground water sampling event in August /September 2007 and will keep records of all samples collected for analysis at the designated laboratory A chain-of-custody record will accompany all samples and will be checked by the appropriate sample

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custodian. All samples will be tagged with pre-numbered and recorded sample tags.

All mine waste samples will be handled in strict accordance with CDPHE SOP 3, "Chain of Custody." Documentation of sample storage and shipment will be included as part of the chain-of-custody procedures.

8.0 QUALITY ASSURANCE / QUALITY CONTROL (QA/QC)

The CDPHE Quality Assurance Project Plan (QAPP), approved by the EPA in January of 2000, will serve as a guide in all sample efforts. Sample bottles will meet EPA specifications, and will be part of the quality control program.

PARCC PARAMETERS

- Precision - the degree of agreement among repeated measurements of the same characteristic, or parameter, and gives information about the consistency (reproducibility) of the method.
- Accuracy - a measure of confidence that describes how close a measurement is to its 'true' value.
- Representativeness -the extent to which measurements actually represent the 'true' environmental conditions.
- Completeness - the comparison between the amount of data you planned to collect and analyze versus how much usable data was collected and analyzed. Normally expressed as a percentage.
- Comparability - the degree to which data can be compared directly to similar studies.

Field Parameters: Precision and accuracy for chemical measurements such as pH, temperature, conductivity and dissolved oxygen will be determined according to the EPA Chemical Methods Manual, EPA Region VIII's Standard Operating Procedures (SOP) for Field Samplers, or the manufacturer's specifications. Stream flow measurements will occur during the same general time period that the surface water samples are collected only if conditions allow safe access. Flow measurement methods will follow the Regional SOP for field sampling.

All field meter calibration procedures will be conducted according to USEPA requirements and will follow the EPA Laboratory's Hydrolab SOP (EPA 2006). The pH, dissolved oxygen, and conductivity electrodes will be calibrated each morning before use. Whenever readings appear unusual, performance will be checked using the calibration solutions stored with the meter. Field personnel will recalibrate the meter as necessary as described in the Hydrolab SOP. Field meters will be charged before use and supplies including probe-filling solutions, membranes, and calibration buffers solutions will be changed before the start of the field event and when a problem is encountered. Field meters or probes will be replaced if they do not meet EPA requirements.

Abiotic Media: Duplicate samples will be collected from surface water and ground water and sent to the laboratory for all chemical analyses. Field blanks, consisting of ultra-pure laboratory DI water, will also be processed in the field and submitted to the laboratory to check on the sample container, filtration apparatus and acids used in preservation. One set of duplicate and blank samples will be collected for every 20 locations sampled in the field. Additional sample volumes will be provided to the laboratory

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for internal QA/QC purposes for preparation of matrix-spike and matrix-spike-duplicate QC samples. All samples will be analyzed by either a privately contracted laboratory or through the Contract Laboratory Program (CLP) of the EPA. Post analysis review of the sample results, calibrations and Quality Control (QC) analysis or a Quality Assurance (QA) review of all analyses, samples and data will be conducted to ensure that data produced are of known quality and can be used as intended.

Toxicity Testing: Toxicity data will be analyzed according to the procedures outlined in the EPA Acute Toxicity manual (*Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms*, October 2002). Data acceptability for toxicity tests will be determined from laboratory control tests and a reference toxicant test. One duplicate sample will be collected and run concurrently during toxicity testing to document precision.

Control samples will be run for each toxicity test to check on the health of test organisms and the quality of reconstituted water used for sample dilution. A greater than 10% control mortality of test organisms will result in the test's failure. The regional laboratory will also perform a reference toxicant test using a salt solution as a QC check. Reference toxicant results will be compared with those from previous toxicity testing events to see if problems exist with laboratory cultures or dilution water.

Region VIII standard report forms for toxicological analyses will be used during the tests. All data and significant observations from tests will be kept on the test data sheet or in a bound notebook. Any deviations from the required test procedures will be documented. Test results will be kept on file by the EPA Office of Technical and Management Services Laboratory for future reference.

Biotic Sampling: Methods for collection and identification of macroinvertebrate data will follow protocols outlined in the EPA Region VIII Macroinvertebrate SOP (EPA 2002) and sampling and taxonomic identification will be conducted by or supervised by trained individuals. Fish surveys will be conducted by or supervised by trained individuals using Colorado Department of Wildlife protocols.

Laboratory QA/QC: Matrix Spike/Matrix Spike Duplicates (MS/MSD) for each matrix sampled on a one MS/MSD per 20 samples collected. These MS/MSD samples will be utilized for internal laboratory Quality Assurance/Quality Control (QA/QC) purposes. All samples will be analyzed by either a privately contracted laboratory or through the Contract Laboratory Program (CLP) of the EPA. Post analysis review of the sample results, calibrations and Quality Control (QC) analysis or a Quality Assurance (QA) review of all analyses, samples and data will be conducted to ensure that data produced are of known quality and can be used as intended.

9.0 CONTROL OF CONTAMINATED MATERIALS

The sampling team will dispose of all wastes produced during the investigation in accordance with CDPHE SOP 8, "Investigation Derived Waste" and methods outlined in EPA document 540-G-91-009 entitled "Management of Investigation-Derived Wastes During Site Inspections." Disposable sampling equipment, rubber gloves, and protective outerwear will be decontaminated, bagged, removed from the site and disposed of as a non-hazardous solid waste.

10.0 DATA REDUCTION, VALIDATION, AND REPORTING

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Records will be kept of actual sample locations and sample points will be accurately located on appropriate topographic maps. Procedures will provide documentation of changes in sample locations as they occur in the field due to unanticipated site conditions. Sample locations and sample collection procedures will be documented through the keeping of a field notebook and photographs. The results of the analyses conducted by the laboratory, including raw data sheets, QA/QC report, and a summary of the data, will be forwarded to Bill Schroeder, EPA Office of Technical and Management Services (TMS) Laboratory. The laboratory will provide the data in electronic format to Mr. Schroeder in the form of an Excel spreadsheet. If any laboratory QA/QC does not meet EPA acceptance criteria, Bill Schroeder will be immediately notified for further instructions. The results of the water chemistry and flow data will be evaluated and summarized by EPA personnel. Data validation for chemical analyses conducted by Region VIII will follow USEPA procedures. Within eight weeks of receipt of the QC reviewed or validated analytical data, an analytical report will be prepared for submission to the CDPHE Brownfield's/Voluntary Cleanup Coordinator and the EPA Brownfield's Coordinator.

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11.0 REFERENCES

American Geological Services, Inc. (AGS), 2003a, Summit County Brownfields Study (Phase I) – *report pending (Mark Arnold/Brian Lorch; only data obtained to date).*

American Geological Services, Inc. (AGS), 2003b, Summit County Brownfields Study (Phase II) – *report pending (Mark Arnold/Brian Lorch; only data obtained to date).*

Bencala, K.E. and McKnight, D.M., 1987, Identifying In-Stream Variability – Sampling Iron in an Acidic Stream: *Chemical Quality of Water and the Hydrologic Cycle*, R.C. Averett and D.M. McKnight (Eds.).

Bencala, K.E., McKnight, D.M., and Zellweger, G.W., 1987, Evaluation of Natural Tracers in an Acidic and Metal-Rich Stream: *Water Resources Research,* Vol. 23, No. 5, May, pp. 827-836.

Bencala, K.E., McKnight, D.M., and Zellweger, G.W., 1990, Characterization of Transport in an Acidic and Metal-Rich Mountain Stream Based on a Lighium Tracer Injection and Simulations of Transient Storage: *Water Resources Research,* Vol. 26, No. 5, May, pp. 989-1000.

Bencala, K.E., Kimball, B.A., and McKnight, D.M., 1991, Use of Variation in Solute Concentration to Identify Interactions of the Substream Zone with Instream Transport: *in* Proceedings, U.S. Geological Survey Toxics Substances Hydrology Program, U.S. Geological Survey Water-Resources Investigations Report 91-4034.

Bird, D.A, 2003, Characterization of anthropogenic and natural sources of acid rock drainage at the Cinnamon Gulch abandoned mine land inventory site, Summit County, Colorado: Environmental Geology, 44:919-932.

Colorado Department of Public Health and the Environment (CDPHE). 2000. Quality Assurance Project Plan and Standard Operating Procedures. January 2000.

Colorado Department of Public Health and Environment (CDPHE), 2000b, Status of Water Quality in Colorado – 2000: Water Quality Control Division, Prepared in Fulfillment of Section 305(b) of the Clean Water Act of 1977 (P.L. 95-217), December, pp. II-32 and III/3-59.

Colorado Division of Water Resources. 2007. Colorado Wells, Applications, and Permits Database.

Duren, Sabre M. 2004. Quantitative Hydrologic Evaluation of Dissolved Metal Loading and Transport in Peru Creek, an Acid Rock Drainage Stream; Summit County, Colorado. M.S. Thesis, University of Colorado at Boulder.

Fey, D.L., Church S.E., Unruh, D.M., and Bove, D.J., 2002, Water and Sediment Study of the Snake River Watershed, Colorado – October 9-12, 2001: U.S. Geological Survey Open-File Report 02-0330, 39 p. and Appendix (7 tables).

Fey, D.L., Church, S.E., Unruh, D.M., and Bove, D.J., 2002, Water and sediment study of the Snake River watershed, Colorado, October 9-12, 2001: USGS Open-File report 02-0330, 41p.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Holm, J.D., Sullivan, T., and Stenulson, B.K., 1978, The restoration of Peru Creek – Analyzing the feasibility of abating toxicity associated with abandoned mine drainage: Colorado 208 Water Quality Unit, CWQCD, USFS report.

Holm, J.D., Sullivan, Timothy, and Stenulson, B.K., 1979, The Restoration of Peru Creek – Analyzing the Feasibility of Abating Toxicity Associated with Abandoned Mine Drainage: Colorado 208 Water Quality Unit in Cooperation with the Colorado Water Quality Control Division and the United States Forest Service, October, 65 p. *[KC, red notebook; DMG files]*.

McKnight, D.M. and Bencala, K.E., 1988, Diel Variations in Iron Chemistry in an Acidic Stream in the Colorado Rocky Mountains, U.S.A.: *Arctic and Alpine Research*, Vol. 20, No. 4, pp. 492-500.

McKnight, D.M. and Bencala, K.E., 1989, Reactive Iron Transport in an Acidic Mountain Stream in Summit County, Colorado – A Hydrologic Perspective: *Geochemica et Cosmochemica Acta*, Vol. 53, pp. 2225-2234.

McKnight, D.M. and Bencala, K.E., 1990, The Chemistry of Iron, Aluminum, and Dissolved Organic Material in Three Acidic, Metal-Enriched, Mountain Streams, as Controlled by Watershed and In-Stream Processes: *Water Resources Research*, Vol. 26, No. 12, December, pp. 3087-3100.

McKnight, D.M., Bencala, K.E., Zellweger, G.W., Aklen, G.R., Feder, G.L., and Thorn, K.A., 1992a, Sorption of Dissolved Carbon by Hydrous Aluminum and Iron Oxides Occurring at the Confluence of Deer Creek with the Snake River, Summit County, Colorado: *Environmental Science & Technology*, American Chemical Society (ACS), Vol. 26, No. 7, July, pp. 1388-1396.

McKnight, D.M. and Feder, G.L., 1984, The Ecological Effect of Acid Conditions and Precipitation of Hydrous Metal Oxides in a Rocky Mountain Stream: *Hydrobiologia*, Vol. 119, No. 2, Dr. W. Junk Publishers, Dordrecht, The Netherlands, pp. 129-138.

McKnight, D.M., Wershaw, R.L., Bencala, K.E., Zellweger, G.W., and Feder, G.L., 1992b, Humic Substances and Trace Metals Associated with Fe and Al Oxides Deposited in an Acidic Mountain Stream: *The Science of the Total Environment*, Elsevier Science Publishers B.V., Amsterdam, The Netherlands, pp. 485-498.

McKnight, D.M., and Bencala, K.E., 1990, The chemistry of iron, aluminum, and dissolved organic material in three acidic, metal-enriched, mountain streams, as controlled by watershed and in-stream process: Water Resources Research, 26:3087-3100.

Munroe, Erik A. 2000. Abandoned Mine Lands in Peru Creek, Deer Creek, upper Snake River, and Swan River Watersheds: Sources of metal contamination to the environment and their amelioration through passive treatment options. White River National Forest Dillon Ranger District.

Northwest Colorado Council of Governments (NWCCOG), 2002, Snake River Sample Analysis and Assessment Plan: Prepared by Lane Wyatt, Acting Director of Watershed Services, Section 319 Grant Application, Project Summary Sheet, 12 p.

Sullivan, A.B., and Drever, J.J., 2001, Spatiotemporal variability in stream chemistry in a high-elevation catchment affected by mine drainage: Journal of Hydrology, 252: 237-250.

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Theobald, P.K., Kalin, H.W., and Hawkins, D.B., 1963, The Precipitation of Aluminum, Iron, and Manganese at the Junction of Deer Creek with the Snake River in Summit County, Colorado: *Geochimica et Cosmochimica Acta*, Vol. 27, pp. 121-132. *[referenced in Tokash (2003)]*

Todd, Andrew S., McKnight, D.M., and Duren, Sabre M., 2004 (draft), Water Quality Characteristics for the Snake River, North Fork of the Snake River, Peru Creek, and Deer Creek in Summit County, 2001-2002: University of Colorado, Institute of Arctic and Alpine Research, Occasional Paper, 16 p., 9 figures, 3 tables, and Appendix A (23 tables). *[draft for review; see TDS 3/18/04 comments]*

Todd, Andrew, McKnight, Diane, and Wyatt, Lane, 2003, Abandoned Mines, Mountain Sports, and Climate Variability – Implications for the Colorado Tourism Economy: *EOS, Transactions, American Geophysical Union (AGU)*, Vol. 84, No. 38, pp. 377 and 386

Todd, A.S., McKnight, D.M., and Duren, S.M., 2005, Water quality characteristics for the Snake River, Peru Creek, and Deer Creek in Summit County, Colorado: 2001 to 2002: INSTAAR Occasional Paper No. 57, 46p.

Trout Unlimited. 2007. Summary Overview of Historic Actions Taken at the Pennsylvania Mine Site Adit Discharge. 2007.

U.S. Environmental Protection Agency (EPA). 1999. EPA Contract Laboratory Program. Statement of Work for Inorganic Analysis. February 1999.

U.S. Environmental Protection Agency (EPA). 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fifth Edition, October 2002

U.S. Forest Service (USFS). 1994. USFS-Abandoned Mined Land Inventory Project, Dillon Ranger District, Final Summary Report. Randall K. Streufert, Colorado Geological Survey.

USGS 2007. Watershed Characterization Report. http://pubs.usgs.gov/of/1997/ofr-97-0151/html/stpmw02.shtml

U.S. Geological Survey (USGS). 1974. 7.5 Minute Topographic Quadrangle, Montezuma, Colorado.

U.S. Geological Survey (USGS). 1987a. 7.5 Minute Topographic Quadrangle, Grays Peak, Colorado.

U.S. Geological Survey (USGS). 1987b. 7.5 Minute Topographic Quadrangle, Keystone, Colorado.

URS Operating Services, Inc., 2001, Site Assessment, Field Sampling Plan, Snake River Technical Support, Summit County, Colorado: Prepared for U.S. Environmental Protection Agency (USEPA), Superfund Technical Assessment and Response Team 2 (START2), Region VIII, Contract No. 68-W-00-118, TDD No. 0108-0005, September 11, 30 p.

URS Operating Services, Inc., 2002, Site Assessment, Analytical Results Report, Phase II – May 2002, Snake River Technical Support, Summit County, Colorado: Prepared for U.S. Environmental Protection Agency

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(USEPA), Superfund Technical Assessment and Response Team 2 (START2), Region VIII, Contract No. 68-W-00-118, TDD No. 0108-0005, November 4, 9 sections (22 p.), and Appendices A through D.

Wong, J.W., 2006, Quantifying the effect of drought on transport processes controlling metal contamination in Peru Creek, an acid mine drainage stream: MS Thesis, University of Colorado, Boulder, 103p.

Wood, R.H., Bird, D.A, and Sares, M.A., 2005, Mine site history and watershed characterization of the Cinnamon Gulch area, Dillon Ranger District, White River National Forest, Summit County, Colorado: CGS Open-File Report 05-1, 149p.

Wyatt, Lane, TDS Consulting Inc., and Walsh Ecological Consultants, Inc., 2002, Blue River Use-Attainability Analysis: Revised Draft Final Report, Prepared on Behalf of the Summit Water Quality Committee, U.S. Environmental Protection Agency, and Colorado Department of Public Health & Environment, April 12, 79 p., references (18 p.), and Appendices A, B, and D.

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Table 1: **Descriptions of AGS Survey Locations to be Sampled in this SI**

AGS #	Mine Grouping	Mine Name	Claim Name	Features Present	Area (Ft2)	Mineralization	Oxidation	Wetlands	Erosion	Discharge
15	Silver Spoon	Silver Spoon	Lucky	Prospect pits, dump	2000	Pyrite	Heavily Oxidized	Present	Minor Rills	None
16	Silver Spoon	Silver Spoon	Lucky	Collapsed adit and dumps	1000	Pyrite, Quartz, Sericite	Heavily Oxidized	Present	Minor Rills	None
17	Silver Spoon	Silver Spoon	Black Crow	Adit and dumps	1000	Pyrite	Heavily Oxidized	Not Present	Minor Rills	0 - 5 gpm
18	Silver Spoon	Silver Spoon	Delaware Extension	Collapsed adit	1000	Pyrite	Heavily Oxidized	Present	Minor Rills	0 - 5 gpm
19	Silver Spoon	Silver Spoon	Lucky Dutchman	Exploration adit	1000	Pyrite, Quartz, Sericite	Heavily Oxidized	Not Present	None	0 - 5 gpm
20	Silver Spoon	Silver Spoon	Lucky Dutchman	Exploration adit	1000	Pyrite, Sphalerite	Heavily Oxidized	Not Present	None	0 - 5 gpm
22	Silver Spoon	Silver Spoon	Delaware Extension	Open adit, dump	1000	Pyrite, Quartz, Sericite	Heavily Oxidized	Present	Minor Rills	0 - 5 gpm
23	Silver Spoon	Silver Spoon	Black Crow	Open adit and dump	1000	Pyrite	Heavily Oxidized	Present	Minor Rills	0 - 5 gpm
33	Penn Mine	Pennsylvania	Tram Mill Site	Mill/tailings	2000	Pyrite	Heavily Oxidized	Not Present	Minor Rills	None
35	Penn Mine	Pennsylvania	Leadville	Collapsed adit/small dump	500	Pyrite, Sericite	Heavily Oxidized	Not Present	None	None
44	Silver Spoon	--	USFS - claim unknown	Collapsed adit, dump	3000	Pyrite, Galena, Quartz	Heavily Oxidized	Present	Minor Rills	0 - 5 gpm
45	Silver Spoon	--	USFS - claim unknown	4 collapsed shafts, large dumps	10000	Pyrite, Galena	Heavily Oxidized	Not Present	Minor Rills	None
51	Delaware	--	Annex	Collapsed adit, dump, discharge pond	15000	Pyrite, Galena	Heavily Oxidized	Present	None	> 15 gpm
63	Decatur Townsite	--	Decatur Townsite	Dump, collapsed prospect pits	500	Pyrite, Quartz, Sericite	Heavily Oxidized	Present	None	None
107	Penn Mine	Pennsylvania	El Jebel	Adit discharge point from treatment shed	2500	Pyrite	Heavily Oxidized	Present	None	6 - 15 gpm
108	Penn Mine	Pennsylvania	El Jebel	Collapsed crusher/stamp mill?	4000	Pyrite	Heavily Oxidized	Present	Minor Rills	None
109	Penn Mine	Pennsylvania	USFS - claim unknown	Closed Adit	100	Pyrite	Heavily Oxidized	Present	Minor Rills	None
110	Penn Mine	Pennsylvania	El Jebel	Dump	1800	Pyrite	Heavily Oxidized	Present	Minor Rills	None
111	Decatur Townsite	--	Forest City	Mill, crusher-stamp mil, tails	3000	Pyrite, Galena, Chalcopyrite	Heavily Oxidized	Not Present	None	None
113	Decatur Townsite	--	Forest City	Dump	1000	Pyrite, Chalcopyrite, Galena	Heavily Oxidized	Not Present	None	None
114	Penn Mine	--	Badger	Adit, dam, vent raiser pipe	500	Pyrite, Chalcopyrite, Galena, Sphalerite	Heavily Oxidized	Not Present	None	6 - 15 gpm
115	Penn Mine	--	Giant Mill Site	Dump above mill	500	Pyrite, Chalcopyrite, Galena, Sphalerite	Heavily Oxidized	Not Present	None	None
116	Penn Mine	--	Badger	Toe of tailings pond	2000	Pyrite, Chalcopyrite, Galena, Sphalerite	Heavily Oxidized	Not Present	None	None
117	Penn Mine	--	Giant Mill Site	Dump	5000	Pyrite, Chalcopyrite, Galena, Sphalerite	Heavily Oxidized	Not Present	None	None
118	Penn Mine	--	Giant Mill Site	Dump	10000	Pyrite, Chalcopyrite, Galena, Sphalerite	Heavily Oxidized	Not Present	None	None
119	Penn Mine	--	Evergreen Tunnel Lode	Collapsed adit	1000	Pyrite, Chalcopyrite	Unutilized	Not Present	None	None
120	Penn Mine	--	Evergreen Tunnel Lode	Shaft and dump	2000	Pyrite, Chalcopyrite	Unutilized	Not Present	None	None
128	Brittle Silver	--	White Sparrow	Open/Partly Collapsed Adit, dump	1200	Quartz, Sericite, Pyrite	Heavily Oxidized	Not Present	Minor Rills	None
130	Brittle Silver	--	USFS - claim unknown	Dog Hole, dump	200	Quartz, Pyrite, Sericite	Heavily Oxidized	Not Present	Minor Rills	None
131	Brittle Silver	--	USFS - claim unknown	Dog Hole, dump	300	Quartz, Pyrite, Sericite	Heavily Oxidized	Not Present	Minor Rills	None
132	Brittle Silver	--	Brittle Silver	Dog Hole, dump	700	Quartz, Pyrite, Sericite	Heavily Oxidized	Not Present	Minor Rills	None
133	Brittle Silver	--	Brittle Silver	Dog Hole, dump	225	Quartz, Pyrite, Sericite	Heavily Oxidized	Present	None	None
134	Brittle Silver	--	Brittle Silver	Collapsed Adit (Shaft?), dump	1200	Quartz, Sericite, Pyrite, Galena	Weakly Oxidized	Not Present	Minor Rills	None
135	Brittle Silver	--	Peavy	Collapsed Shaft (?), dump	900	Quartz, Sericite, Pyrite	Heavily Oxidized	Not Present	Minor Rills	None

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Table 1: **Descriptions of AGS Survey Locations to be Sampled in this SI**

136	Brittle Silver	--	Peavy	Dog Hole, dump	450	Quartz, Sericite, Pyrite	Heavily Oxidized	Not Present	Minor Rills	None
137	Brittle Silver	--	Brittle Silver	Collapsed Adit, dump	2400	Quartz, Pyrite, Sericite, Galena, Sphalerite	Heavily Oxidized	Not Present	Minor Rills	None
138	Brittle Silver	--	Peavy	Trench, dump	1125	Quartz, Sericite, Pyrite	Heavily Oxidized	Not Present	Minor Rills	None
148	Silver Spoon	Silver Spoon	USFS - claim unknown	Collapsed Adit, Teetering Collar, dump	4200	Quartz, Sericite, Pyrite, Galena	Heavily Oxidized	Not Present	Severe Rills and Gullies	None
149	Silver Spoon	Silver Spoon	USFS - claim unknown	Open Adit, dump	3300	Quartz, Sericite, Pyrite, Galena, Sphalerite, Clay	Heavily Oxidized	Not Present	Severe Rills and Gullies	0 - 5 gpm
151	Silver Spoon	Silver Spoon	USFS - claim unknown	Old Trench, dump	270	Quartz, Sericite, Pyrite	Weakly Oxidized	Not Present	None	0 - 5 gpm
152	Silver Spoon	Silver Spoon	Black Crow	Open Adit, dump	1390	Quartz, Sericite, Pyrite, Galena	Heavily Oxidized	Not Present	Minor Rills	None
174	Brittle Silver	--	USFS - claim unknown	Collapsed adit with small dump	600	Quartz, Sericite, Pyrite	Weakly Oxidized	Not Present	Minor Rills	None
178	Brittle Silver	--	Little Nell	Collapsed shaft, dump, and two pits	5300	Quartz, Sericite, Pyrite, Chalcopyrite	Heavily Oxidized	Not Present	Minor Rills	None
213	Silver Spoon	--	USFS - claim unknown	Adit, dump, rail	5950	Quartz, Sericite	Heavily Oxidized	Not Present	Severe Rills and Gullies	None

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Table 2: Mine Waste and Feature Drainage Samples							
Sample ID	Mine Grouping	Comp or Grab	AGS #	Soil #	Water #	Flow Measurement	Sample Event
SS-15	Silver Spoon	Comp	15	3	1	No	Aug/Sept-2007
SS-16	Silver Spoon	Comp	16	2	1	No	Aug/Sept-2007
SS-17	Silver Spoon	Comp	17	2	1	Yes	Aug/Sept-2007
SS-18	Silver Spoon	Comp	18	2	1	Yes	Aug/Sept-2007
SS-19	Silver Spoon	Comp	19	2	1	Yes	Aug/Sept-2007
SS-20	Silver Spoon	Comp	20	2	1	Yes	Aug/Sept-2007
SS-22	Silver Spoon	Comp	22	2	1	Yes	Aug/Sept-2007
SS-23	Silver Spoon	Comp	23	2	1	Yes	Aug/Sept-2007
PM-33	Penn Mine	Comp	33	2	0	No	Aug/Sept-2007
PM-35	Penn Mine	Comp	35	2	0	No	Aug/Sept-2007
SS-44	Silver Spoon	Comp	44	3	1	Yes	Aug/Sept-2007
SS-45	Silver Spoon	Comp	45	4	0	No	Aug/Sept-2007
DT-51	Delaware	Comp	51	4	1	Yes	Aug/Sept-2007
DT-63	Decatur Townsite	Comp	63	2	1	No	Aug/Sept-2007
PM-107	Penn Mine	Comp	107	2	1	Yes	Aug/Sept-2007
PM--108	Penn Mine	Comp	108	2	1	No	Aug/Sept-2007
PM-109	Penn Mine	Comp	109	2	0	No	Aug/Sept-2007
PM-110	Penn Mine	Comp	110	2	0	No	Aug/Sept-2007
BS-111	Brittle Silver	Comp	111	2	0	No	Aug/Sept-2007
BS-113	Brittle Silver	Comp	113	2	0	No	Aug/Sept-2007
PM-114	Penn Mine	Comp	114	3	1	Yes	Aug/Sept-2007
PM-115	Penn Mine	Comp	115	2	0	No	Aug/Sept-2007
PM-116	Penn Mine	Comp	116	2	1	Yes	Aug/Sept-2007
PM-117	Penn Mine	Comp	117	3	0	No	Aug/Sept-2007
PM-118	Penn Mine	Comp	118	4	0	No	Aug/Sept-2007
PM-119	Penn Mine	Comp	119	2	0	No	Aug/Sept-2007
PM-120	Penn Mine	Comp	120	2	0	No	Aug/Sept-

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Table 2: Mine Waste and Feature Drainage Samples							
Sample ID	Mine Grouping	Comp or Grab	AGS #	Soil #	Water #	Flow Measurement	Sample Event
							2007
SS-148	Silver Spoon	Comp	148	2	0	No	Aug/Sept-2007
SS-149	Silver Spoon	Comp	149	2	1	Yes	Aug/Sept-2007
SS-151	Silver Spoon	Comp	151	2	1	Yes	Aug/Sept-2007
SS-152	Silver Spoon	Comp	152	2	1	Yes	Aug/Sept-2007
SS-211	Silver Spoon	Comp	211	2	0	No	Aug/Sept-2007
SS-213	Silver Spoon	Comp	213	2	0	No	Aug/Sept-2007
BS-178	Brittle Silver	Comp 1	174	2	0	No	Aug/Sept-2007
BS-130	Brittle Silver	Comp 1	130	2	0	No	Aug/Sept-2007
BS-131	Brittle Silver	Comp 1	131	2	0	No	Aug/Sept-2007
BS-132	Brittle Silver	Comp 1	132	2	0	No	Aug/Sept-2007
BS-133	Brittle Silver	Comp 1	133	2	0	No	Aug/Sept-2007
BS-134	Brittle Silver	Comp 1	134	2	0	No	Aug/Sept-2007
BS-128	Brittle Silver	Comp 2	128	2	0	No	Aug/Sept-2007
BS-174	Brittle Silver	Comp 2	178	2	0	No	Aug/Sept-2007
BS-135	Brittle Silver	Comp 3	135	2	0	No	Aug/Sept-2007
BS-136	Brittle Silver	Comp 3	136	2	0	No	Aug/Sept-2007
BS-137	Brittle Silver	Comp 3	137	2	0	No	Aug/Sept-2007
BS-138	Brittle Silver	Comp 3	138	2	0	No	Aug/Sept-2007
SW Duplicate	QA/QC	QA/QC	--	--	1	No	Aug/Sept-2007
GW Duplicate	QA/QC	QA/QC	--	--	2	No	Aug/Sept-2007
Total Number of Samples				100	21		

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Table 3: Peru Creek sampling locations and samples to be collected at each location.

Sampling Location	Latitude	Longitude	Elevation (HAE)	Description:	Field Parameters		Water Quality	Biology			Sediment Quality
					GPS	Temp, Cond, pH, DO, and Flow	Total and Dissolved Metals, Alk + SO4 + Cl, DOC)	Tox Testing	Macro-invertebrates	Fish	Sediment Metals
Snake River Stations (downstream to upstream):											
SW-082	39 36 19.30 N	105 56 36.06 W	9281	Snake River downstream of the North Fork of Snake River at USGS gage. From large parking lot, drive toward the resort and turn left at the River Lodge(?) sign. Pass the first set of condos, turn right and drive across a large parking lot with telephone poles.	No	Yes	Yes	Yes - 100%	Yes	Yes	Yes
SW-117	39 36 19.00 N	105 56 30.08 W	9288	Snake River upstream of the North Fork of Snake River. From Montezuma Road, turn right on Hunki Dori Road. Drive to the end and turn right into Hunki Dori parking lot. Sample upstream of walking bridge at the base of the gondola.	No	Yes	Yes	Yes - 100%, 90%, 75%, 50%, 25%, 12.5%, 0%	No	Yes	Yes
SW-050	39 35 57.29 N	105 53 00.04 W	9710	Snake River downstream of Peru Creek. Site is approximately 1/4 to 1/2 mile upstream of EPA site 39355710553. Sample upstream of where Montezuma Road crosses the Snake at the concrete blocks.	No	Yes	Yes	Yes - 100%, 90%, 75%, 50%, 25%, 12.5%, 6.25%, 0%	Yes	Yes	Yes
SW-047	39 35 29.46 N	105 52 17.47 W	9948	Snake River upstream of Peru Creek. Site is in the same location as EPA location 39353010521.	No	Yes	Yes	Yes - 100%, 75%, 56.3%, 42.2%, 31.6%, 0%	Yes	Yes	Yes
SW-045	39 33 50.29 N	105 51 38.80 W	10518	Snake River downstream of Deer Creek confluence. Sample at USGS staff gage downstream of the road. Make sure sample is well-mixed.	No	Yes	Yes	No	No	No	Yes
SW-044	39 33 49.50 N	105 51 37.21 W	10522	Snake River upstream of Deer Creek confluence. Sample at the USGS staff gage.	No	Yes	Yes	No	No	No	Yes

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Sampling Location	Latitude	Longitude	Elevation (HAE)	Description:	Field Parameters		Water Quality	Biology			Sediment Quality
					GPS	Temp, Cond, pH, DO, and Flow	Total and Dissolved Metals, Alk + SO4 + Cl, DOC)	Tox Testing	Macro-invertebrates	Fish	Sediment Metals
Snake River Tributaries (downstream to upstream):											
SW-083	39 36 28.26 N	105 56 27.10 W	9315	North Fork of Snake River upstream of the confluence with Snake River. Sample where Montezuma Road crosses the creek near the large Keystone Parking lot.	No	Yes	Yes	No	No	No	Yes
EPA-12345	39 34 53.97N	105 52 16.46 W	10236	Saints John Creek just upstream of confluence with the snake river.	No	Yes	Yes	No	No	Yes	Yes
EPA-SJA2	39 34 24.69 N	105 52 46.81 W	10635	Saints John mine adit just upstream of confluence with Saints John Creek. Sample taken at road crossing downstream of where the adit flow combines with clean-looking seep water.	No	Yes	Yes	No	No	No	Yes
EPA-SJ2	39 34 12.67 N	105 52 53.91 W	10770	Saints John Creek upstream of confluence with adit flow at road crossing. Sample taken across from cabin "The Berry Home - 1869."	No	Yes	Yes	No	No	Yes ?	Yes
SW-043	39 33 47.49 N	105 51 37.52 W	10536	Deer Creek upstream of confluence with Snake River. Sample in wooded area near the USGS gage about 100+ yards upstream of confluence.	No	Yes	Yes	No	No	Yes low Priority	Yes

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Table 3: Peru Creek sampling locations and samples to be collected at each location.

Sampling Location	Latitude	Longitude	Elevation (HAE)	Description:	Field Parameters		Water Quality	Biology			Sediment Quality
					GPS	Temp, Cond, pH, DO, and Flow	Total and Dissolved Metals, Alk + SO4 + Cl, DOC)	Tox Testing	Macro-invertebrates	Fish	Sediment Metals
Peru Creek (downstream to upstream):											
SW-049	39 35 57.26 N	105 51 27.60 W	10138	Peru Creek 1/2 mile upstream of confluence with the Snake River. Sample where Peru Creek road crosses the creek at the USGS gage. Same location as EPA site 39355710551.	No	Yes	Yes	Yes - 100%, 50%, 25%, 12.5%, 6.25%, 0%	Yes	Yes	Yes
SW-085	39 36 00.38 N	105 50 40.58 W	10336	Peru Creek upstream of Chihuahua Gulch confluence. Access site at the new 1-room cabin that is on the right side of the road going upstream. Sample beside the old mine building and upstream of the waterfall.	No	Yes	Yes	Yes – 100%, 50%, 25%, 12.5%, 6.25%, 0%	No	No	Yes
SW-158	39 35 53.96 N	105 49 41.72 W	10594	Peru Creek midway between Warden and Cinnamon Gulch. Sample just upstream of the "canyon" waterfall area where creek is beside the road. Sample downstream of the beaver ponds.	No	Yes	Yes	No	No	No	Yes
PC-4	39 36 04.56 N	105 49 05.84 W	10736	Peru Creek downstream of Pennsylvania Mine discharge and upstream of Cinnamon Gulch confluence. Access site approximately 200+ meters upstream of an old mine shack on stream right. Site is immediately downstream of a beaver dam with a 4-foot waterfall.	No	Yes	Yes	Yes - 100%, 50%, 25%, 12.5%, 6.25%, 0%	Yes	No	Yes
SW-140	39 36 08.40 N	105 48 41.49 W	10845	Peru Creek upstream of confluence with Pennsylvania Mine discharge. Sample upstream of road crossing.	No	Yes	Yes	Yes – 100%	Yes	Yes	Yes
PC-6	39 36 14.69 N	105 48 20.44 W	10924	Peru Creek upstream of confluence with Shoe Basin and Pennsylvania Mine discharges. Sample where stone house is located beside the road.	No	Yes	Yes	No	No	No	Yes
Peru Creek Tributaries (downstream to upstream):											
SW-Chi	39 36 04.91 N	105 50 36.10 W	10376	Chihuahua Gulch upstream of confluence with Peru Creek. Sample downstream of Peru Creek road crossing.	No	Yes	Yes	No	No	No	Yes

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Table 3: Peru Creek sampling locations and samples to be collected at each location.

Sampling Location	Latitude	Longitude	Elevation (HAE)	Description:	Field Parameters		Water Quality	Biology			Sediment Quality
					GPS	Temp, Cond, pH, DO, and Flow	Total and Dissolved Metals, Alk + SO4 + Cl, DOC)	Tox Testing	Macro-invertebrates	Fish	Sediment Metals
Warden Gulch (downstream to upstream):											
SW-168	39 35 55.91 N	105 49 56.38 W	10485	Warden Gulch upstream of confluence with Peru Creek. Access site beyond the grave and where the power poles cross the road. Look for a flat area just downstream of "the canyon" that has lots of rocks/scree alongside the road. Park here and walk downhill to the site.	Yes	Yes	Yes	No	No	No	Yes
WG-SE	39 35 39.36 N	105 50 00.17 W	10808	Southeast tributary to Warden Gulch. From WGM-1, hike upstream 150+ yards and sample tributary that flows in on stream right. Be sure to sample far enough upstream so flow from a spring located between Warden and the east fork is not collected in the sample.	No	Yes	Yes	No	No	No	Yes
WG-2	39 35 29.88 N	105 50 06.31 W	11033	Warden Gulch upstream of SE tributary at first road crossing. Sample upstream of the road.	No	Yes	Yes	No	No	No	Yes
WG-3	39 35 11.46 N	105 50 06.27 W	11357	East branch of Warden Gulch at road crossing. Continue up the road from WG-2 and sample the first branch at the road crossing. Flow here appears to be less than the west branch.	No	Yes	Yes	No	No	No	Yes
WG-4	39 35 11.05 N	105 50 08.23 W	11353	Western branch of Warden Gulch at road crossing. Continue up the road about 100 meters past WG-3 to access this site. Sample upstream of the road.	No	Yes	Yes	No	No	No	Yes
WG-???				Upstream headwater location on Warden Gulch. Site yet to be determined.	Yes	Yes	Yes	No	No	No	Yes

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Table 3: Peru Creek sampling locations and samples to be collected at each location.

Sampling Location	Latitude	Longitude	Elevation (HAE)	Description:	Field Parameters		Water Quality	Biology			Sediment Quality
					GPS	Temp, Cond, pH, DO, and Flow	Total and Dissolved Metals, Alk + SO4 + Cl, DOC)	Tox Testing	Macro-invertebrates	Fish	Sediment Metals
Cinnamon Gulch (downstream to upstream):											
SW-Cina	39 35 56.69 N	105 48 57.32 W	10913	Cinnamon Gulch ups. of confluence with Peru Creek. Access site by driving Penn Mine road past the concrete house and settling ponds. Stop at the first stream crossing and hike uphill past the Brittle Silver mine dumps and sample upstream of the flow split at the top of a rocky section of hill.	No	Yes	Yes	No	No	No	Yes
SW-Up Cinn	39 35 34.41 N	105 48 49.18 W	11349	Cinnamon Gulch upstream of mine adit flow. Site is downstream of a small inflow that enters on stream right but about 100 yards upstream of old cribbing across the creek.	No	Yes	Yes	No	No	No	Yes
SW-ECinn	39 35 34.08 N	105 48 49.02 W	11349	East fork of Cinnamon Gulch just upstream of confluence with Cinnamon Gulch. Site is just upstream of where the road crosses Cinnamon and is about 100 yards upstream of the mine cribbing / dam structure. Flow enters Cinnamon on stream right.	No	Yes	Yes	No	No	No	Yes
SW-Cinn3	39 35 34.03 N	105 48 49.31 W	11350	Cinnamon Gulch upstream of the confluence with the East Fork of Cinnamon.	No	Yes	Yes	No	No	No	Yes
SS-1	39 35 13.79 N	105 48 55.37 W	11683	Silver Spoon Mine Adit as it comes out of the ground. Site is located at the top of Cinnamon Gulch Road and serves as the headwaters to Cinnamon Gulch.	No	Yes	Yes	No	No	No	No
SW-116	39 36 00.59 N	105 48 45.21 W	10856	Pennsylvania Mine discharge upstream of confluence with Peru Creek. Sample in May 2006 was taken beside the newly constructed concrete building. Location could be moved downstream of the holding pond for future sampling events.	No	Yes	Yes	No	No	No	No
Total Number of Samples:					**1**	**29**	**29**	**6 Def Tests +2 Profiles 42 Test Conc.**	**6**	**9**	**27**

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Table 4: Field parameters to be measured when collecting Peru Creek water quality samples

Variable, Units	Method	Container Type and Preservative	Holding Time	Reporting Limits
Flow, cfs	EPA R8 SOP 722	NA	Field	0.1 cfs
Temperature, EC	EPA 170.1	NA	Field	0.1 EC
Specific Conductance, μSiemens/cm	EPA 120.1	NA	Field	1 ΦSiemens/cm
pH, standard units	EPA 150.1	NA	Field	0.01 su
Dissolved Oxygen, mg/l	EPA 360.1	NA	Field	0.1 mg/l

Table 5: Dissolved metals to be analyzed from Peru Creek water quality samples

Variable, Units	Method	Container Type and Preservative	Holding Time	Required Reporting Limits
Aluminum, μg/l	EPA 200.7	250 ml HDPE Bottle, HNO_3 to pH #2	6 months	30 μg/l
Arsenic, μg/l	EPA 200.8	250 ml HDPE Bottle, HNO_3 to pH #2	6 months	5 μg/l
Cadmium, μg/l	EPA 200.8	250 ml HDPE Bottle, HNO_3 to pH #2	6 months	0.2 μg/l
Calcium, mg/l	EPA 200.7	250 ml HDPE Bottle, HNO_3 to pH #2	6 months	200 μg/l
Chromium, μg/l	EPA 200.7	250 ml HDPE Bottle, HNO_3 to pH #2	6 months	2 μg/l
Copper, μg/l	EPA 200.7	250 ml HDPE Bottle, HNO_3 to pH #2	6 months	10 μg/l
Iron, μg/l	EPA 200.7	250 ml HDPE Bottle, HNO_3 to pH #2	6 months	200μg/l
Lead, μg/l	EPA 200.8	250 ml HDPE Bottle, HNO_3 to pH #2	6 months	1 μg/l
Magnesium, mg/l	EPA 200.7	250 ml HDPE Bottle, HNO_3 to pH #2	6 months	200 μg/l
Manganese, μg/l	EPA 200.7	250 ml HDPE Bottle, HNO_3 to pH #2	6 months	4 μg/l
Nickel, μg/l	EPA 200.8	250 ml HDPE Bottle, HNO_3 to pH #2	6 months	0.5 μg/l
Potassium, mg/l	EPA 200.7	250 ml HDPE Bottle, HNO_3 to pH #2	6 months	1000 μg/l
Selenium, μg/l	EPA 200.8	250 ml HDPE Bottle, HNO_3 to pH #2	6 months	1μg/l
Silver, μg/l	EPA 200.7	250 ml HDPE Bottle, HNO_3 to pH #2	6 months	2 μg/l
Sodium, mg/l	EPA 200.7	250 ml HDPE Bottle, HNO_3 to pH #2	6 months	500 μg/l
Zinc, μg/l	EPA 200.7	250 ml HDPE Bottle, HNO_3 to pH #2	6 months	10 μg/l
Hardness, mg/l	EPA 200.7	Calc. from dissolved Ca and Mg	6 months	0.2 mg/l

Note: "Dissolved Metals" means those metals which will pass through a 0.45 micron membrane filter. The specific method of sample treatment and analysis is defined in Section 4.1.1 of "Methods for Chemical Analysis of Water and Waste," EPA-600/4-79-020, Revised March 1983. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, OH.

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Table 6: Total recoverable metals to be analyzed from Peru Creek water quality samples

Variable, Units	Method	Container Type and Preservative	Holding Time	Required Reporting Limits
Aluminum, μg/l	EPA 200.7	125 ml LDPE Bottle HNO_3 to pH #2	6 months	30 μg/l
Arsenic, μg/l	EPA 200.8	125 ml LDPE Bottle HNO_3 to pH #2	6 months	5 μg/l
Cadmium, μg/l	EPA 200.8	125 ml LDPE Bottle HNO_3 to pH #2	6 months	0.2 μg/l
Chromium, μg/l	EPA 200.7	125 ml LDPE Bottle HNO_3 to pH #2	6 months	2 μg/l
Copper, μg/l	EPA 200.7	125 ml LDPE Bottle HNO_3 to pH #2	6 months	10 μg/l
Iron, μg/l	EPA 200.7	125 ml LDPE Bottle HNO_3 to pH #2	6 months	200 μg/l
Lead, μg/l	EPA 200.8	125 ml LDPE Bottle HNO_3 to pH #2	6 months	1 μg/l
Manganese, μg/l	EPA 200.7	125 ml LDPE Bottle HNO_3 to pH #2	6 months	4 μg/l
Nickel, μg/l	EPA 200.8	125 ml LDPE Bottle HNO_3 to pH #2	6 months	0.5 μg/l
Selenium, μg/l	EPA 200.8	125 ml LDPE Bottle HNO_3 to pH #2	6 months	1 μg/l
Silver, μg/l	EPA 200.7	125 ml LDPE Bottle HNO_3 to pH #2	6 months	2 μg/l
Zinc, μg/l	EPA 200.7	125 ml LDPE Bottle HNO_3 to pH #2	6 months	10 μg/l
Digestion Procedure:				
Total Recoverable Metals - Hotplate	EPA 200.2	"Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements", Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/0100, June 1991		

Note: "Total Recoverable Metals" means the concentration of metals in an unfiltered sample following treatment with hot dilute mineral acid as described in Section 4.1.4 of "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, Revised March 1983. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, OH. If the analytical laboratory is unable to do hotplate digestion, a microwave digestion procedure will also be considered acceptable.

Table 7: Alkalinity, chloride, sulfate, and dissolved organic carbon to be analyzed from Peru Creek water quality samples

Variable, Units	Method	Container Type and Preservative	Holding Time	Required Reporting Limits
Alkalinity, mg/l	EPA 310.1	250 ml HDPE Bottle - Ice to 4C	14 Days	4 mg/l
Chloride, mg/l	EPA 300.0	250 ml HDPE Bottle - Ice to 4C	28 Days	1.0 mg/l
Sulfate, mg/l	EPA 300.0	250 ml HDPE Bottle - Ice to 4C	28 Days	1.0 mg/l
DOC, mg/l	EPA 415.1	250 ml HDPE Bottle, H_3PO_4 to pH#2	28 Days	1.0 mg/l

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Table 8: Macroinvertebrate and Toxicity testing requirements for Peru Creek Samples

Variable, Units	Method	Container Type and Preservative	Holding Time	Required Reporting Limits
96-Hr Acute Toxicity Tests with Rainbow Trout (LC50 in %)	See Below	8 1-Gallon Cubitainers (32 Liters minimum) chilled to 4EC	24 Hours	NA
Macroinvertebrates	EPA R8 Laboratory SOP #705	500 ml to 1000 ml plastic, 70% ETOH	NA	NA

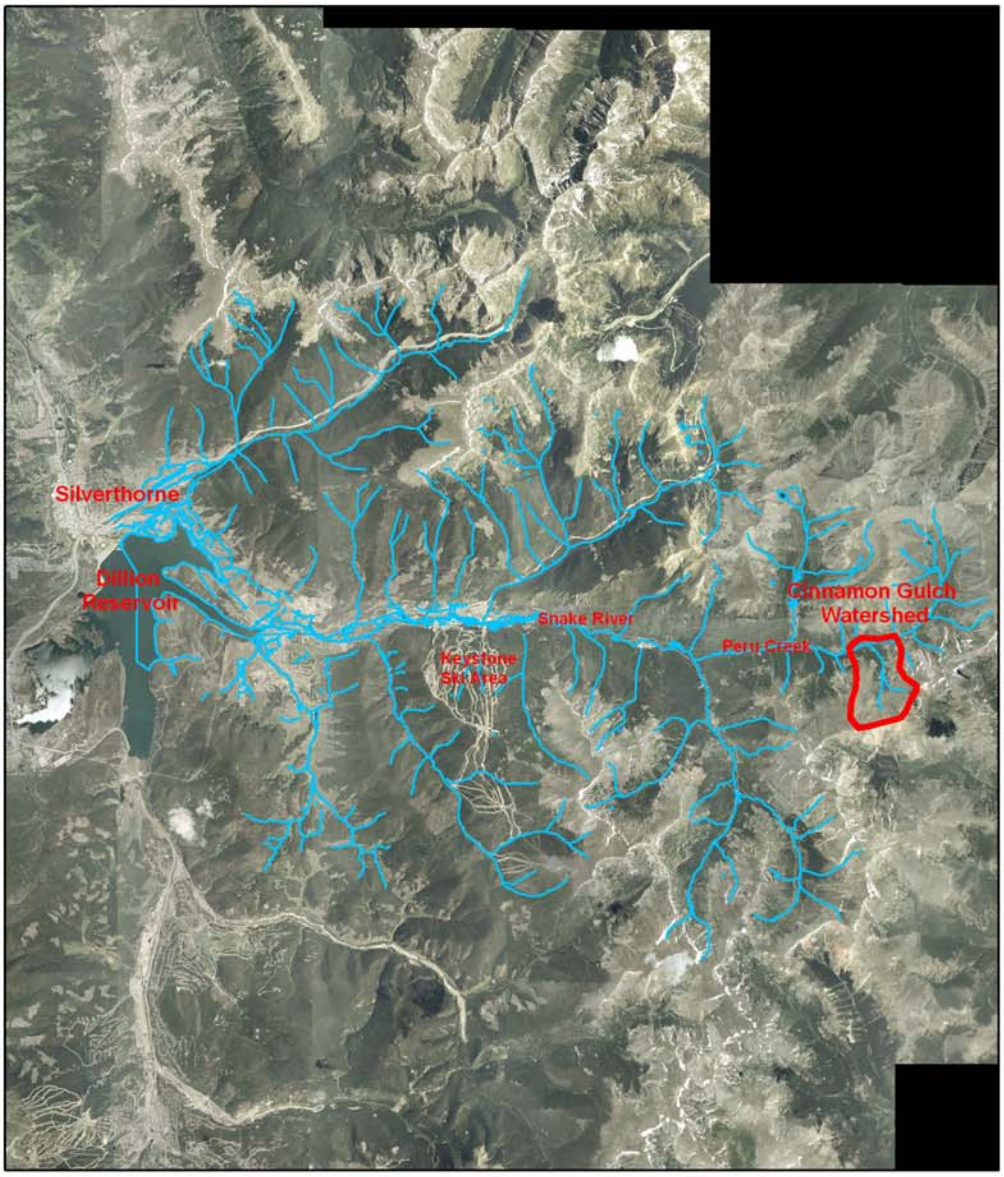
Methods for 48-hr acute toxicity tests will follow procedures outlined in:
"Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms" Fourth Edition, August 1993, Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, OH, EPA/600/4-90/027F.
Note: Toxicity samples will be tested in a definitive series of 100, 50, 25, 12.5, 6.25, and 0 %. If excessive mortality is experienced in the lowest sample dilution within the first 24 hours of the test, the test will be restarted at a lower dilution series.

Table 9: Total recoverable metals to be analyzed from Peru Creek sediment samples

Variable, Units	Method	Container Type and Preservative	Holding Time	Required Reporting Limits
Aluminum, mg/kg	EPA 200.7	60 ml HDPE Bottle, Ice to 4 C	6 months	100 mg/kg
Arsenic, mg/kg	EPA 200.7	60 ml HDPE Bottle, Ice to 4 C	6 months	10 mg/kg
Cadmium, mg/kg	EPA 200.7	60 ml HDPE Bottle, Ice to 4 C	6 months	1 mg/kg
Chromium, mg/kg	EPA 200.7	60 ml HDPE Bottle, Ice to 4 C	6 months	2 mg/kg
Copper, mg/kg	EPA 200.7	60 ml HDPE Bottle, Ice to 4 C	6 months	10 mg/kg
Iron, mg/kg	EPA 200.7	60 ml HDPE Bottle, Ice to 4 C	6 months	200 mg/kg
Lead, mg/kg	EPA 200.7	60 ml HDPE Bottle, Ice to 4 C	6 months	5 mg/kg
Manganese, mg/kg	EPA 200.7	60 ml HDPE Bottle, Ice to 4 C	6 months	4 mg/kg
Nickel, mg/kg	EPA 200.7	60 ml HDPE Bottle, Ice to 4 C	6 months	3 mg/kg
Selenium, mg/kg	EPA 200.7	60 ml HDPE Bottle, Ice to 4 C	6 months	10 mg/kg
Silver, mg/kg	EPA 200.7	60 ml HDPE Bottle, Ice to 4 C	6 months	2 mg/kg
Zinc, mg/l	EPA 200.7	60 ml HDPE Bottle, Ice to 4 C	6 months	10 mg/kg
Digestion Procedure:				
Total Recoverable Metals - Hotplate	EPA 200.2	"Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements", Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/0100, June 1991		

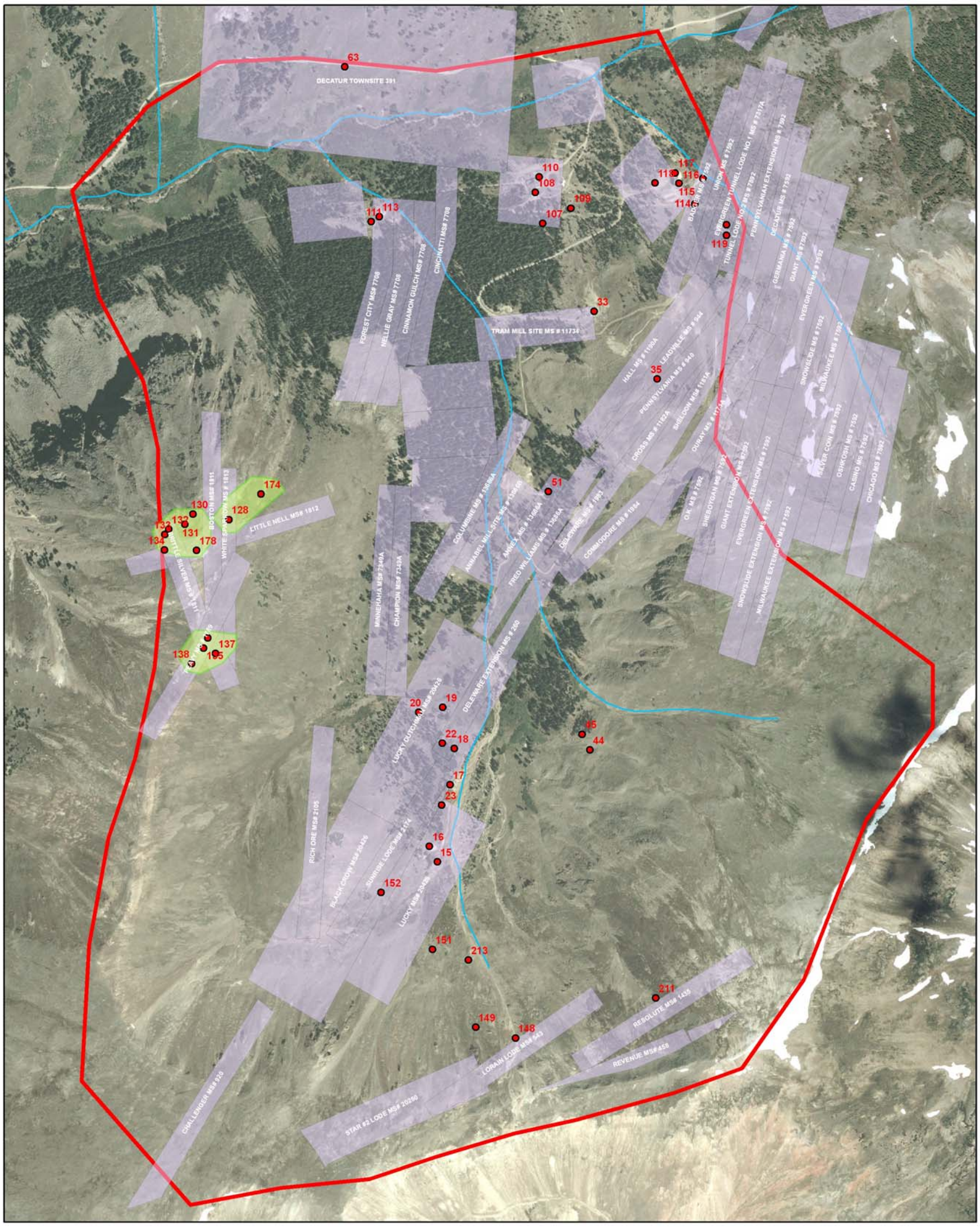
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Figure 1
Cinnamon Gulch and Pennsylvania Mine Site Location



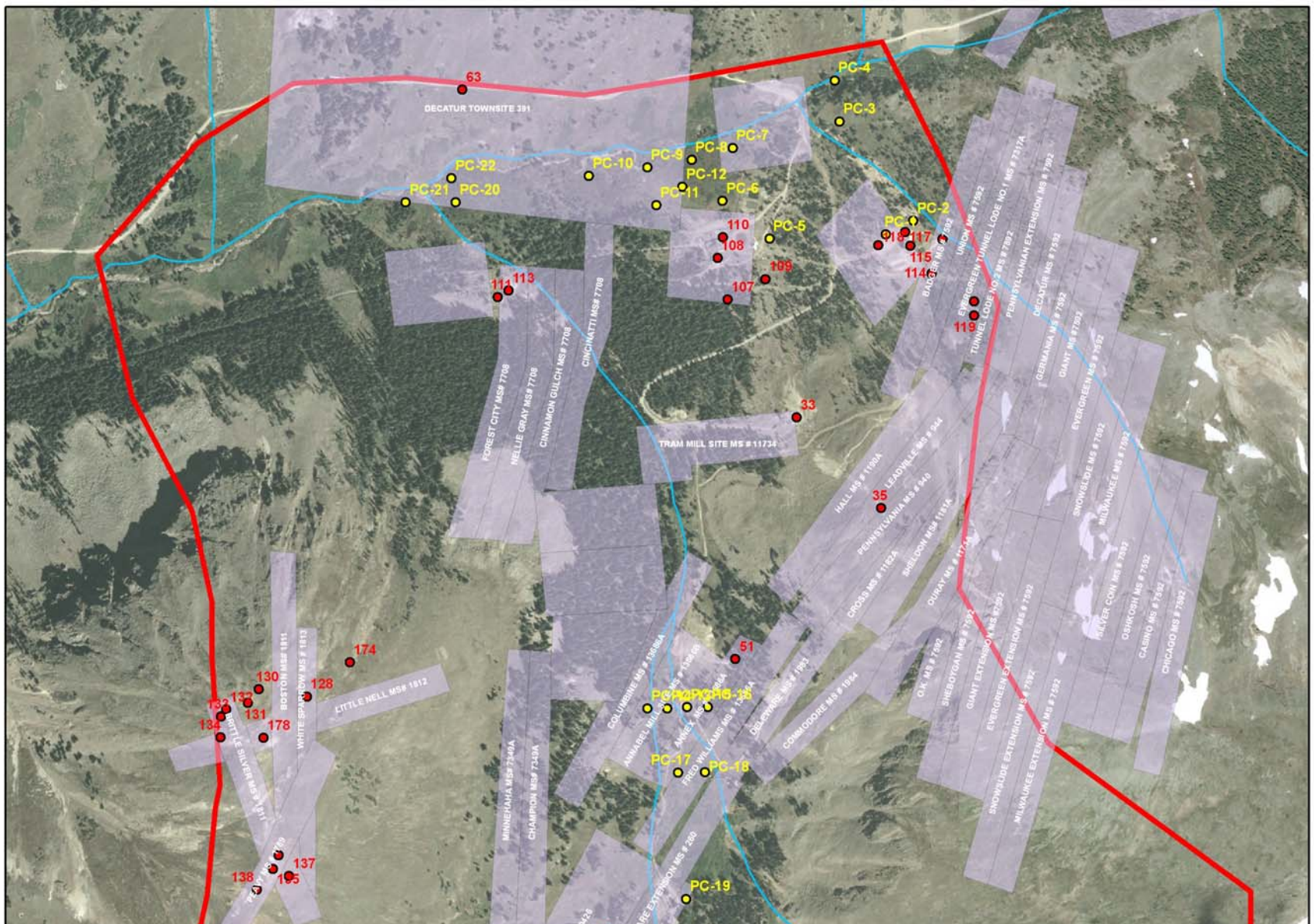
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Figure 2
Cinnamon Gulch and Pennsylvania Mine Site
Mine Waste Sampling Locations



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Figure 3
Cinnamon Gulch and Pennsylvania Mine Site
Water Feature and Ground Water Sample Locations



Legend

- Cinnamon Gulch AGS points to be sampled
- Ground Water Well Location
- Patented Mining Claims
- Cinnamon Gulch Boundary

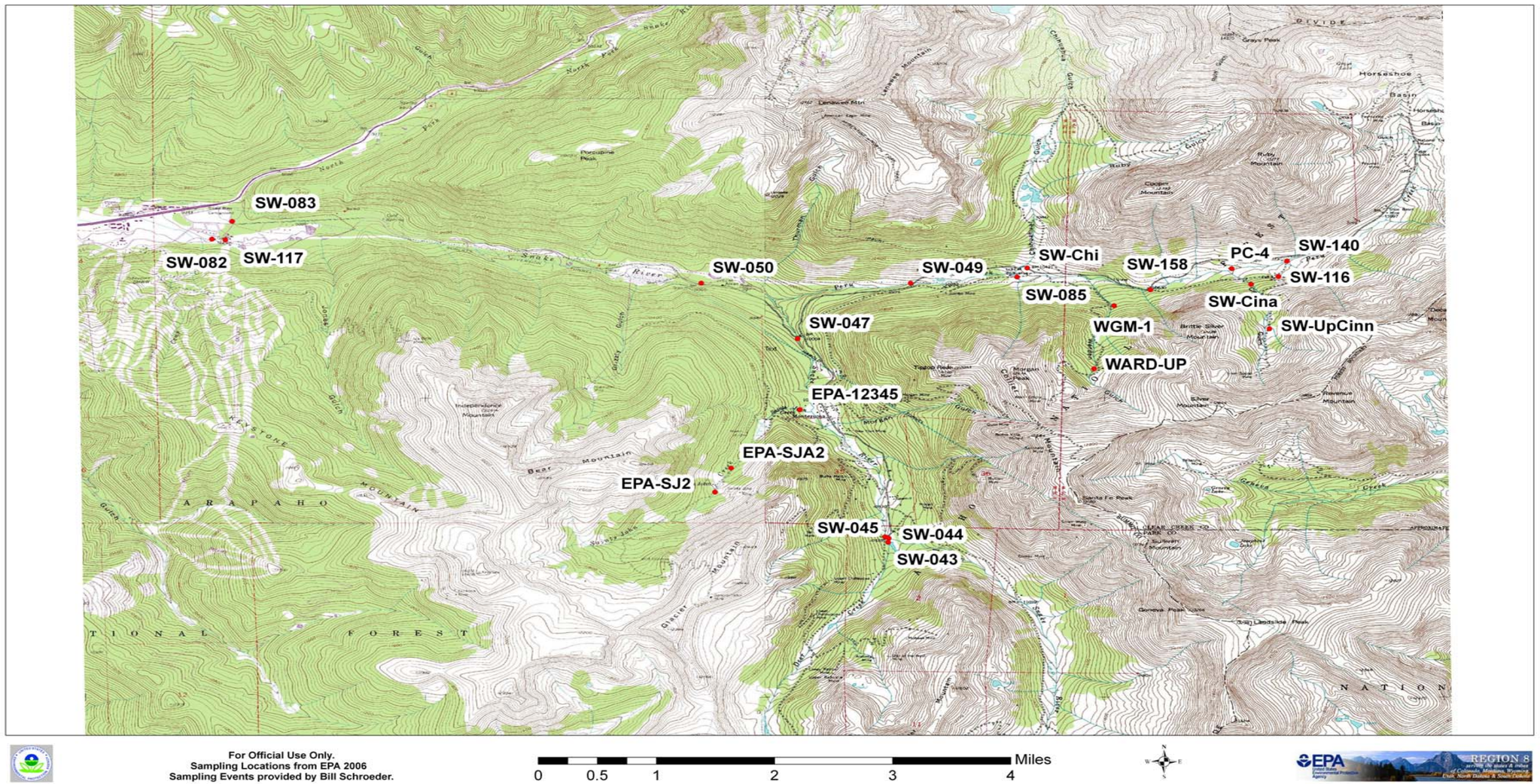
0 70 140 280 420 560 Meters

N

Note - Please refer to Table 2 of text for specific AGS locations with require water feature samples

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Figure 4
Snake River / Peru Creek Water Quality Sampling Locations



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Appendix 1. QUALITY ASSURANCE PROJECT PLAN FOR THE DATA COLLECTION ACTIVITIES OF THE SACRAMENTO RIVER METALS TRANSPORT STUDY

Editors' Note:

The Quality Assurance Project Plan (QAPP) given in Appendix 1 has been edited slightly from its original version. The Plan has been verified against the main report with clarification of some notation. Other areas remain unchanged, though they may differ in the main report; for example, "REC" in the original version of the QAPP remains unchanged but has been changed to "REC_{SPIKE}" and "REC_{SRM}" in the main report. Tables and figures have been reformatted for clarity, typographical errors have been corrected, and references cited in the text but inadvertently left off the references list have been added. No major revisions have been made.

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QUALITY ASSURANCE PROJECT PLAN FOR THE DATA COLLECTION ACTIVITIES OF THE SACRAMENTO RIVER METALS TRANSPORT STUDY

November 1996

Prepared by the U. S. Geological Survey for the
Sacramento Regional County Sanitation District

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1. Title Page and Approvals

REVISED DRAFT QUALITY ASSURANCE PROJECT PLAN FOR THE DATA COLLECTION ACTIVITIES OF THE SACRAMENTO RIVER METALS TRANSPORT STUDY

Implemented by the Sacramento Regional County Sanitation District

Approvals:

Project Manager	Cheryl Cresson, Water Quality Division Chief, SRCSD	Date 12/12/96
QA Manager	Charles N. Alpers, Research Chemist, U.S. Geological Survey	Date 12/4/96
QA Officer	Dr. Howard E. Taylor, Research Chemist, U.S. Geological Survey	Date 12/9/96
State Board Contract Manager	Greg Frantz, California State Water Resources Control Board	Date 12/18/96

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2. Distribution List

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Rosemary Clark, Sacramento Regional County Sanitation District

Greg Frantz, State Water Resources Control Board

Technical Advisory Committee:

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Jim Bybee, National Marine Fisheries Service
Ron Churchill, Department of Mines and Geology, State of California
Val Connor, Regional Water Quality Control Board–Central Valley Region
Bill Croyle, Regional Water Quality Control Board–Central Valley Region
Lee Erickson, Stauffer Management Co.
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Mike Saiki, U.S. Geological Survey, Biological Resources Division
Darell Slotton, University of California–Davis
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Rick Sugarek, U.S. Environmental Protection Agency
Claus Suverkropp, Larry Walker Associates
Jane Vorpagel, Department of Fish and Game, State of California
Dan Welsh, U.S. Fish and Wildlife Service
Ray Zimny, U.S. Army Corps of Engineers

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3. Project Organization and Responsibility

This project will be managed by the Sacramento Regional County Sanitation District (SRCSD). The project manager is Ms. Cheryl Creson, Chief of the Water Quality Division of the SRCSD.

The project quality assurance manager is Dr. Charles N. Alpers, Research Chemist with the U.S. Geological Survey (USGS) in Sacramento. Sample collection and most data acquisition work will be conducted by the USGS.

Analysis of water samples for trace and ultratrace elements (including mercury), and major cations in water, in colloid concentrates, and in sediment extracts will be done by the USGS laboratory in Boulder, Colorado, under the supervision of Dr. Howard E. Taylor, who is the Quality Assurance officer for this work. Dr. Taylor will also assist in training USGS field personnel in sampling and equipment cleaning procedures.

Analysis of nutrients, organic carbon, and major anions in water samples will be done by the USGS National Water Quality Laboratory in Arvada, Colorado. The Quality Assurance officer for this portion of the work is Dr. Peter Rogerson, who is Director of the USGS National Water Quality Laboratory (NWQL).

Analysis of iron redox species in water samples will be done by the USGS in Sacramento, under the direction of Dr. Charles N. Alpers. Grain size distribution of suspended sediment will be done by the USGS laboratory in Vancouver, Washington. Grain size distribution in colloid concentrates will be done at the USGS laboratory in Boulder, Colorado.

Mössbauer spectroscopy will be done by the Technical University in Munich, Germany under the direction of Dr. Udo Schwertmann. Flow measurements will be provided by the USGS, the California Department of Water Resources, the Bureau of Reclamation, and other water agencies responsible for recording stream flow in the study area.

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A technical advisory committee (TAC) will serve as technical reviewers for the project. The TAC consists of staff from the SRCSD, Larry Walker Associates (contractor to the SRCSD), the California State Water Resources Control Board (SWRCB), the Regional Water Quality Control Board - Central Valley Region, the California Department of Conservation (Division of Mines and Geology), the California Dept. of Fish and Game, the U.S. Environmental Protection Agency (EPA) Region IX, CH2M-Hill (contractor to the U.S. EPA), the U.S. Fish and Wildlife Service, the U.S. Geological Survey, the Bureau of Reclamation, The Bureau of Land Management, the U.S. Forest Service, the U.S. Army Corps of Engineers, the National Marine Fisheries Service, the University of California at Davis, the University of California at Santa Cruz, and commercial and recreational mining interests.

4. Project Description

4.1 Project Definition

Metals from abandoned and inactive mines represent a major source of potentially toxic contamination to fish populations of the Sacramento River in northern California. Concentrations of copper (Cu), zinc (Zn), cadmium (Cd), lead (Pb), and mercury (Hg) are of concern regarding aquatic life at several points in the Sacramento River. The winter-run Chinoook salmon, a federally listed endangered species, has critical habitat in the Sacramento River, as do other threatened aquatic species.

In the lower part of the Sacramento Basin, agricultural activities may be an important source of copper to the river because of the widespread application of compounds such as copper sulfate for control of algae in rice fields. Urban runoff represents a third potential source of metals to the river.

Mercury contamination is a well documented problem in several streams tributary to the Sacramento River. Mercury may be transported to the river from natural geological sources in

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the Coast Ranges, from abandoned mercury mines, from base metal mines, and from mercury which was transported into the Sierra Nevada and foothill regions for use in historical gold mining operations. Non-point sources associated with urban land use may also contribute mercury to the river environment.

4.2 Project Objectives and Approach

The overall objective of the study is to quantify the speciation and transport of copper, zinc, lead, cadmium, and mercury in the Sacramento River below Shasta Dam and to identify sources of trace metals to the Sacramento River, including mines, agriculture, and urban runoff. The geochemical studies are designed to determine the processes affecting the transport mechanisms of dissolved metals and metals associated with fine-grained sediments. Improved knowledge of these issues will provide an understanding of how the river responds chemically to introduced metals. This understanding is critical to determining the benefits throughout the watershed of ongoing and proposed remediation at the Iron Mountain Mines Superfund site and at other mines in the vicinity of Shasta Lake and to determining the likelihood that such remediation may or may not lead to the reduction of metal loadings in the Sacramento River.

Three critical unanswered questions with regard to metals in the Sacramento River will be addressed: (1) What is the distribution and speciation of metals in the dissolved, colloidal, and suspended phases? (2) How do metal concentration and speciation change as a function of distance down-river? and (3) What is the relative magnitude of metal sources, including mine drainage, agricultural drainage, and urban runoff?

Water samples will be collected and analyzed at selected locations to characterize the grain size of suspended particles having elevated concentrations of metals and to determine concentrations of dissolved metals. Tangential-flow ultrafiltration using filter membranes rated at 10,000 Nominal Molecular Weight Units (NMWU, or daltons), equivalent to 0.005 μm pore diameter, will be used to process large volumes of river water so that suitable amounts of colloidal material can be concentrated for analysis. Water samples also will be filtered with conventional 0.45 micrometer pore-diameter membranes for comparison with data collected by

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the National Water Quality Assessment Program (NAWQA) of the U.S. Geological Survey and other agencies collecting water-quality data in the Sacramento River Basin. However, the tangential-flow system is expected to provide a more reliable separation of solids because partial clogging of membrane pores is avoided (e.g. Horowitz and others, 1994).

Filtrates from 0.005 equivalent and 0.45 micrometer pore-diameter membranes and colloids concentrated during ultrafiltration will be analyzed chemically to determine the distribution of metals and major elements in different suspended size fractions and in the operationally defined "dissolved" phase. Trace elements and major cations will be determined by inductively coupled plasma/atomic emission spectrometry (ICP–AES) initially, followed by inductively coupled plasma/mass spectrometry (ICP–MS) for all elements with concentrations less than about 500 μg/L. The methodology that will be used was developed by the USGS National Research Program (Garbarino and Taylor, 1995) to achieve state-of-the-art detection limits for trace metals. Concentrations of mercury will be determined by cold vapor atomic fluorescence spectrometry (CV–AFS). Major anions will be determined by ion chromatography (IC). Ferrous and total iron will be determined by using ultraviolet (UV) spectroscopy with ferrozine as the complexing agent (Stookey, 1970). Aqueous speciation and ion-paring analysis will be carried out using the computer program WATEQ4F (Ball and Nordstrom, 1991) or the equivalent update. Light-scattering spectrometry and X-ray sedimentation analysis will be used to characterize the grain size distribution of the colloidal concentrates.

Mineralogy of suspended and colloidal material will be assessed using X-ray diffraction (XRD) and low-temperature Mössbauer spectroscopy, techniques required to determine the crystallinity and structure of poorly crystalline forms of hydrous iron and aluminum oxides and hydroxy-sulfates (Murad and others, 1994). Extended X-ray Absorption Fine Structure (EXAFS) spectroscopy and related methods will be investigated in an attempt to determine the nature of chemical bonds between trace metals and hydrous iron and aluminum oxide particles, and in particular to distinguish between metal adsorption and co-precipitation (e.g. Waychunas and others, 1993). This distinction is important for determining geochemical mechanisms that could lead to bioavailability via release of trace metals from suspended solids, for example: (a) desorption of metals from Fe-Al-hyroxy-sulfate mineral surfaces in response to pH change, (b) release of metals associated with Fe-Al-hydroxy-sulfate minerals by mineral dissolution driven by iron photoreduction of pH change, and (c) oxidative dissolution of mono-sulfide minerals.

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Temperature, specific conductance, pH and alkalinity will be measured in the field.

Bed sediment samples will be collected at all the sampling sites. Sediment samples will be screened using river water to exclude material coarser than 63 μm, consistent with protocols used elsewhere in the Sacramento Basin by USGS as part of the NAWQA program. Sequential extractions will be carried out on bed-sediment samples and colloidal concentrates using a series of increasingly strong acids and oxidizing (or reducing) agents (Chao and Zhou, 1983) to determine the likely mineral or metal-organic hosts for different trace metals (Cu, Zn, Cd, Pb, Hg, Fe, and Al). The extracted solutions will be analyzed by ICP–AES, ICP–MS, and CV–AFS. Minerology and trace-metal bonding characteristics will be evaluated using the techniques previously described (XRD, Mössbauer spectroscopy, and EXAFS).

Sediment pore water will be extracted and analyzed for metal concentrations at sites where suitable fine grained sediment deposits exist, to aid in the evaluation of water-sediment interactions. Aqueous speciation and ion-paring analysis will be carried out on sediment pore water using the computer program WATEQ4F (Ball and Nordstrom, 1991), or updated equivalent, for the sediment environments.

4.3 Sampling Site Locations

Sample site locations for both water and sediment sampling are shown in Figures 1 and 2. The effect of metal concentrations in Spring Creek will be assessed by taking one sample in Keswick Reservoir below Shasta Dam, another sample in the Spring Creek arm of Keswick Reservoir, and a third sample below Keswick Dam. The four other sampling sites for both water and sediment are located on the Sacramento River farther downstream of Keswick Dam. Data from these sites will be used to assess changes in concentrations, loading, and partitioning of metals as they are transported downstream and also to identify and characterize any additional contaminant sources from agricultural and urban land use in the lower areas of the basin. In addition, sediment samples will be taken from five to eight sites along the Sacramento River between Keswick Dam and Red Bluff, plus from one tributary creek near Redding. These additional sediment samples will taken at sites where caddis fly larvae will be collected for a complementary study of metal bioaccumulation.

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Currently identified sampling sites:

Site name	USGS Site number
1) Keswick Reservoir below Shasta Dam	404259122252501
2) Keswick Reservoir, Spring Creek Arm	403750122272301
3) Sacramento River below Keswick Dam	403633122264301
4) Sacramento River near Bend Bridge	11377100
5) Sacramento River at Colusa	11389500
6) Sacramento River near Verona	11425500
7) Sacramento River at Freeport	1144765

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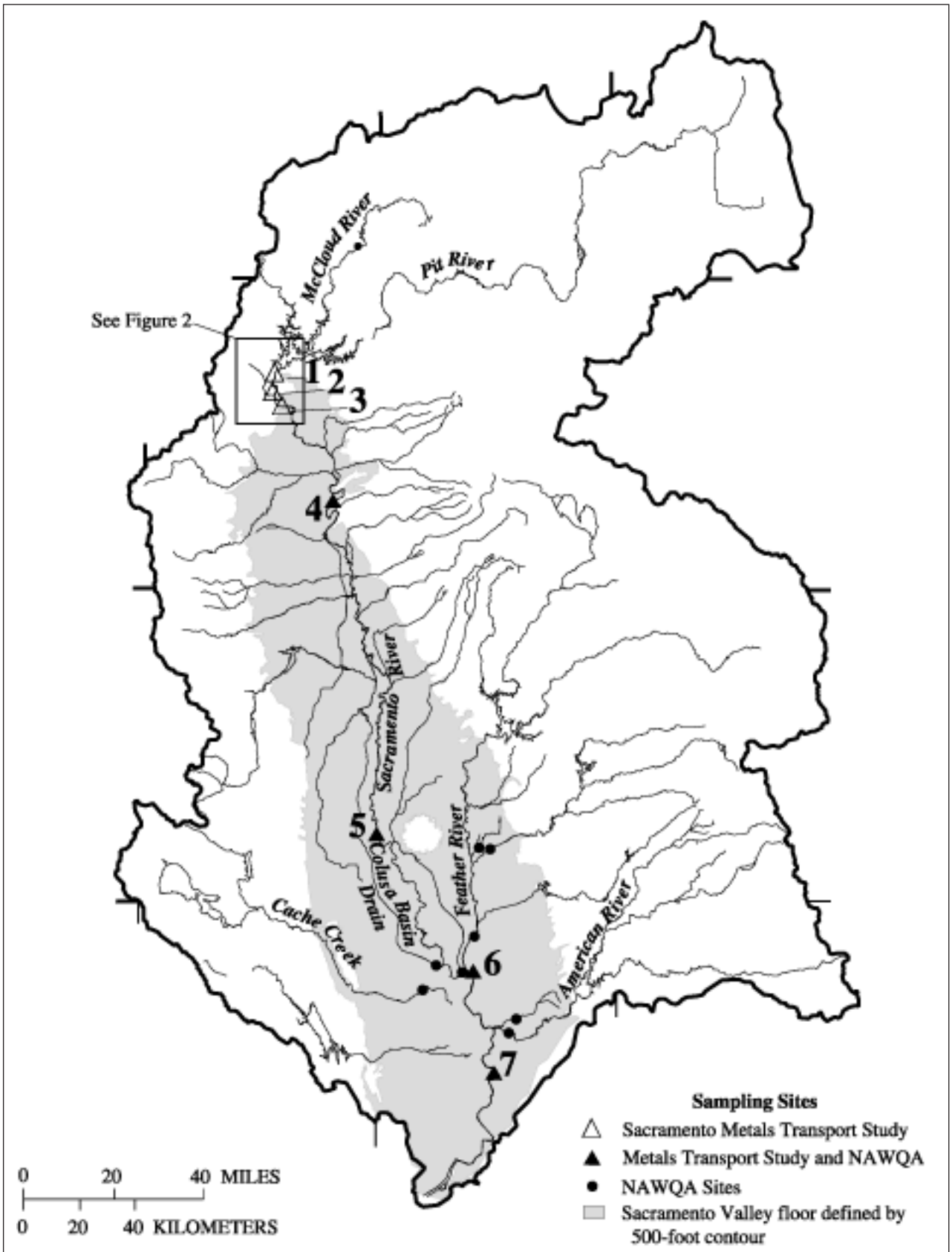


Figure 1. Location of sampling sites and site numbers.

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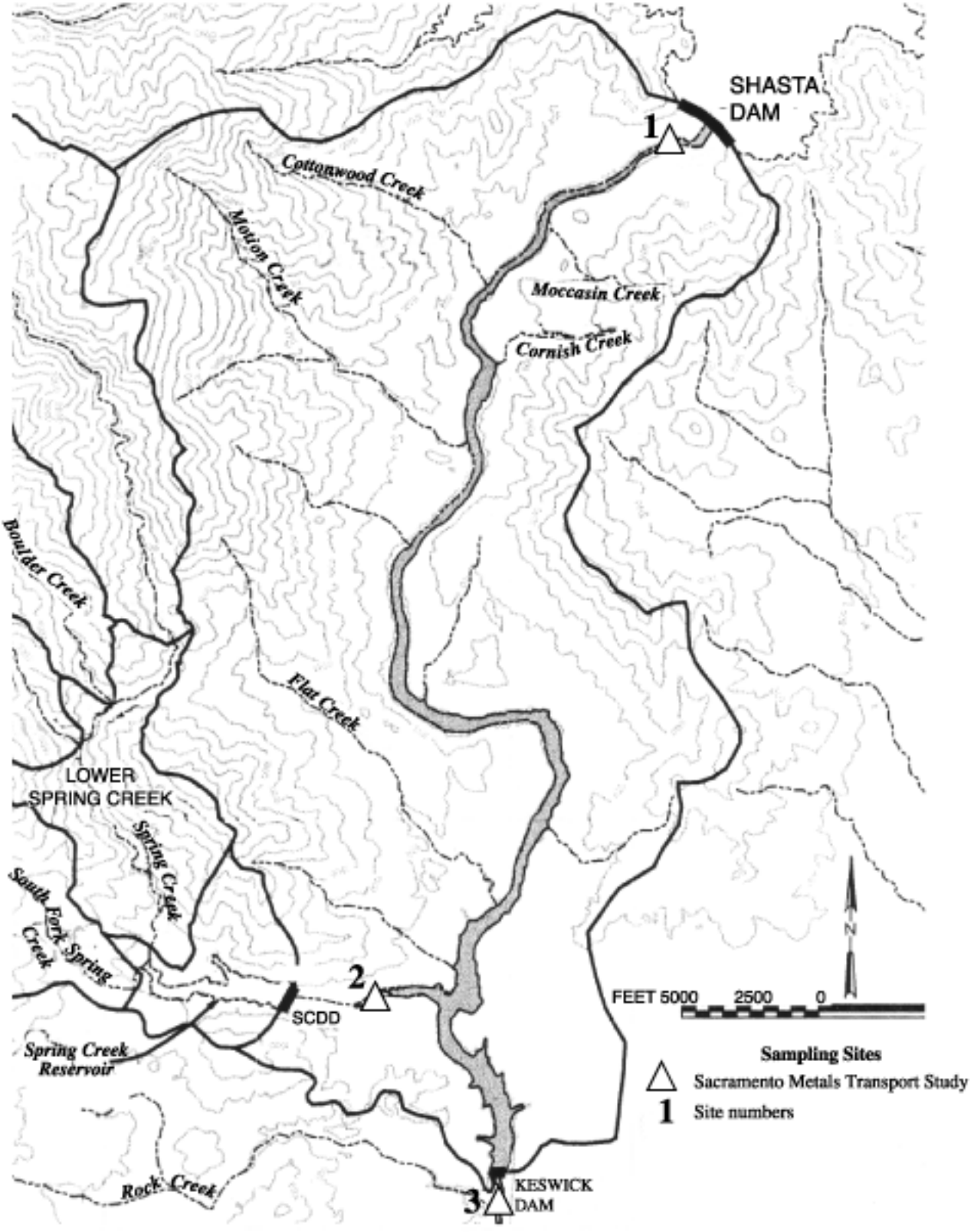


Figure 2. Sites in and below Keswick Reservoir.

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4.4 Sampling Schedule

Sample collection is expected to be done between July 1996 and June 1997. Samples will be collected periodically throughout the year to assess effects of seasonal changes and also during high flows associated with winter storms to assess the effect of these events on the loading and partitioning of metals in the river. A minimum of six water samples and one bed sediment sample will be collected at each site.

1996	July	Water samples
	Aug.	Bottom sediment samples
	Sept.	Water samples
	Nov.	Water samples collected at predicted lowest flows of the year
1997	Dec./Jan.	Water samples collected during storm events
	Feb./Mar	Water samples collected during storm events
	May/June	Water samples collected during the period of rice field drainage
	July	Water samples (if funding is sufficient)

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5. Quality Assurance Objectives for Field and Laboratory Measurements

The objective of data collection in this project is to produce data that represent, as closely as possible, in situ conditions of the sampled water body. This objective will be achieved by using accepted methods to collect and analyze water and sediment samples and by evaluating field and laboratory measurements in terms of detection limits, precision, accuracy, and completeness as summarized in Table 1 through 3. The detection limits in Tables 1 and 2 are based on previous work done by the analytical laboratory (under the direction of Dr. Howard Taylor) that will conduct the analyses for this study. Actual limits of detection and quantitation for this study will be determined using data analysis techniques described by Garbarino and Taylor (1995).

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Table 1. Quality assurance data objectives for chemical analyses of water samples

[concs., concentrations; CV–AFS, cold-vapor/atomic fluorescence spectrometry; IC, ion chromatography; ICP–AES, inductively coupled plasma/atomic emission spectrometry; ICP–MS, inductively coupled plasma/mass spectrometry; IRS, infrared spectrometry; REC, percentage recovery; ROE, residual on evaporation; RPD, relative percentage difference, SRM, Standard Reference Material; UV, ultraviolet spectroscopy, μg/L, microgram per liter; %, percent]

Constituent	Method	Detection Limit	Precision	Accuracy	Completeness
Copper	ICP–MS	0.02 μg/L	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Zinc	ICP–MS	0.08 μg/L	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Lead	ICP–MS	0.06 μg/L	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Cadmium	ICP–MS	0.05 μg/L	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Mercury	CV–AFS	0.0004 μg/L	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Iron	ICP–AES	5 μg/L	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Iron (II) and total (III by difference)	UV	10 μg/L	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Aluminum	ICP–MS	0.2 μg/L	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Sulfate	IC	0.1 mg/L	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Chloride	IC	0.1 mg/L	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%

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Table. 1 "continued"

Constituent	Method	Detection Limit	Precision	Accuracy	Complete-ness
Fluoride	IC	0.1 mg/L	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Total dissolved solids	Gravimetric (ROE)	1 mg/L	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Nitrite	IC	0.01 mg/L as nitrogen	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Nitrate + nitrite	Colorimetric (cadmium reduction-diazotization)	0.05 mg/L as nitrogen	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Ammonia	Colorimetric (salicylate-hypochlorite)	0.01 mg/L as nitrogen	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Ammonia + organic nitrogen	Colorimetric (salicylate-hypochlorite)	0.20 mg/L as nitrogen	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Phosphorus (total)	Colorimetric (phosphomolyb-date)	0.01 mg/L as phosphorus	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Phosphorus (orthophos-phate	Colorimetric (phosphomolyb-date)	0.01 mg/L as phosphorus	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Organic carbon	Wet oxidation, IRS	0.1 mg/L	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%

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Table 2. Quality assurance data objectives for chemical analyses of bed-sediment samples and colloid concentrates, including sequential extractions

[concs., concentrations; ICP–MS, inductively coupled plasma/mass spectrometry; CV–AFS, cold-vapor/atomic fluorescence spectrometry; ICP–AES, inductively coupled plasma/atomic emission spectrometry; REC, percent recovery; ROE, residual on evaporation; RPD, relative percentage difference, SRM, Standard Reference Material; %, percent; µg/g, microgram per gram]

Constituent	Method	Detection Limit	Precision	Accuracy	Complete-ness
Copper reduction oxidation residual	ICP–MS	 0.3 0.4 2	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Zinc reduction oxidation residual	ICP–MS	 0.1 4 5	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Lead reduction oxidation residual	ICP–MS	 0.3 1.5 2	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Cadmium reduction oxidation residual	ICP–MS	 0.3 1.5 3	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Mercury reduction oxidation residual	CV–AFS	 0.004 0.02 0.06	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Iron (total) reduction oxidation residual	ICP–AES	 1 5 10	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%
Aluminum reduction oxidation residual	ICP–MS	 2 10 20	Duplicate RPD ≤25% or ±50% at concs. <10 times the detection limit	SRM REC or Lab Spike = 100% ±25% or ±50% at concs. <10 times the detection limit	90%

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Table 3. Quality assurance data objectives for field measurements of water quality

[DIFF, diffraction; REC, percentage recovery. °C, degree Celsius. %, percent. mg/L, milligram per liter]

Measurement	Method	Accuracy	Completeness
Alkalinity	Gran titration	REC = 100% ±25%	90%
pH	Electrochemical	DIFF <0.1 unit	90%
Temperature	Thermistor	DIFF <1°C	90%
Dissolved oxygen	Electrochemical	DIFF <0.2 mg/L	90%
Specific conductance	Electrical resistance	DIFF ±3%	90%

6. Specific Routine Procedures Used to Assess Data

Detection limits used in this study are the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. Detection limits must be low enough to evaluate the presence of a substance at the concentrations of interest.

Precision (or variability) is the degree of similarity among independent measurements of the same quantity. The precision of laboratory analytical data is evaluated by randomly submitted split samples and evaluated in terms of relative percentage difference (RPD).

$$\text{RPD} = \frac{\text{difference between reported values}}{\text{average reported value}} \times 100$$

Accuracy (bias or systematic error) is defined in this study as the measure of the degree of conformance of values generated by a specific method with the true or expected value of that measurement. The accuracy of field measurements is evaluated by the use of standard methods of analysis with the appropriate calibration standards. The accuracy of laboratory analytical data is assessed by analyzing standard reference materials (SRM) or the recovery of known concentrations of analytes in spiked samples and calculating the percent recovery (REC) where:

$$\text{RPD} = \frac{\text{measured value}}{\text{expected value}} \times 100$$

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Both precision and accuracy will be enhanced by the use of standardized methods for sample collection, handling and preservation and the analysis of field and laboratory blank samples.

Completeness is measured as the percent of valid analyses (those meeting the accuracy and precision objectives). Water samples will be taken at seven sites on at least six occasions for a total of at least 42 samples. Therefore, to achieve 90 percent completeness, 38 of 42 samples must meet the quality assurance data objectives.

7. Sample Collection

Sampling procedures and many of the analytical methods used in this study are identical to those used in the USGS NAWQA project currently underway in the Sacramento River Basin. Data produced by the two studies will be directly comparable to one another.

Representative water samples for total and dissolved constituent analysis will be collected concurrently with samples for colloidal analysis at each site.

7.1 Surface Water Sampling Procedures

Samples from sites accessible by bridge only (Sacramento River below Shasta Dam and Sacramento River at Bend Bridge, during high flow) will be collected using a USGS D77 sampler (Horowitz and others, 1994) shown in Figure 3. The bronze body of the sampler has been epoxy coated to eliminate potential contamination from the sampler itself. The 3-L sample bottle and all parts of the equipment that contact the sample are made of Teflon. The sampler will be suspended from a boat at other sites where it is used. The D-77 sampler is designed to fill the sample bottle in an isokinetic manner (the water enters the sampler at the same velocity as the

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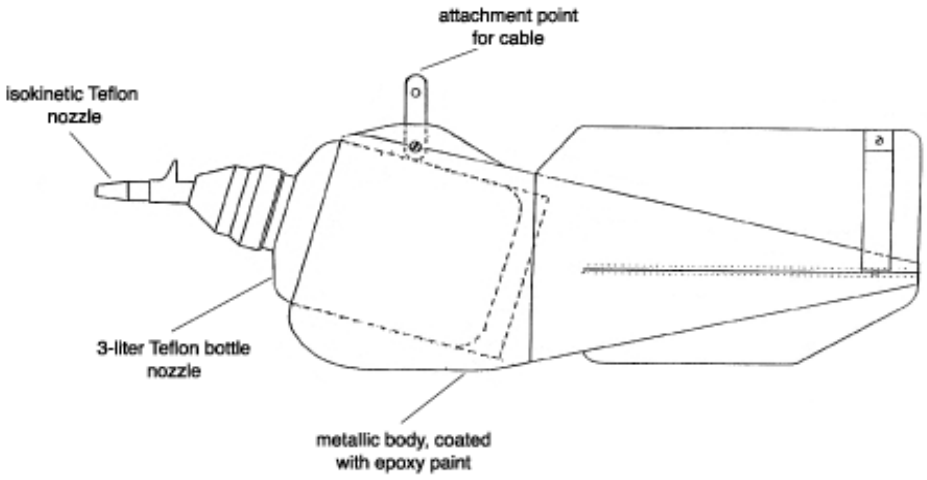


Figure 3. Depth-integrating sampler, D-77.

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water near the sampler) to ensure that concentrations of suspended sediment particles are representative of concentrations in the water sampled. This equipment is capable of collecting an isokinetic sample to a depth of about 15 feet. If this depth is exceeded at a sampling station, a modification of the sampler will be made to extend its depth range. This modification consists of using a perforated polyethylene bottle with a Teflon bag liner in place of a standard 3-L bottle. A set of Teflon bags will be dedicated to each sampling site to minimize potential for contamination between sites.

Samples from the Sacramento River below Keswick Dam will be collected with Teflon tubing using a peristaltic pump from near shore directly into Teflon bottles and polyethylene carboys. The narrow and turbulent channel at this location appears very well mixed and boat access is not feasible. The same approach will be used to sample the Sacramento River below Shasta Dam at low flows. The sites accessible by boat (Spring Creek arm of Keswick Reservoir, and the Sacramento River at Colusa, Verona, and Freeport) also will be sampled using Teflon tubing and a peristaltic pump. The tubing will be secured in 10-feet of polyvinyl chloride (PVC) pipe and suspended into the water column from the bow of the boat to minimize potential contamination. During sample collection, the depth of the tubing intake will be varied between 1 and 9 feet below the river surface.

Mercury and lead will be sampled from a single vertical traverse near the middle of the river. Composite samples for other trace metals, major ions, and other constituents will be collected using equal-discharge-increment methods (Edwards and Glysson, 1988) which provide a cross-section transect sample whose concentration is discharge weighted, both vertically and laterally. Between 5 and 15 lateral points will be sampled on each cross-section. Because of the size of the river and the minimum fill rates of the D77 sampler, sample volume is expected to be between 6 to 20 L and will require multiple sample bottles. Samples for total and dissolved constituents will remain in the 3-L Teflon bottle in which they were collected until processing, or will be composited in a Teflon-lined churn. Because a large volume of water is needed to recover an adequate mass of colloidal material, samples collected for colloid analysis from the vertical stations on a cross-section transect will be composited in one or more 25-L polyethylene carboys. Concentrations of metals in the colloidal fractions are expected to be high enough that any slight

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contamination or adsorption resulting from the use of polyethylene rather than Teflon containers should not be significant. Each site will have a dedicated set of carboys that will be used only at that site in order to minimize the potential for contamination between sites.

Samples for mercury and lead analyses will be collected from just one sampling point located at the center of flow using a specially cleaned sample bottle and nozzle. While a width integrated sample provides more confidence that the sample is truly representative of the river at the time of sampling, it also exposes the sample to the atmosphere and potential contamination for a longer period of time. Because concentrations of dissolved mercury and lead are expected to be low, it will be critical to achieve minimal levels of contamination.

7.1.1 Subsample requirements

Once a representative sample of the river water has been collected, a variety of subsamples will be split and processed before shipping to laboratories. Processing will take place in a mobile laboratory that was built for use in the USGS NAWQA program. The mobile lab allows for efficient sample preparation in a clean environment. Subsample requirements for the project are listed in Table 4.

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Table 4. Subsamples of water required for scheduled analyses

[*, triple-distilled HNO_3 for ultratrace element preservation; (a), a split sample will be held for archive purposes. conc., concentration; CA, California, CO, Colorado; EXAFS, extended X-ray absorption fine structure; NMWL, nominal molecular weight limit; Poly, polyethylene; USGS, U.S. Geological Survey; XRD, X-ray diffraction. μm, micrometer]

Matrix/filtration	Analysis	Preservation	Bottle type, volume	Laboratory
Whole water	Mercury	Oxidize ($K_2Cr_2O_7$) Acidify (HNO_3*)	Glass, 125 mL (a)	USGS–Boulder, CO
	Lead	Acidify (HNO_3*)	Teflon, 250 mL	USGS–Boulder, CO
	Major/trace elements (cations)	Acidify (HNO_3*)	Poly, 125 mL (a)	USGS–Boulder, CO
	Major elements (anions)	None	Poly, 125 mL (a)	USGS–Arvada, CO
	Suspended sediment conc.	None	Poly, 1,000 mL	USGS–Salinas, CA
0.45 μm filtrate (silver filter)	Organic carbon, dissolved	None	Glass, baked, 125 mL	USGS–Arvada, CO
Silver filter retentate	Organic carbon suspended	None	Petri dish	USGS–Arvada, CO
0.45 μm filtrate (capsule filter)	Mercury	Oxidize ($K_2Cr_2O_7$) Acidify (HNO_3*)	Glass, 125 mL (a)	USGS–Boulder, CO
	Lead	Acidify (HNO_3*)	Teflon, 250 mL	USGS–Boulder, CO
	Major/trace elements (cations)	Acidify	Poly, 125 mL (a)	USGS–Boulder, CO
	Major elements (anions)	None	Poly, 125 mL	USGS–Arvada, CO
	Iron, redox speciation	Acidify (HCl), chill	Poly, amber, 125 mL	USGS–Sacramento, CA
	Nutrients	Chill	Poly, amber, 125 mL	USGS–Arvada, CO
10,000 NMWL filtrate 0.005 μm equivalent	Mercury	Oxidize ($K_2Cr_2O_7$) Acidify (HNO_3*)	Glass, 125 mL (a)	USGS–Boulder, CO
	Lead	Acidify (HNO_3*)	Teflon, 250 mL	USGS–Boulder, CO
	Lead isotopes	Acidify (HNO_3*)	Teflon, 1 L	USGS–Denver, CO
	Major/trace elements (cations)	Acidify (HNO_3*)	Poly, 125 mL (a)	USGS–Boulder, CO
	Major elements (anions)	None	Poly, 125 mL (a)	USGS–Arvada, CO
	Iron, redox speciation	Acidify (HCl), chill	Poly, amber, 125 mL	USGS–Sacramento, CA

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Table 4. "*continued*"

Matrix/filtration	Analysis	Preservation	Bottle type, volume	Laboratory
Colloidal concentrate	Major/trace elements (cations) total and sequential extractions	Chill	Teflon, 500–1,000 mL	USGS–Boulder, CO
	Lead, total and isotopes	Acidify (HNO_3*)	Teflon, 250 mL	USGS–Denver, CO
	XRD	Chill	Poly, 100 mL	USGS–Sacramento, CA
	EXAFS	Chill	Poly, 100 mL	USGS–Menlo Park, CA Stanford University
	Mössbauer spectroscopy	Freeze dry	Poly, wide-mouth, 200 mL	Technical University, Munich, Germany
	Particle size distribution (light scattering spectrometry)	Chill	Poly, 100 mL	USGS–Boulder, CO

7.1.2 Sample processing and Preservation

7.1.2.1 Splitting

The cone splitter (Figure 4) is used by the USGS NAWQA program as the primary splitter to divide the collected sample into subsamples for inorganic-constituent, and suspended-sediment analyses (Ward and Harr, 1990; Capel and others, 1995). Subsamples for filtered inorganic-constituent, suspended-sediment, and field analyses will be collected from the first set of split samples from the cone splitter. Subsequent splits will be used to collect subsamples for raw (unfiltered) inorganic-constituent analyses. Samples for tangential-flow filtration will be composited in a Teflon-lined churn, from which water will be pumped using Teflon tubing.

Samples from the D-77 sampler will be collected in several (two to five) 3-L Teflon bottles

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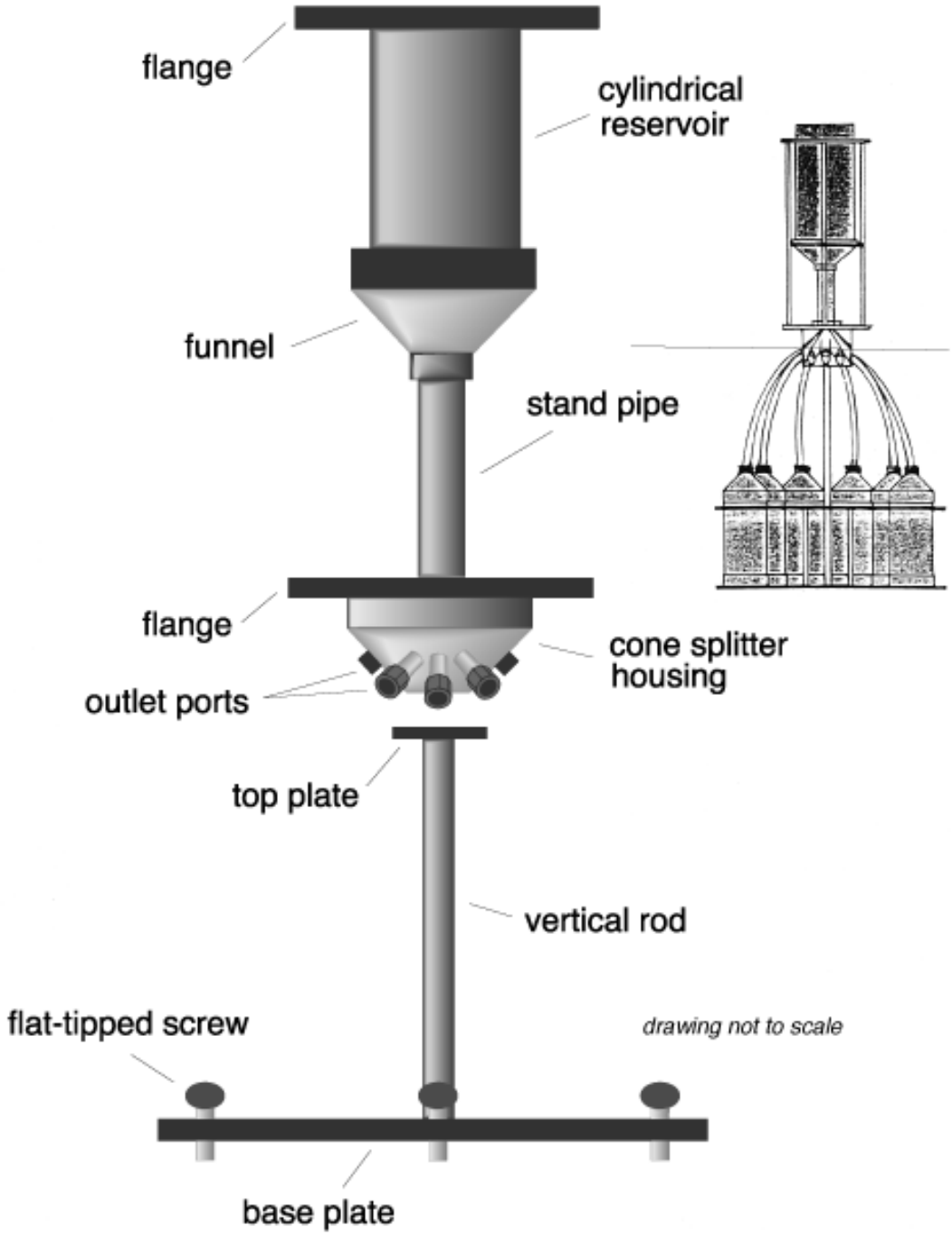


Figure 4. The all-Teflon cone splitter and stand.

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and later poured into the cone splitter. This method allows the cone splitter to function as both a splitter and compositor.

The splitting process is as follows:

1. Set up the cone splitter on a flat, open area. A level splitter is critical to performance. All Teflon tubes should be approximately the same length and the entire apparatus including subsample containers is tented in a plastic bag. Water is poured directly into the splitter reservoir through a hole in the plastic tent, which is covered between pours.

2. Field rinse all sample-collection and splitting equipment with native water. Collect the rinse water near the shore to avoid heavy suspended sediments. Pour rinse water from the D-77 sample bottle through the Teflon cap and nozzle and into the cone splitter. Three 1-L rinses are effective then one 3-L rinse.

3. Place subsample containers under each outlet tube. The tubes need only extend into the receiving containers far enough to prevent spillage.

4. Agitate the sample (10 to 15 seconds) in the D-77 bottle to resuspend the sediments. Invert the bottle over the cone splitter reservoir. Sample transfer should be rapid. Maintain a head of water above the standpipe to prevent air from entering the splitting block.

5. Remove subsample containers from cone splitter and cap immediately.

6. An additional split is necessary to obtain the smaller volumes of some required subsamples. Reload splitter ports with the required bottles and pour a subsample from the first set of split samples.

7. Disassemble the cone splitter after completing the sample processing and clean before reuse or storing.

7.1.2.2 Filtration–0.45 μm filter, organic carbon

The filtration procedure for dissolved and suspended organic carbon analyses uses a stainless-steel or Teflon-pressure filter assembly fitted with a 47-mm diameter, silver, 0.45-μm

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pore-size filter and a peristaltic pump that has been fitted with Tygon tubing to filter the sample. Filtering and processing methods follow NAWQA protocol (Shelton, 1994).

7.1.2.3 Filtration–0.45 μm, inorganic constituents

A disposable 0.45 μm capsule filter (Gelman 12175) will be used to filter samples for major and trace element analyses. This filter is currently being used by the NAWQA program in the Sacramento Basin and the 0.45 μm pore size is used to define the dissolved phase in most water-quality studies. The use of this filter will facilitate comparison of results obtained by this study with results from NAWQA and other studies in the basin. Data obtained with the 0.45 μm filter will also be used to compare the results from standard versus ultrafiltration methods.

A peristaltic pump head with Tygon tubing or a Teflon diaphragm pump head with corrugated Teflon tubing will be used to create the pressure required to force samples through the filter units. The detailed procedures follow NAWQA protocols (Shelton, 1994).

7.1.2.4 Filtration–10,000 NMWU (0.005 μm equivalent)

A Minitan tangential-flow filter apparatus will be used to obtain a filtered sample for analysis of dissolved constituents defined by the less than 0.005 μm equivalent particle size. A Pellicon tangential-flow filter apparatus will be used to obtain a concentrate of colloidal material. Cleaning procedures will be the same as for other equipment that contacts the water sample. To further minimize contamination, a dedicated set of filter membranes and tubing for both Minitan and Pellicon filters will be used at each sampling site. The carboy(s) containing the sample collected for colloid processing will be allowed to sit quietly for an hour before filtration to let the coarser grained suspended sediment settle to the bottom of the container. Water will be pumped from a fixed distance above the sediment layer in order to eliminate the larger size fractions from the colloidal concentrate.

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7.1.2.5 Preservation

Samples for trace metal analyses and major cations will be stabilized by acidifying to a pH of 2 or less with ultrapure (distilled) concentrated nitric acid. In addition, potassium dichromate will be added to samples for mercury analysis. Samples for redox speciation will be stabilized by acidifying to a pH of 2 or less with ultrapure (distilled) hydrochloric acid, then chilled on ice. Samples collected for organic carbon and nutrient analysis will be chilled on ice during storage and transportation to the lab. Preservation of all samples to be collected is summarized in Table 4. Samples will be processed inside a plastic tent set up on a counter of the mobile laboratory to minimize atmospheric contamination during handling.

7.2 Bed Sediment Sampling Procedures

Sediment samples will be collected using either a coring device for deep-water sites or large plastic spoons for shallow-water sites. Sediment cores will be collected in pre-washed, acid-cleaned 10.2-cm diameter, acrylic butyrate core liners placed in a gravity corer. The gravity corer will be slowly lowered into the sediment to avoid disturbance of the sediment-water interface. The cores will be kept upright and carefully transported to a field-based laboratory. Shallow-water sampling will be carried out according to USGS NAWQA protocols (Shelton and Capel, 1994). For samples for which it is determined that extraction of pore waters will not be needed, samples will be screened with a nylon screen and river water to exclude material greater than 63 μm.

7.2.1 Subsample requirements

Once a sample of the bed sediment has been collected, a variety of subsamples will be split and

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processed before shipping to laboratories. Subsample requirements are listed in Table 5.

Table 5. Subsamples of bed sediment required for scheduled analyses

[EXAFS, extended X-ray absorption fine structure; NMWL, nominal molecular weight limit; Poly, polyethylene; USGS, U.S. Geological Survey; XRD, X-ray diffraction. mL, milliliter]

Matrix	Analysis	Preservation	Bottle type, volume
Pore water	Major/trace elements (cations)	Acidify	Poly, 125 mL
	Major elements (anions)	None	Poly, 125 mL
Bed sediment	Tract elements, sequential extractions	Chill	Poly, wide-mouth 100 mL
	Sulfur speciation	Chill	Poly, wide-mouth 100 mL
	XRD	Chill	Poly, wide-mouth 100 mL
	EXAFS	Chill	Poly, wide-mouth 100 mL
	Mössbauer spectroscopy	Freeze-dry	Poly, wide-mouth 100 mL
	Particle size distribution (sieve, sedigraph)	Chill	Poly, wide-mouth 100 mL

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7.2.2 Sample Processing and Preservation

Cores selected for pore water extraction will be sectioned for sediment and associated porewater within 8 to 24 hours of collection. The water overlying the sediment will first be siphoned off to within 1 to 10 cm of the interface using Tygon tubing. A sample of this overlying water will then be taken with a 10- to 30-mL plastic syringe. The cores will be extruded and sectioned into appropriate depth intervals in a nitrogen-filled glove bag, to minimize oxidation of the pore water. The individual core sections will then be placed in nitrogen-filled 50-mL centrifuge tubes. Subsamples of whole sediment will also be taken for determination of water content and grain-size distribution. The centrifuge tubes will be removed from the glove box and centrifuged at 1500 rpm for 20 minutes to separate porewater and sediment. The tubes will then be returned to the glove box and the supernatant extracted with pre-cleaned 2-mL plastic syringes and filtered through 0.2-μm cartridge filters. Filtered portions will be placed into acid-cleaned 30-mL polyethylene bottles for metal determinations. If there is sufficient volume, portions of pore water will also be taken for alkalinity and anion determinations. The pore water, samples for metal analysis will be acidified to pH < 2 with ultrapure, concentrated nitric acid. Sediment samples will be placed in acid washed containers and packed in ice for transport to the analytical laboratories.

7.3 Equipment cleaning

The sample collecting and processing equipment is soaked in dilute phosphate-free detergent solution, rinsed with tap water, soaked in 5.0 percent hydrochloric acid (HCl), and rinsed extensively with deionized water prior to each field trip and between sites. Detergents and acids will be used with care to avoid possible contamination of the sample by their residue. A thorough native-water rinse is required at each field site before sampling to remove any remaining cleaning agents and equilibrate the equipment to the sampling conditions. Details on procedures are outlined below.

The sampler bottle, cap and nozzle, cone splitter, churn splitter, filter support, pumphead,

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tubing, and any other equipment that will contact the sample are cleaned prior to each field trip and between sites as follows:

1. Disassemble (if necessary) wearing vinyl gloves.
2. Soak for 30 minutes in a 0.2-percent solution of phosphate-free detergent and scrub with a nonmetallic brush. Use a small bottle brush for the cone-splitter parts.
3. Change gloves and rinse thoroughly with warm tap water to remove all soap residue.
4. Soak for 30 minutes in a solution of 5.0-percent hydrochloric acid. Swirling the equipment in the acid solution will adequately desorb any metals not removed during the washing process. The used acid/water solution should be placed in a waste container for proper disposal.
5. Change gloves and rinse three times with deionized water.
6. Protect areas of the equipment that will contact the sample with Teflon tape and place in a sealable plastic bag for storage and transport.
7. Rinse sampling and splitting equipment at the site with 2- to 3-L of native water before sampling.
8. Rinse sampling and splitting equipment with deionized water immediately after each use.

Equipment used for filtering the organic-carbon samples will be baked at 450°C for 2 hours or cleaned using organic-free deionized water and aggressive scrubbing. Equipment will be isolated from any procedure using methanol.

8. Sample Custody

All bottles will be clearly labeled with a waterproof marker or preprinted labels so the laboratories can identify the samples. The minimum information required is the project name, site identification number, date and time, and type of analysis requested.

An analytical services request form with the same information as the bottle labels will be included with each sample. Copies of the forms will be retained by data management personnel.

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Samples will be shipped to laboratories by Federal Express overnight service from Sacramento or Redding, California, within 24 hours of collection and processing. Laboratories will notify data management personnel by electronic mail or standard mail service upon the arrival of samples.

9. Field Measurements

Specific conductance, dissolved oxygen, temperature, and pH, will be determined using the detailed procedures in the NAWQA field guide (Shelton, 1994). Alkalinity will be determined by Gran titration (Stumm and Morgan, 1981) using 0.16 N hydrochloric acid with a Hach digital titrator. Specific conductance, pH, and alkalinity will be determined from a split of the unfiltered water sample. Dissolved oxygen and temperature will be measured instream at the center of flow when feasible, otherwise from near shore. Stream discharge will be determined at all river sites. Sites 4 through 7 are located at USGS gaging stations and discharge will be estimated using current stage-discharge ratings from measurements of stage at the time of sampling (Rantz and others, 1982). Discharge at site 3 (below Keswick Dam) will be determined from BOR flow release records at Keswick Dam. Flow into the Spring Creek arm of Keswick Reservoir will be determined from Spring Creek Reservoir and power plant release records.

10. Calibration Procedures and Frequency

All laboratory apparatuses (analytical balances, volumetric equipment, deionized water systems, etc. are calibrated on at least an annual basis. Laboratory instruments (ICP–AES, ICP–MS, etc.) are recalibrated with each batch of samples that are analyzed. Calibration standards are rerun (as samples) at a 10 percent frequency (l out of 10 samples analyzed) to check for instrument drift. If drift exceeds predetermined limits (variable for instrument, element, and concentration level), the instrument is recalibrated prior to analysis of additional samples.

Usually five, and a minimum of three calibration standards (matrix matched), are used to establish calibration curves, bracketing the concentration range expected for the samples.

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Field instruments (electrical conductance, pH, and dissolved oxygen) are calibrated prior to each measurement following standard USGS procedures (Ward and Harr, 1990; Shelton 1994). Certified and dated conductance and pH standards are supplied by the USGS National Water Quality Laboratory. Dissolved oxygen meters are calibrated from the theoretical oxygen saturation of a humid atmosphere at a given temperature and pressure.

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11. Analytical Procedures

11.1 Concentration of dissolved major and trace elements:

The analytes are determined sequentially according to mass, on a single aliquot utilizing argon plasma ionization, mass spectrometric separation, and electron multiplier detection. Each analysis is based on the mean of three replicate determinations. Trace metals and major cations are analyzed using a Perkin Elmer-Sciex, Model 5000, modified inductively coupled plasma–mass spectrometer (ICP–MS). Major cations are analyzed using a Jarrell-Ash, Model 975, inductively coupled plasma–atomic emission spectrometer (ICP–AES). Calibration is performed using a reagent blank and three multi-element calibration standards. Linear regression analysis, through zero and based on 3 points, is employed to generate the calibration equation. All standards and samples are blank subtracted to insure correction for contamination as well as background correction. The methodology has been previously described (Taylor and others, 1990; Taylor and Garbarino, 1991; Garbarino and Taylor, 1995). Mercury is analyzed by cold vapor–atomic fluorescence spectroscopy using methods described by Roth (1994). Major anions will be analyzed using an ion chromatograph calibrated with at least 3 standards.

11.2 Redox speciation of iron:

Iron redox species will be determined with a colorimetric method using ferrozine as the color producing reagent (Stookey, 1970). For the determination of Fe(II), the 562-nm absorbance of the acidified and buffered sample will be measured with a Perkin Elmer Model Lambda 3b UV/VIS spectrophotometer equipped with 1-cm cells for the 10–40 μg range and 5-cm cells for the 2–10 μg range. For the determination of total Fe, a hydroxylamine hydrochloride reductant will be added. Ferric iron will be calculated from the difference between total Fe and Fe(II).

11.3 Sequential extractions of bed sediment and colloidal material:

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Three individual extractions will be done to each bed-sediment or colloidal concentrate sample, following the methods described by Hayes (1993). Hydroxylamine-HCl (0.25 M) will be used to dissolve the inorganic fraction of exchangeable metal ions in the sample. Potassium persulfate (0.17M) in 2 percent (volume/volume) sulfuric acid will be used to oxidize organic material. Dissolution of the silicates and other refractory material will be done in strong acids (hydrofluoric, nitric, and hydrochloric acids).

Each extraction will be analyzed by ICP–MS, ICP–AES, CV–AFS, and IC using methods described above for the analysis of major and trace elements in water samples.

11.4 Analysis of sulfur speciation and mineralogy in colloid concentrates and bed sediment:

The forms of sulfur in colloidal concentrates and bed sediment is determined from the chemical analysis of a series of extractions (Rice and others, 1993). An initial extraction with acetone is done to remove elemental sulfur. This extraction is followed by treatment with hot 6 *N* HC to dissolve acid-volatile sulfides (AVS, assumed to be monosulfides). The sulfide is purged from the sample with nitrogen gas and precipitated as silver sulfide. The hot-acid-treated sample is then filtered and soluble inorganic sulfates are determined by analyzing barium sulfate precipitated from the filtrate following the addition of barium chloride. The retentate is again extracted with acetone to recover AVS oxidized to sulfur during the acid treatment. Disulfide remaining in the retentate are determined following Cr^{2+} reduction/ distillation and precipitation as silver sulfide. Organically bound sulfur is determined from the residue of the disulfide sulfur determination by using an Eschka fusion to form SO_4^{2-} followed by precipitation as barium sulfate. Modifications to the above procedure, such as the addition of stannous chloride to the hot acid treatment, will be done to evaluate the effect of ferric iron (especially diagenetic ferric oxides) on the recovery of acid-volatile sulfides. To evaluate the significance of any recovery of pyritic sulfur as AVS, the analyses will be followed by determination of sulfur isotopy ($\delta^{34}S$-values) and mass-balance calculations.

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11.5 Mineralogy of colloidal material and bed sediment using X-ray diffraction

X-ray Diffraction (XRD) can provide a qualitative determination of the mineral phases present in the sediment and the suspended sediments. The data will be collected on a Scintag Pad V Automated Diffractometer, which is equipped with a scitillation detector and a graphite monochromator. Computer control of data collection and of data processing will enhance mineral identification and characterization. When concentration in a sample is less than approximately 5 percent or if the mineral is poorly crystalline, the phase in question often must be concentrated by physical differences (density, shape, size, or magnetic susceptibility) or by chemical means (chemical etching of specific phases usually called selective extractions). This equipment is available in the USGS California District laboratory. Emphasis will be placed on characterizing those mineral phases involved with metal transport.

11.6 Mineralogy using Mössbauer spectroscopy:

Information from ^{57}Fe Mössbauer spectroscopy will be combined with X-ray diffraction techniques to evaluate poorly crystallized minerals produced by the oxidation of sulfides. This has proven useful for identification of iron bearing minerals of small particle size and low concentration. A ^{57}Co source of λ-ray is used to generate the Mössbauer spectra which are characteristic of specific iron minerals.

11.7 Bonding of metals using EXAFS:

Extended X-ray Absorption Fine Structure (EXAFS) spectroscopy will be used to determine the local molecular structure about the average Fe ion in poorly crystallized ferrihydrite polymers. EXAFS spectra will be recorded in both transmission and fluorescence modes at the Stanford Synchrotron Radiation Laboratory using methods described by Waychunas and others (1993).

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11.8 Particle size distribution:

Particle size distributions of suspended sediment samples wet sieve and sedigraph methods. Particle size distributions of colloidal material will be determined using light scattering spectrometry.

11.9 Lead-isotope methods:

An appropriate weight of sediment sample or colloid material is digested in 2*M* HCl + H_2O_2 to provide a minimum amount of Pb for isotopic analysis (generally 50 ng or more). Digestions are done in FEP Teflon ware under laminar flow conditions using ultra-pure reagents, as described by Church and others (1993). Lead is separated in the bromide medium on Dowex 1x8 anion exchange using micro-columns. Analysis of the lead is performed on a multi-collector mass spectrometer; precision for the isotopic ratios is better than 1/1000. Analyses of blind duplicates, standards, and SRM 981 are used to monitor reproducibility and correct for isotopic fractionation, as described by Church and others (1993). Blanks are generally less than 1 ng for samples of this size. All analytical procedures and done under chain-of-custody protocol, under the direction of Dr. Stan Church, who is Quality Assurance officer for the lead isotope analyses.

12. Data Reduction, Validation, and Reporting

Data from field measurements will be entered in the USGS National Water Information Database (NWIS) after each field run. Laboratory data will be transmitted to the USGS project leader on laboratory analysis sheets from each participating laboratory and will include laboratory QA/QC data. Data from all laboratories will be entered into a single spreadsheet database maintained in the USGS Sacramento District office. QA/QC data will be reviewed as it is received and compared to the project Quality Assurance Objectives. Data not meeting objectives will be flagged and included in a report summarizing the activities and results of each sampling run.

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13. Quality Control Checks

The purposes of QC samples are to ensure that reported data represent actual values and that the data are reproducible and precise. The QC samples described below are used to evaluate field techniques, equipment, and laboratory methods.

13.1 Laboratory Analysis

Laboratory analysis of major and trace elements in water samples, colloid concentrates, and bed-sediment extracts will be done by the USGS lab in Boulder, Colorado (under the direction of Dr. Howard Taylor). All sample determinations will be performed in triplicate, with the mean value of the triplicate analysis being reported as the most probable value of concentration. Outliers are evaluated and rejected when statistically valid.

Standard Reference Materials (National Institute of Standards, USGS, and other sources) similar in composition (both matrix and analyte concentration) to the samples being analyzed, are processed and analyzed at a frequency of at least 10 percent of the samples determined. Control charts are maintained to assure that interference corrections (where necessary) or calibration procedures are within predetermined specifications.

Laboratory blanks are used to evaluate bias from deionized water and laboratory reagents used during the sample analysis.

When necessary and where appropriate, spike additions are made to samples, prior to analysis, to evaluate spike recovery. This is particularly important when preconcentration matrix separation, or speciation considerations are implemented.

All samples in a given batch are analyzed in random order to minimize errors associated with sample composition.

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Split samples for metals other than mercury will be sent periodically to both Dr. Howard Taylor's lab and the USGS National Water Quality Laboratory (NWQL) to evaluate any differences in the analytic results from the two labs. The NAWQA program uses the NWQL for its routine inorganic chemical analysis (Stanley and others, 1992), and comparison of the two labs is necessary to ensure comparability between data sets created by the two projects. Splits of mercury samples will be sent to the USGS lab in Madison, Wisconsin (under the direction of Dr. David Krabbenhoft). The Madison lab is also analyzing mercury for the Sacramento NAWQA project.

13.2 Field procedures

Equipment blanks will be done prior to or during the first field run to ensure that contaminants are not being introduced by any apparatus or procedure. During the sample collection phase of the project, field blanks will be processed through each stage of sample collection and processing to evaluate potential field contamination problems. Field blanks will be collected between samples at least once during each field run to check the efficacy of equipment cleaning procedures between sites. Additional field blanks will be collected at sites deemed critical to the successful completion of the study objectives.

Triplicate field samples will be collected at all locations at least once during the study to establish field sampling and processing variance. The triplicate samples will be treated as separate unknowns as they are submitted to the laboratories.

14. Performance and Systems Audits

All field personnel participate in the annual USGS National Field Quality Program. Reference samples for pH, specific conductance, and alkalinity are analyzed with the equipment used in field determinations. Satisfactory determinations are based on deviation from the most probable value

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for each coded reference sample: pH, ± 0.1 unit; specific conductance, ±4%, and alkalinity, ±1.5 standard deviations.

15. Preventative Maintenance

Field meters and other equipment are inspected and tested before field runs and worn or defective parts replaced. New batteries are installed at least annually or when meters have been inactive for an unknown length of time. Backup meters, spare parts (electrodes, membranes, filling solutions, etc.), and batteries are taken into the field for unscheduled replacement or repair.

16. Corrective Action

Corrective action will be taken before completion of any further field work if instrument malfunction is observed. Corrective action will also be taken whenever data are determined to be outside acceptable limits, as defined by the Quality Assurance Objectives.

Corrective action shall include reanalysis of samples after problems have been remedied. The method of standard additions may be used if spike recoveries are found to be outside acceptable limits. Any data not meeting Quality Assurance Objectives, but judged of potential usefulness, will be flagged and its deficiencies with regard to quality criteria described.

17. Quality Assurance Reports to Management

Following the review of field and laboratory results from each sampling run, the USGS project leader will compile a Quality Assurance Report summarizing the work completed, results of performance evaluations, results of data quality assessments, significant problems and recommended solutions. Summaries of this information will be included in quarterly project status reports.

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18. References

Ball, J.W., and Nordstrom, D.K., 1991, User's Manual for WATEQ4F, with revised thermodynamic data base and test cases for calculating speciation of major, trace, and redox. elements in natural waters: U.S. Geological Survey, Open-File Report 91-183, 189 p.

Capel, P.D., Nacionales, F.C., Larson, S.J., 1995, Precision of a splitting device for water samples: U.S. Geological Survey, Open-File Report 95-293, 6 p.

Chao, T.T., and Zhou, L., 1983, Extraction techniques for selective dissolution of amorphous iron oxides from soils and sediments: Soil Sci. Soc. Am. J., v. 47, p. 225–232.

Church, S.E., and others, 1993, Geochemical and lead-isotope data from stream and lake sediments, and cores from the Upper Arkansas River drainage: Effects of mining at Leadville Colorado on heavy-metal concentrations in the Arkansas River: U.S. Geological Survey, Open-File Report 93-534, 61 p.

Edwards, T.K., and Glysson, G.D., 1988, Field methods for measurement of fluvial sediment: U.S. Geological Survey Techniques of Water-Resources Investigations, Book 3, Chapter C2, U.S. Geological Survey, Open-File Report 86-531, 118 p.

Garbarino, J.R., and Taylor, H.E., 1995, Inductively coupled plasma-mass spectrometric method for the determination of dissolved trace elements in natural water: U.S. Geological Survey, Open-File Report 94-358, 88 p.

Hayes, H.C., 1993, Metal associations in suspended sediments and bed sediments from the Mississippi River: Golden, Colorado School of Mines, Department of Chemistry and Geochemistry, M.S. Thesis, 131 p.

Horowitz, A.J., Demas, C.R., Fitzgerald, K.K., Miller, T.L., and Rickert, D.A., 1994, U.S. Geological Survey protocol for the collection and processing of surface-water samples for the subsequent determination of inorganic constituents in filtered water: U.S. Geological Survey, Open-File Report 94-539, 59 p.

Murad, E., Schwertmann, U., Bigham, J.M., and Carlson, L., 1994, Mineralogical characteristics of poorly crystallized precipitates formed by oxidation of Fe^{2+} in acid sulfate waters; in Alpers, C.N., and Blowes, D.W., editors, Environmental Geochemistry of Sulfide Oxidation, Washington, D.C., American Chem. Soc., Symposium Series v. 550, p. 190–200.

Rantz, S.E., and others, 1982, Measurement and computation of streamflow: Volume 1. Measurement of stage and discharge and volume 2. Computation of discharge: U.S. Geological Survey, Water-Supply Paper 2175, 631 p.

Rice, C.A., Tuttle, M.L., and Reynolds, R.L., 1993, The analysis of forms of sulfur in ancient sediments and sedimentary rocks: comments and cautions: Chemical Geology, v. 107, p. 83–95.

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Roth, D.A., 1994, Ultratrace analysis of mercury and its distribution in some natural waters of the United States: Fort Collins, Colorado State University, Department of Chemistry, PhD. thesis, 309 p.

Shelton, L.R., 1994, Field guide for collecting and processing stream-water samples for the National Water Quality Assessment Program: U.S. Geological Survey, Open-File Report 94-455, 42 p.

Shelton, L.R., and Capel, P.D., 1994, Guidelines for collecting and processing samples of stream bed sediment for analysis of trace elements and organic constituents for the National Water Quality Assessment Program U.S. Geological Survey, Open-File Report 94-458, 20 p.

Stanley, D.L., Shampine, W.J., and Schroder, L.J., 1992, Summary of the U.S. Geological Survey National Field Water Quality Assurance Program from 1979 through 1989: U.S. Geological Survey, Open-File Report 92-163, 14 p.

Stookey, L.J., 1970, Ferrozine - A new spectrophotometric reagent for iron: Analytical Chemistry, v. 42, p. 779–781.

Stumm, W., and Morgan, J.J., 1981, Aquatic Chemistry: John Wiley & Sons, New York, 780 p.

Taylor, H.E., and Garbarino, J.R., 1991, The measurement of trace metals in water resource—monitoring samples by inductively coupled plasma-mass spectrometry: Spectrochimica Acta Rev., Vol. 14, Nos. 1-2, p. 33–43.

Taylor, H.E., Garbarino, J.R., and Brinton, T.I., 1990, The occurrence and distribution of trace metals in the Mississippi River and its tributaries: The Science of the Total Environment, Elsevier Science Publishers, p. 369–384.

Ward, J.R., Harr, C.A., editors, 1990, Methods for collection and processing of surface-water and bed-material samples for physical and chemical analyses: U.S. Geological Survey, Open-File Report 90-140, 71 p.

Waychunas, G.A., Rea, B.A., Fuller, C.C., and Davis, J.A., 1993, Surface chemistry of ferrihydrite: Part 1. EXAFS studies of the geometry of coprecipitated and adsorbed arsenate: Geochimica et Cosmochimica Acta, v. 57, p. 2251–2269.

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EPA REGION 8 QUALITY MANAGEMENT PLAN CROSSWALK

This crosswalk will be used to review the Quality Management Plans (QMPs) that are submitted to the Region 8 Quality Assurance Office for review under ANSI/ASQC E4-1994, *Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs* and EPA Order 2105-0. Items from this checklist are discussed in detail in EPA Manual CIO 2105-P-01-0 and in *EPA Requirements for Quality Management Plans (QA/R-2)*. Consult these resources for more information on the items below.

Note that all items below must be included in a QMP. If an item is not relevant (including those that are specific to EPA organizations) an explanation must be provided. Also note that process may either be described or referenced in the QMP; however, all references should be readily accessible within the organization and provided to the R8 QA Staff with the QMP.

Organization		**Applied to (e.g., contract number, IGMS number)**	
QMP Title		**Organization Contact and phone number**	
QMP Version and Date		**QMP Active Period**	
EPA Project Officer and phone number		**Date Received for Review**	
Other EPA contact and phone number		**Date Review Completed**	
QA Program Reviewer		**QMP Approval Date**	

Documents reviewed: **QMP/date/cover period** *(Yes/No/Not Provided)* **Work Statement (PPA, SOW, e.g.)** *(Yes/No/Not Provided)* **QARF (contracts only)** *(Yes/No/Not Provided)* **Dollar amount** *(minimum, total value & duration)*	
Is QMP consistent with the work statement (current/next year) (Yes/No)	
Summary of Comments: **Note:** In addition to addressing concerns in the Summary of Comments, the Grantee/Contractor must also respond to the issues identified in the Comment section(s) that includes a "**Response (date)**" and "**Resolved (date)**."	

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Element	Page(s)	Acceptable (Yes/No/NA)	Comments
MANAGEMENT AND ORGANIZATION			
1. Signed and dated by senior manager?			
2. Signed and dated by senior line management?			
3. Signed and dated by organization's QA manager?			
4. Includes signature lines for : • R8 Project Officer, and			
• R8 QA Manager's approval?			
5. Includes signature line for EPA Grant Award Official approval (if applicable)?			
6. Includes statement of the organization's QA policy?			
a. QA policy statement includes general objectives/goals?			
b. QA policy statement includes allocation of intramural, extramural, and travel funds and personnel?			
7. Includes organizational chart?			
a. Organizational chart identifies all components of organization?			
b. Organizational Chart identifies position of QA manager?			
c. Organizational Chart identifies lines of reporting of the QA manager?			
d. Organization Chart identifies any other QA staff?			

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Element	Page(s)	Acceptable (Yes/No/NA)	Comments
8. Includes discussion of authorities of the QA manager and staff?			
9. Documents the independence of QA manager?			
10. Describes procedures to ensure QA staffs have access to appropriate levels of management?			
11. Discusses technical activities or programs that require quality management?			
12. Discusses where oversight of delegated or extramural programs is needed?			
13. Identifies where internal coordination of QA and QC activities among organizations is needed?			
14. Discusses how management assures understanding and implementation in all programs?			
15. Describes process for resolving disputes?			
QUALITY SYSTEM COMPONENTS			
16. Includes description of quality system?			
17. Describes principal quality system components (e.g., quality system documentation, annual reviews and planning, project-specific quality documentation? (Note, identify components in Column 3.)			
18. Description of components includes how they are implemented?			

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Element	Page(s)	Acceptable (Yes/No/NA)	Comments
19. Description of components includes responsibilities of management and staff?			
20. Lists tools for implementing each component (e.g., QMPs, Quality Systems Audits, Training Plans, QA Project Plans? (Note: list tools in Column 3.)			
21. Identifies internal organizations that develop QMPs?			
22. Identifies review and approval procedures for these internal QMPs?			
23. Includes assurance that QA responsibility is incorporated into performance standards (consistent with Agency personnel policy)?			
QUALIFICATIONS AND TRAINING			
24. States policy regarding QA training for management and staff?			
25. Describes process for identifying, ensuring, and documenting that personnel have necessary quality-related qualifications?			
26. Describes process for ensuring personnel maintain quality-related qualifications?			
27. Describes process for identifying the need for quality-related retraining based on changing requirements?			
28. Includes roles, responsibilities, and authorities in description of above processes?			
PROCUREMENT OF ITEMS AND SERVICES			

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Element	Page(s)	Acceptable (Yes/No/NA)	Comments
29. Describes process for reviewing and approving all extramural agreements (grants, cooperative agreements and contracts)?			
a. Review process ensures documents are complete and accurate?			
b. Review process ensures agreement clearly describes the item or service needed?			
c. Review process ensures agreement describes the associated technical and quality requirements?			
d. Review process ensures agreement describes the quality system elements for which the supplier is responsible?			
e. Review process ensures that the supplier's conformance to the customer's requirements will be verified?			
30. Describes process for reviewing and approving applicable responses to solicitations to ensure that they satisfy all technical and quality requirements?			
a. Review process ensures the review of evidence of the supplier's capability to satisfy EPA quality requirements?			
b. Review process ensures procured items and services are acceptable?			
31. Describes process for review and approval of suppliers' quality-related documentation (e.g., QA Project Plans and QMPs)?			
32. Includes discussion of any policy and criteria for delegations of review of QA Project Plans and QMPs?			

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Element	Page(s)	Acceptable (Yes/No/NA)	Comments
33. Describes process to ensure EPA extramural agreement policies satisfied?			
34. Includes roles, responsibilities, and authorities in description of above processes?			
DOCUMENTS AND RECORDS			
35. Describes process for identifying quality-related documents and records (including electronic) requiring control?			
36. Describes process for preparing, reviewing, approving, issuing, using, authenticating, and revising documents and records?			
37. Describes process for ensuring that records and documents accurately reflect completed work?			
38. Describes process for maintaining documents and records including transmittal, distribution, retention, access, preservation, traceability, retrieval, removal of obsolete documentation, and disposition?			
39. Describes process for establishing and implementing appropriate chain of custody and confidentiality procedures for evidentiary records?			
40. DELETED			
41. Includes roles, responsibilities, and authorities in description of above processes?			
COMPUTER HARDWARE AND SOFTWARE			

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Element	Page(s)	Acceptable (Yes/No/NA)	Comments
42. Describes process for developing, installing, testing, using, maintaining, controlling, and documenting computer hardware and software?			
43. Describes process for assessing and documenting the impact of changes to user requirements and/or the hardware and software on performance?			
44. Describes process for evaluating purchased hardware and software?			
45. Describes process for ensuring that data and information produced from or collected by computers meet applicable requirements and standards?			
46. Includes roles, responsibilities, and authorities in description of above processes?			
47. DELETED			
PLANNING			
48. Includes a description of the systematic planning process for environmental data operations?			
a. Does process include identification and involvement of all customers and suppliers?			
b. Does process include description of the project goal, objectives, and questions and issues to be addressed?			
c. Does process include identification of project schedule, resources, milestones, and any applicable requirements?			

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Element	Page(s)	Acceptable (Yes/No/NA)	Comments
d. Does process include identification of the type and quantity of data needed and how the data will be used to support the project's objectives?			
e. Does process include specification of performance criteria for measuring quality?			
f. Does process include specification of needed QA and QC activities to assess the quality performance criteria?			
g. Does process include description of how, when, and where the data will be obtained (including existing data) and identification of any constraints on data collection?			
h. Does process include description of how the acquired data will be analyzed, evaluated, and assessed against its intended use and the quality performance criteria?			
49. Describes process for developing, reviewing, approving, implementing, and revising QA Project Plans?			
50. Describes process for evaluating and qualifying data collected for other purposes or from other sources?			
51. Includes roles, responsibilities, and authorities in description of above processes.			
IMPLEMENTATION OF WORK PROCESSES			
52. Describes process for ensuring that work is performed according to planning and technical documents?			
53. Describes process for identifying operations needing procedures?			

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Element	Page(s)	Acceptable (Yes/No/NA)	Comments
54. Describes process for preparation, review, approval, revision, and withdrawal of these procedures?			
55. Describes policy for use of these procedures?			
56. Describes process for controlling and documenting the release, change, and use of planned procedures?			
a. Process includes description of necessary approvals?			
b. Process includes removal of obsolete documentation from work areas?			
c. Process includes verification that the changes are made as prescribed?			
57. Includes roles, responsibilities, and authorities in description of above process?			
ASSESSMENT AND RESPONSE			
58. Describes the process for assessing the adequacy of the quality system at least annually?			
59. Describes the process for planning, implementing and documenting assessments and reporting results to management?			
a. Process includes selecting an assessment tool, the expected frequency, and the roles and responsibilities of assessors?			
b. Process includes determining the level of competence, experience and training needed for assessment personnel?			

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Element	Page(s)	Acceptable (Yes/No/NA)	Comments
c. Process includes ensuring that personnel have no real or perceived conflict of interest, and have no direct involvement or responsibility for the work being assessed?			
d. Process includes ensuring that personnel conducting assessments have sufficient authority, access to programs and managers, access to documents and records, and organizational freedom?			
60. Describes process for management's review of, and response to, findings?			
61. Describes process for identifying how and when corrective actions are to be taken in response to the findings of the assessment?			
a. Process includes ensuring corrective actions are made promptly?			
b. Process includes confirming the implementation and effectiveness of any corrective action?			
c. Process includes documenting actions?			
62. Describes process for addressing disputes encountered as a result of assessments?			
63. Includes roles, responsibilities, and authorities in description of above processes?			
QUALITY IMPROVEMENT			
64. Describes process for ensuring that conditions adverse to quality are prevented, identified promptly, corrected promptly and that actions are taken toward prevention, documented and actions tracked to closure?			

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Element	Page(s)	Acceptable (Yes/No/NA)	Comments
65. Describes process for encouraging staff to establish communications between customers and suppliers, identify process improvement opportunities, and identify and propose solutions for problems?			
66. Includes roles, responsibilities, and authorities in description of above processes?			
OTHER REVIEW CRITERIA			
67. Are regulatory or other citations accurate?			
68. Are organizational units identified consistent with the most recent reorganization?			
69. Are past QA management assessment (MSR/QSA/other) findings resolved? (Put date of Final Report in Column 3.)			
70. Are activities described in the QMP consistent with Work Plans?			
71. Are tasks proposed for other organizations not covered solely by this QMP documented elsewhere (e.g., in another organization's QMP)?			

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APPENDIX 5—CASE STUDIES OF SAMPLING AND MONITORING

Compiled by Kathleen S. Smith, Virginia T. McLemore, and Carol C. Russell

Reviewed by Thomas Moyer and David Rathke

Series Title: Management Technologies for Metal Mining Influenced Water

Volume 6: Sampling and Monitoring for the Mine Life Cycle

Editors: Virginia T. McLemore, Kathleen S. Smith, and Carol C. Russell

Publisher: Society for Mining, Metallurgy & Exploration Inc. Englewood, Colorado

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MULTIDISCIPLINARY CHARACTERIZATION OF HISTORICALLY MINED WATERSHEDS

Animas River Watershed Study Area, Colorado

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Purpose of case study: The Animas River watershed, Colorado, had been extensively altered and heavily mined for more than a century. Could we identify the different sources of metals and acidity? What remedial work could be done to improve water quality?

Brief site description and history: The study area covered about 150 mi^2 of the upper Animas River watershed. The watershed is the site of the large Silverton caldera, an Oligocene volcanic edifice that was extensively altered and mineralized (Bove et al. 2007b; Dalton et al. 2007; Mast et al. 2007; Yager and Bove 2007). Mining of base- and precious-metal vein deposits began in 1871 (Jones 2007). Early production was processed first by hand-trimming of silver-bearing galena ore and shipment by pack animals to smelters. Because there was no market and no smelting process developed for the recovery of zinc, sphalerite was discarded as mine waste or left in the mine. Shortly thereafter, small stamp mills were installed at many of the mines and several ill-fated attempts were made to smelt the ore in Silverton. With the arrival of the railroad in 1882, ore concentrate was increasingly shipped to the ASARCO smelter in Durango (Jones 2007; Sloan and Skowronski 1975). Sphalerite was first recovered and a zinc concentrate shipped by rail for smelting in 1915 (Jones 2007). Major Pb-Zn-Ag, and subsequently Au production continued until 1991 when the Sunnyside mine ceased operation. Mine and mill tailings were dumped into lakes and surface streams resulting in widespread environmental consequences. Mine and mill tailings were largely impounded by 1935 (Jones 2007), but tailing deposits that predate that timeline, as well as accidental releases of tailings into the Animas River, resulted in a heavily contaminated river bed (Church et al. 2007a; Vincent and Elliott 2007). A total of 373 mines, many small stamp mills, and a few larger mills produced an estimated 18.1 million short tons of mine waste, which were disposed of largely on the adjacent land or in surface streams and later in unlined impoundments in the watershed (Church et al. 2007b; Jones 2007). Historical accounts indicated that a cold-water fishery did not exist in much of the basin at the time the early miners arrived. Fish were present downstream of the confluence of the three major tributaries, including Mineral and Cement Creeks, and the Animas River (Besser et al. 2007). Water quality rapidly deteriorated shortly after the introduction of major mining activities in the 1890s following the arrival of the railroad. As a direct result of this historical mining, the town of Durango (50 mi downstream) was forced to cease using the Animas River as a source of domestic water by the early 1900s (Jones 2007). In addition, seepage from these unlined tailings repositories and from early mine-rock waste deposited in the riparian habitat along the creek and river courses resulted in large, non-point sources of metals and acidity (Kimball et al. 2007; Walton-Day et al. 2007). Many mine adits were draining highly acidic (pH <1), metal-laden water (Kimball et al. 2007; Nash and Fey 2007; Wright et al. 2007b). Acidic mine pools also appeared to contribute to some groundwater sources (Church et al. 2007b; Wright et al. 2007a, b).

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Problem definition: The watershed had been extensively mined for about a century—hundreds of abandoned mine sites and thousands of adits were present in the watershed. Water quality in Mineral and Cement Creeks and the upper reaches of the Animas River did not support aquatic life (Besser and Leib 2007). A small trout population existed in parts of the Animas River, in South Fork Mineral Creek, and in some tributary creeks upstream of the town of Silverton where mining was not extensive and where carbonate rocks buffered water quality (Besser et al. 2007; Bove et al. 2007a).

Define questions and objectives: With the closure of the last mine, the local economy changed from a mining to a tourism-based economy. Could a trout fishery be restored to other parts of the watershed to enhance tourism? What was the original water quality in the watershed? This study was conducted to determine to what extent water quality degradation was the result of historical mining and to what extent it was caused by weathering of hydrothermally altered rock. What studies best delineate the sources of metals that should or could be remediated? How much of the acidity and metals currently present were released as the result of historical mining? What is the practical limit of restoration and what are realistic expectations for recovery?

Conceptual model: Based on the site history (Jones 2007), we hypothesized that large mine-waste dumps near water courses had the highest probability of being metal sources (Nash and Fey 2007). Adit drainage also had a high probability of being a source of metals and acidity (Kimball et al. 2007; Wright et al. 2007a, b). Some mines were sited along or in contact with large geologic structures (fractures and faults) that may serve as fluid-flow paths (Bove et al. 2007b; Yager and Bove 2007). Weathering of hydrothermally altered rock, particularly the acid-sulfate and the quartz-pyrite-sericite alteration that was extensive in the watershed (Bove et al. 2007 a, b; Dalton et al. 2007; Mast et al. 2007; Wright et al. 2007b), was also anticipated to be a source of metals and acidity. Widespread distribution of ferricrete in stream banks indicated that acidic groundwater drainage derived from hydrothermally altered areas of the Animas River watershed predated historical mining activity and substantially influenced stream water chemistry (Verplanck et al. 2007; Yager et al. 2003).

Data, information, and risk evaluation: An initial water-quality study was conducted by the Colorado Department of Public Health and Environment (CDPHE), but the geologic setting was so complex that many questioned the applicability of these data to the problem because causes and effects were not addressed. The USGS was asked to do an integrated, multidisciplinary study of the site as a part of the Abandoned Mine Lands Initiative (AMLI; http://amli.usgs.gov, accessed 9/27/13). Many stakeholders were not willing to allocate responsibility for the metals and acidity contamination without evaluation of the natural geologic component. Scientific data were needed by the Federal and State agencies before proceeding with remediation work. The assessment of risk was multifold. Besser and Braumbaugh (2007) used chemical data in liver tissue to assess the toxicity of dissolved metals to trout. They also evaluated the toxicity of metals in the food chain to show that metals ingested by trout were the source of toxicity. Furthermore, metal concentrations increased with higher tropic levels. Copper was primarily the metal that limited trout populations. Besser and Leib (2007) used field collected water samples to test toxicity in the laboratory. Stream water was found to be less toxic than metals in sediment, and the toxicity of stream water was reduced in the presence of sediment, presumably due to sorption of dissolved metals. Models of stream toxicity were developed using year-round water

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quality data from stream gauges on the three major tributaries, Mineral Creek, Cement Creek, and the Animas River at Silverton to show when dissolved metals concentrations exceeded toxicity limits to trout species (Leib et al. 2007). Toxicity of stream water was associated with elevated concentrations of zinc and copper. Laboratory tests indicated that dissolved zinc was highly toxic to amphipods, less toxic to fathead minnows, and least toxic to brook trout. Besser et al (2007) used hazard-quotient methods to assess the toxicity of both individual dissolved metals and metals in sediment as well as the combined metal toxicity in both media to assess risk to trout and aquatic invertebrates in individual stream reaches where the pH was greater than 4.5. Aluminum toxicity was high in mixing zones where increase in pH resulted in precipitation of aluminum hydroxide that coated the gills of aquatic species causing death by suffocation. Finally, toxicity risk was assessed for cadmium, copper, and zinc on the basis of synoptic sampling made during periods of low flow and modeling of seasonal variations in dissolved metals concentrations by Leib et al (2007). Anderson (2007) conducted detailed studies of macroinvertebrates at various sites in the watershed to demonstrate the effects of historical mining on the food web and to establish sites for future monitoring of macroinvetebrate population changes to assess watershed recovery.

Costs and consequences of not sampling and monitoring: The threat of listing the Animas River watershed as a CERCLA (Superfund) site by the USEPA was imminent. Large expenditures of funds were proposed by the local stakeholders group for engineering solutions to remove point sources of contamination even though the scope and extent of metal and acidity contamination were not fully understood. The mining company that had closed the last mine in the area wanted to be released from their remediation bond so that they could exit the site without the threat of further liability.

Data needs: We characterized the watershed using water and sediment geochemistry (Church et al. 2007b; Kimball et al. 2007; Mast et al. 2007;Wright et al. 2007b), and geologic mapping (Dalton et al. 2007; Yager and Bove 2007a), including detailed studies of the type and extent of hydrothermal alteration (Bove et al. 2007b), mine-waste site characterization (Nash and Fey 2007), groundwater flow paths, and discharge (Kimball et al. 2007; Wright et al. 2007a, b). Sediment samples analyzed for metals (Al, As, Cd, Co, Cu, Fe, Mn, Ni, Pb, and Zn) provided data on the long-term loads of metals and their variation by season, by flow rate, and their variation from year to year (Church et al. 2007a). Sampling of surface water quality during summer low-flow (more than 170 sites) and high-flow (about 120 sites) conditions provided a baseline for water quality during the spring and summer (Wright et al. 2007a). Water quality was also assessed during winter low-flow (about 20 sites) conditions isolated groundwater contributions (winter sampling provides results from groundwater sources alone; Wright et al. 2007a). Many in-stream tracers were run on most segments of the highly impacted stream reaches to identify both discrete and non-point sources of metals and acidity (Kimball et al. 2007; Walton-Day et al. 2007). Model simulations of these data provided valuable insight to assess the effects of specific proposed removal actions. Detailed studies of water quality from various types of hydrothermal alteration resulted in the identification of most problematic natural sources of metals (Bove et al. 2007a). Detailed evaluations of the chemistry of mine wastes provided insight into proposed cover or removal actions (Fey et al. 2011). Discrete sources of contamination could be evaluated for potential removal or *in situ* mitigation whereas non-point sources were considered to be the result of weathering of mineralized rock. Macroinvertebrate

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and fish population studies (Besser et al. 2007; Besser and Brumbaugh 2007) and toxicological studies (Besser and Leib 2007) were conducted in stream reaches where aquatic life could survive, and modeling studies indicated the seasonal variations where various trout species could survive during parts of the year (Leib et al. 2007).

Brief sampling and monitoring plan: An extensive water-quality monitoring plan was in place and monitored by CDPHE as mandated by the terms of the mining operation permit (Church et al. 2007c). Additional oversight was conducted by the stakeholder`s group in cooperation with Federal agencies and the USEPA to verify the water quality data reported by the mining company. Multiple blind water quality and sediment samples were collected during USGS studies to verify both our own data as well as to verify those analyzed by other Federal and State agencies and stakeholders.

Pilot study: The initial study of water and sediment conditions, funded by downstream water users, evaluated data from about 35 sites selected throughout the watershed to assess the scope of the problem (Church et al. 1997). A much more detailed scope of study was developed after the USGS initiated the extensive environmental studies undertaken under the USGS AMLI (http://amli.usgs.gov, accessed 9/27/13).

Analyze and manage data: Extensive database in Access and GIS (Sole et al. 2007; available online at http://pubs.usgs.gov/pp/1651/downloads, accessed 9/27/13).

Involvement of stakeholders: Members of the community and Federal agency representatives met monthly at stakeholder meetings to keep all interested parties informed of their activities. As USGS results became available, USGS personnel would travel to these meetings and present the results and coordinate with the local stakeholders' group. Both the U.S. Bureau of Land Management (USBLM) and U.S. Forest Service (USFS) personnel would attend the stakeholders meeting each month and provide feedback on stakeholder needs as well as their reactions to new data.

Decisions made: Pre-mining conditions were estimated from a study of pre-mining sediment collected from old stream terraces dated using both carbon-14 (^{14}C) and dendrochronology of trees rooted in the pre-mining sediment (Church et al. 2007a; Vincent et al. 2007). These data were compared with current conditions in both mined and unmined but hydrothermally altered watersheds to show how much historical mining had changed the aquatic habitat (Mast et al. 2007; Wright et al. 2007b) and to provide a pre-mining sediment baseline against which remediation could be evaluated (Anderson 2007; Church et al. 2007c; Finger et al. 2007; Walton-Day et al. 2007).

Conclusions: Remediation in the watershed would be a long-term, incremental process. By 2007, more than $20,000,000 had been spent on remediation, largely on removal of mill tailings deposited in the riparian zone of the Animas River either prior to 1935 or from a massive failure in the tailings wall of one of the tailings repository at the Mayflower mill site. Some progress in improved water quality has been documented at the watershed scale (Church et al. 2007c). Much, but not all, of the metal and acidity loads currently present in the stream reaches were the result of historical mining activities (Bove et al. 2007a, b Church et al. 2007b; Nash and Fey

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2007; Walton-Day et al. 2007; Wright et al. 2007a). We recommended that remediation funds spent in the future should focus on achievable goals where technology has been proven (Finger et al. 2007; Runkel and Kimball 2002). Waiting for natural erosional processes to remove tailings from the Animas River would require centuries of time before there would be any meaningful recovery of aquatic life (Vincent and Elliott 2007).

Were the data quality objectives met? Yes

Costs and benefits: This project was intended to evaluate geological and engineering studies that cross disciplinary boundaries in a cooperative effort to provide a holistic snapshot of the current aquatic environment in the watershed (Church et al. 2007d). Different methods and disciplines of studies provided different types of information, some of which were more beneficial to the remediation efforts than others (see Church et al. 2007c; Finger et al. 2007). Coupled sediment and water-quality data provided both time-integrated and instantaneous data to evaluate metal sources (Church et al. 2007a; Wright et al. 2007a). In-stream tracer studies (using lithium bromide–LiBr; Kimball et al. 2007) and solute-modeling studies of these data (Walton-Day et al. 2007) allowed evaluation of remedial and engineering objectives and their best-case scenario results (Church et al. 2007c; Runkel and Kimball 2002).

Lessons learned:

1. Hydrothermal alteration is a natural source of metals and acidity—not all elevated concentrations of metals and low-pH conditions found in streams are the result of historical mining (Bove et al 2007 a, b; Mast et al. 2007; Wright et al. 2007a).
2. Geologic setting influences the remedial remedies that will be effective. Buffering by carbonate present in the rocks in the study area affects aquatic habitat (Bove et al. 2007a, b; Mast et al. 2007).
3. In watersheds where the macroinvertebrate populations are severely impaired or absent, water-quality and sediment data can be used in conjunction with modeling to predict when peaks of acidity and metals in streams impair aquatic life (Besser and Brumbaugh 2007; Besser and Leib 2007).
4. Water chemistry is a transient indicator of metal toxicity whereas sediment data provide a time-integrated measure of the environment. During the spring, colloidal sediment that accumulated during low-flow conditions is flushed out of the stream (Fey et al. 2002). Both water and sediment data sets are needed to evaluate the aquatic environment. Changes in flow rate between years will result in sediment data that reflect the amount of flushing of colloidal sediment-bound metals out of the streambed at differing rates (Church et al. 2007a).
5. Understanding stream geomorphology is important in evaluating the distribution, removal, and long-term effects of remedial actions (Vincent and Elliott 2007).

Recommendations: Careful evaluation of the geologic setting, hydrothermal alteration, and mining history is recommended before developing engineering solutions to remediate watersheds impacted by historical mining (Church et al. 2007a, c).

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Bibliography:

Anderson, C.R., 2007, Effects of mining on benthic macroinvertebrate communities and monitoring strategy, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 851-872. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Besser, J.M., and Brumbaugh, W.G., 2007, Status of stream biotic communities in relation to metal exposure, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 823-835. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Besser, J.M., and Leib, K.J., 2007, Toxicity of metals in water and sediment to aquatic biota, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 837-849. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Besser, J.M., Finger, S.E., and Church, S.E., 2007, Impacts of historical mining on aquatic ecosystems—An ecological risk assessment, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 87-106. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Bove, D.J., Mast, M.A., Dalton, J.B., Wright, W.G., and Yager, D.B., 2007a, Major styles of mineralization and hydrothermal alteration and related solid- and aqueous-phase geochemical signatures, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 161-230. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Bove, Dana J.; Yager, Douglas B.; Mast, M. Alisa; Dalton, J. Brad, 2007b, Alteration map showing major faults and veins and associated water-quality signatures of the Animas River watershed headwaters near Silverton, southwest Colorado: U.S. Geological Survey Scientific Investigations Map 2976, scale 1:24,000. http://pubs.usgs.gov/sim/2976/, accessed 9/27/13.

Church, S.E., Kimball, B.A., Fey, D.L., Ferderer, D.A., Yager, T.J., and Vaughn, R.B., 1997, Source, transport and partitioning of metals between water, colloids, and bed sediments of the Animas River, Colorado: U.S. Geological Survey Open-File Report 97-151, 135 p. http://pubs.er.usgs.gov/publication/ofr97151, accessed 9/27/13.

Church, S.E., Fey, D.L., and Unruh, D.M, 2007a, Trace elements and lead isotopes in modern streambed and terrace sediment—Determination of current and premining geochemical baselines, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 571-642. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Church, S.E., Mast, M.A., Martin, E.P., and Rich, C.L., 2007b, Mine inventory and compilation of mine-adit chemistry data, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas

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River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 255-310. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Church, S.E., Owen, J.R., von Guerard, Paul, Verplanck, P.L., Kimball, B.A., and Yager, D.B., 2007c, The effects of acidic mine drainage from historical mines in the Animas River watershed, San Juan County, Colorado—What is being done and what can be done to improve water quality? *in* J.V. DeGraff, ed., Geological Society of America Reviews in Engineering Geology, v. XVII, p. 47-83.

Church, S.E., von Guerard, Paul, and Finger, S.E., eds., 2007d, Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, 6 plates, 1 DVD, 1,096 p. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Dalton, J.B., Bove, D.J., Mladinich, C.S., and Rockwell, B.W., 2007, Imaging spectroscopy applied to the Animas River watershed and Silverton caldera, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 141-159. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Fey, D.L., Wirt, L.; Besser, J.M.; Wright, W.G., 2002, Water quality and aquatic toxicity data of 2002 spring thaw conditions in the upper Animas River watershed, Silverton, Colorado: U.S. Geological Survey Open-File Report 2002-488, 31 p.

Fey, D.L., Church, S.E., Driscol, R.L., and Adams, M.G., 2011, Multiple applications of the USEPA 1312 leach procedure to mine waste from the Animas River watershed, southwest Colorado: Geochemistry: Exploration, Environment, Analysis, v. 11, p. 163-178.

Finger, S.E., Church, S.E., and von Guerard, P., 2007, Potential for successful ecological restoration, remediation, and monitoring, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 1065-1078. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Jones, W.R., 2007, History of mining and milling practices and production in San Juan County, Colorado, 1871–1991, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 39-86. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Kimball, B.A., Walton-Day, K., and Runkel, R.L., 2007, Quantification of metal loading by tracer injection and synoptic sampling, 1996–2000, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 417-495. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Leib, K.J., Mast, M.A., and Wright, W.G., 2007, Characterization of mainstem streams using water-quality profiles, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 543-570. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

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Mast, M.A., Verplanck, P.L., Wright, W.G., and Bove, D.J., 2007, Characterization of background water quality, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 347-386. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Nash, J.T., and Fey, D.L., 2007, Mine adits, mine-waste dumps, and mill tailings as sources of contamination, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 311-345. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Runkel, R.L., and Kimball, B.A., 2002, Evaluating remedial alternatives for an acid mine drainage stream—Application of a reactive transport model: Environmental Science and Technology, v. 36, p. 1093-1101.

Sloan, R.E., and Skowronski, C.A., 1975, The Rainbow Route, an illustrated history of the Silverton Railroad, the Northern Silverton Railroad, and the Silverton, Gladstone, & Northerly Railroad: Denver, Colo., Sundance Publications Limited, 416 p.

Sole, T.C., Granitto, M., Rich, C.L., Litke, D.W., and Pelltier, R.T., 2007, Digital databases and CD-ROM for the Animas River watershed, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 1079-1096. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Verplanck, P.L., Yager, D.B., Church, S.E., and Stanton, M.R., 2007, Ferricrete classification, morphology, distribution, and 14C age constraints, in Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 721-744. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Vincent, K.R., Church, S.E., and Wirt, L., 2007, Geomorphology of Cement Creek and its relation to ferricrete deposits, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, pp. 745-773.

Vincent, K.R., and Elliott, J.G., 2007, Response of the upper Animas River downstream from Eureka to discharge of mill tailings, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 889-941. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Walton-Day, K., Paschke, S.S., Runkel, R.L. and Kimball, B.A., 2007, Using the OTIS solute-transport model to evaluate remediation scenarios in Cement Creek and the upper Animas River, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 973-1028. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Wright, W.G., Kimball, B.A., and Runkel, R.L., 2007a, Effects of the May Day mine site on stream-water quality in the Cement Creek basin, August 2000, *in* Church, S.E., von

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Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 943-972. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Wright, W.G., Simon, W., Bove, D.J., Mast, M.A., and Leib, K.J., 2007b, Distribution of pH values and dissolved trace-metal concentrations in streams, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 497-541. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Yager, D.B., and Bove, D.J., 2007, Geologic framework, *in* Church, S.E., von Guerard, Paul, and Finger, S.E., eds., Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: U.S. Geological Survey Professional Paper 1651, p. 107-140. http://pubs.usgs.gov/pp/1651/, accessed 9/27/13.

Yager, D.B., Church, S.E., Verplanck, P.L., and Wirt, Laurie, 2003, Ferricrete, manganocrete, and bog iron occurrences with selected sedge bogs and active iron bogs and springs in the upper Animas River watershed, San Juan County, Colorado: U.S. Geological Survey, Miscellaneous Field Studies Map, MF-2406.

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Boulder River Watershed Study Area, Montana

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Purpose of case study: The Boulder River watershed study area, Montana, contained some unique glacial geomorphic features and geologic settings that contributed to the environmental impact of historical mining (Cannon et al. 2004; O'Neill et al. 2004). Base metals were released from historical mill workings that were saturated by rising water during spring runoff. As a result, contaminated stream water and sediment were released from the impounded mill site when the ice dam broke and impaired fish habitat in upper Basin Creek.

Brief site description and history: A portion of the Boulder River watershed consisting of three tributary drainages to the Boulder River, approximately 46 mi^2 in area, is located in a glaciated terrain with large broad valleys in Montana. The Boulder batholith contains a fractured aquifer, which is essentially dry below 100-ft depth. It is overlain by Eocene gravel, which is the surface aquifer, although this upper aquifer is not uniformly present throughout the watershed (McDougal et al. 2004; O'Neill et al. 2004). Small-scale underground mining began to exploit base- and precious-metal, polymetallic vein deposits hosted in the Boulder batholith about a century ago. Hydrothermal alteration, largely propylitic, is localized to a few meters adjacent to the mineralized veins and is not a source of meals or acidity. Mining of the polymetallic vein deposits ceased about 1950 (Nimick et al. 2004).

Problem definition: Tributaries to the Boulder River were contaminated by Pb, Zn, and Cu downstream from the mines (Church et al. 2004; Nimick and Cleasby 2004). Upstream, some of these tributaries had an excellent native trout fishery (Finger et al. 2004). Some of the lower reaches of the tributaries supported brown and rainbow trout. No trout were present downstream of major adit drainages. Downstream of some of these tributaries trout in the Boulder River were impaired by introduction of high metal concentrations (Farag et al. 2004, 2007). Tailings were present in some of the riparian zones adjacent to the streams (USGS 2004). During spring runoff the valley in the upper reach of Basin Creek was blocked by ice, and tailings deposits in the drainage were flooded (Cannon et al. 2004). This resulted in seasonal control of the metal mobility into Basin Creek. Water quality below mine sites in the area is often good. Mines located along the major structural features have adit drainages with pH values about 3.5 (Nimick and Cleasby 2004). These adit drainages saturated mill tailings downstream, and they constituted large metal reservoirs. One small open-pit mine along High Ore Creek had deposited mine tailings in the stream channel (Gelinas and Tupling 2004). Trout were absent in this tributary.

Define questions and objectives: Determine where the major metal sources are in the watershed, assess how they impact trout survival and growth, and advise Federal land managers on what remedial actions might be used to mitigate the environmental impact.

Conceptual model: Based on the site history, we hypothesized that large mine-waste dumps near water courses had the highest probability of being metal sources. Adit drainage flows also had a high probability of being metal sources and were related to large geologic structures (fractures and faults; McDougal et al. 2004; O'Neill et al 2004). We characterized the watershed using both water and sediment geochemistry (Church et al. 2004; Nimick and Cleasby 2004)

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followed by 96-hour *in situ* toxicity studies using trout fingerlings (Farag et al. 2004, 2007). Because the water quality was generally good, we were evaluating chronic toxicity effects.

Data, information, and risk evaluation: The Montana Department of Environmental Quality (MDEQ) had completed some initial water-quality measurements and had completed a characterization study of mine-waste piles (Metesh et al. 1994, 1995). The U.S. Bureau of Mines (USBM) and the USFS had also done some evaluation of the effects of mine wastes on water quality. The USGS was asked to do an integrated, multidisciplinary study of the site as a part of the AMLI (http://amli.usgs.gov, accessed 9/27/13). Remediation work was needed to restore sport fisheries, which was one major economic driver in the local economy. Federal and State agencies needed a complete understanding of the major factors causing contamination before proceeding with remedial action. Biologic tissues were collected to evaluate the toxicological effects of metals on aquatic life (Farag et al 2004, 2007).

Costs and consequences of not sampling and monitoring: Continued impairment of biological communities and the loss of tourist dollars because of poor quality of trout fisheries resulted in a poor local economy.

Data needs: Fifty to sixty samples of bottom sediment (wet sieved to <2 mm on site) and water (filtered and unfiltered) were collected and analyzed for complete water chemistry (Lambing et al. 2004; Nimick and Cleasby 2004). Leaching studies of the sediment samples were conducted to determine metal concentrations carried in the iron-hydroxide phases (e.g., colloidal phase; Church et al. 2004). In-stream tracer studies (lithium bromide–LiBr) were conducted in selected reaches of the tributaries to isolate contributions from major sources and to test for groundwater influx (Kimball et al. 2004). Selected sites were sampled for biofilm, macroinvertebrates, and trout. The biofilm samples were analyzed for metals. The macroinvertebrates were analyzed for species diversity. The trout tissues were analyzed for metallothionein to determine the stress on internal organs (Farag et al. 2004, 2007).

Brief sampling and monitoring plan: We collected stream sediments, water, biofilm, macroinvertebrates, and trout throughout the watershed. Samples were collected from sites both upstream and downstream from the proposed remediation sites.

Pilot study: Selected sites downstream of remediated sites (<10) were chosen for sampling of macroinvertebrates following remedial action to test if macroinvertebrates are the best indicator of the recovery of the ecosystem (Farag et al. 2007).

Analyze and manage data: Extensive database in Access and GIS (Rich et al. 2004; database available at http://pubs.usgs.gov/pp/2004/1652/, accessed 9/27/13).

Involvement of stakeholders: We coordinated field work with local land holders, Federal and State agencies, and concerned citizens in the immediate area around Boulder, Montana.

Decisions made: We recommended that 96-hour *in situ* survival tests using trout fingerlings be conducted annually to determine mortality rates to monitor recovery of the trout fishery (USGS 2004).

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Conclusions: The trout fishery could be restored. Remediation could be phased in over a period of years followed by monitoring to evaluate recovery of the fishery (Finger et al. 2004). For example, Gelinas and Tupling (2004) summarized the remediation work on High Ore Creek. The USBLM and the State of Montana reclamation work in this drainage has resulted in a successful fish habitat in the major streams in the watershed (e.g., see www.scribd.com/doc/6150534/final10year-decade-of-progress-bs, www.hookandbullet.com/fishing-high-ore-creek-boulder-mt/, and www.hookandbullet.com/fishing-cataract-creek-boulder-mt/, accessed 9/27/13).

Were the data quality objectives met? Yes.

Costs and benefits: This study was designed to test good scientific practices that can be applied to other watershed areas and, therefore, was more costly than will be needed in the future for these deposit types in these geologic settings. As a result of this approach, additional sites were identified that were sediment traps that needed to be remediated. In addition, remediation sites were prioritized and alternative remedial approaches were recommended.

Lessons learned:

1. Macroinvertebrates are the best indicators of environmental recovery because of their multiple life cycles, and native species are available in tributary streams (Farag et al. 2004).
2. Water chemistry is a transient indicator of the aquatic habitat because it changes throughout the day, season, and year (Kimball et al. 2004; Lambing et al. 2004; Nimick and Cleasby 2004).
3. Transfer of metals from the colloidal component of the sediment to biofilm was identified as a likely pathway for metals into biota (Farag et al. 2004, 2007).
4. Identifying pathways of metals from sources into the rivers was problematic (Kimball et al. 2004). The role of geologic structures also must be understood (McDougal et al. 2004; O'Neill et al. 2004).
5. Tracer studies are important in identifying sources of metals (Kimball et al. 2004), but geologic pathways are not identified.
6. Any watershed study must have a fundamental conceptual model of groundwater flow (McDougal et al. 2004).
7. During spring runoff, large amounts of metals were mobilized by snow melt, which saturated the mill tailings in the riparian zone and moved metals into the upstream reaches of Basin Creek (Cannon et al. 2004).

Recommendations: USGS (2004) recommended 96-hour *in situ* tests on trout fingerlings for mortality rates to monitor recovery to assess success of remediation.

Bibliography:

Cannon, M.R., Church, S.E., Fey, D.L., McDougal, R.R., Smith, B.R., and Nimick, D.A., 2004, Understanding trace-element sources and transport to upper Basin Creek in the vicinity of the Buckeye and Enterprise mines, *in* Nimick, D.A., Church, S.E., and Finger, S.E., eds., Integrated investigations of environmental effects of historical mining in the Basin and

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Boulder mining districts, Boulder River watershed, Jefferson County, Montana: U.S. Geological Survey Professional Paper 1652, p. 401-456. http://pubs.usgs.gov/pp/2004/1652/, accessed 9/27/13.

Church, S.E., Unruh, D.M., Fey, D.L., and Sole, T.C., 2004, Trace elements and lead isotopes in streambed sediment in streams affected by historical mining, *in* Nimick, D.A., Church, S.E., and Finger, S.E., eds., Integrated in*ves*tigations of environmental effects of historical mining in the Basin and Boulder mining districts, Boulder River watershed, Jefferson County, Montana: U.S. Geological Survey Professional Paper 1652, p. 279-336. http://pubs.usgs.gov/pp/2004/1652/, accessed 9/27/13.

Farag, A.M., Nimick, D.A., Kimball, B.A., Church, S.E., Skaar, Don, and Brumbaugh, W.G., McConnell, Elizabeth, and Hogstrand, Christer, 2004, Aquatic health and exposure pathways of trace elements, *in* Nimick, D.A., Church, S.E., and Finger, S.E., eds., Integrated investigations of environmental effects of historical mining in the Basin and Boulder mining districts, Boulder River watershed, Jefferson County, Montana: U.S. Geological Survey Professional Paper 1652, p. 369-400. http://pubs.usgs.gov/pp/2004/1652/, accessed 9/27/13.

Farag, A.M., Kimball, B.A., Nimick, D.A., Church, S.E., and Brumbaugh, W.G., 2007, Concentrations of metals in water, sediment, biofilm, benthic macroinvertebrates and fish in the Boulder River Watershed, Montana, and the role of colloids in metal uptake: Archives of Environmental Contamination and Toxicology, v. 52, p. 397-409.

Finger, S.E., Farag, A.M., Nimick, D.A., Church, S.E., and Sole, T.C., 2004, Synthesis of water, sediment, and biological data using hazard quotients to assess ecosystem health, *in* Nimick, D.A., Church, S.E., and Finger, S.E., eds., Integrated investigations of environmental effects of historical mining in the Basin and Boulder mining districts, Boulder River watershed, Jefferson County, Montana: U.S. Geological Survey Professional Paper1652, p. 29-48. http://pubs.usgs.gov/pp/2004/1652/, accessed 9/27/13.

Gelinas, S.L., and Tupling, Robert, 2004, Monitoring remediation—Have mine-waste and mill-tailings removal and flood-plain restoration been successful in the High Ore Creek valley? *in* Nimick, D.A., Church, S.E., and Finger, S.E., eds., Integrated investigations of environmental effects of historical mining in the Basin and Boulder mining districts, Boulder River watershed, Jefferson County, Montana: U.S. Geological Survey Professional Paper 1652, p. 457-474. http://pubs.usgs.gov/pp/2004/1652/, accessed 9/27/13.

Kimball, B.A., Runkel, R.L., Cleasby, T.E., and Nimick, D.A., 2004, Quantification of metal loading by tracer injection and synoptic sampling, 1997-1998, *in* Nimick, D.A., Church, S.E., and Finger, S.E., eds., Integrated investigations of environmental effects of historical mining in the Basin and Boulder mining districts, Boulder River watershed, Jefferson County, Montana: U.S. Geological Survey Professional Paper 1652, p. 191-263. http://pubs.usgs.gov/pp/2004/1652/, accessed 9/27/13.

Lambing, J.H., Nimick, D.A., and Cleasby, T.E., 2004, Short-term variation of trace-element concentrations during base flow and rainfall runoff in small basins, August 1999, *in* Nimick, D.A., Church, S.E., and Finger, S.E., eds., Integrated investigations of environmental effects of historical mining in the Basin and Boulder mining districts, Boulder River watershed, Jefferson County, Montana: U.S. Geological Survey Professional Paper 1652, p. 263-278. http://pubs.usgs.gov/pp/2004/1652/, accessed 9/27/13.

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

McDougal, R.R., Cannon, M.R., Smith, B.D., and Ruppert, D.A., 2004, Hydrogeology of the Boulder River watershed study area, and examination of regional ground-water flow using interpreted fracture mapping from remote sensing data, *in* Nimick, D.A., Church, S.E., and Finger, S.E., eds., Integrated investigations of environmental effects of historical mining in the Basin and Boulder mining districts, Boulder River watershed, Jefferson County, Montana: U.S. Geological Survey Professional Paper 1652, p. 337-368. http://pubs.usgs.gov/pp/2004/1652/, accessed 9/27/13.

Metesh, J.J., Lonn, J.D., Duaime, T.E., and Wintergerst, Robert, 1994, Abandoned-inactive mines program, Deerlodge National Forest—Volume I, Basin Creek Drainage: Montana Bureau of Mines and Geology Open-File Report 321, 131 p.

Metesh, J.J., Lonn, J.D., Duaime, T.E., and Wintergerst, Robert, 1995, Abandoned-inactive mines program, Deerlodge National Forest—Volume II, Cataract Creek Drainage: Montana Bureau of Mines and Geology Open-File Report 344, 201 p.

Nimick, D.A., Church, S.E., and Finger, S.E., eds., 2004, Integrated investigations of environmental effects of historical mining in the Basin and Boulder mining districts, Boulder River watershed, Jefferson County, Montana: U.S. Geological Survey Professional Paper 1652, 524 p. http://pubs.usgs.gov/pp/2004/1652/, accessed 9/27/13.

Nimick, D.A., and Cleasby, T.E., 2004, Trace elements in water in streams affected by historical mining, chap. D5 in Nimick, D.A., Church, S.E., and Finger, S.E., eds., Integrated investigations of environmental effects of historical mining in the Basin and Boulder mining districts, Boulder River watershed, Jefferson County, Montana: U.S. Geological Survey Professional Paper 1652, p. 155-190. http://pubs.usgs.gov/pp/2004/1652/, accessed 9/27/13.

O'Neill, J.M., Lund, Karen, Van Gosen, B.S., Desborough, G.A., Sole, T.C., and DeWitt, D.H., 2004, Geologic framework, *in* Nimick, D.A., Church, S.E., and Finger, S.E., eds., Integrated investigations of environmental effects of historical mining in the Basin and Boulder mining districts, Boulder River watershed, Jefferson County, Montana: U.S. Geological Survey Professional Paper 1652, p. 48-88. http://pubs.usgs.gov/pp/2004/1652/, accessed 9/27/13.

Rich, C.L., Litke, D.W., Granitto, Matthew, Pelltier, R.T., and Sole, T.C., 2004, Digital databases and CD-ROM for the Boulder River watershed, *in* Nimick, D.A., Church, S.E., and Finger, S.E., eds., Integrated investigations of environmental effects of historical mining in the Basin and Boulder mining districts, Boulder River watershed, Jefferson County, Montana: U.S. Geological Survey Professional Paper 1652, p. 503-524. http://pubs.usgs.gov/pp/2004/1652/, accessed 9/27/13.

USGS (U.S. Geological Survey), 2004, Summary and conclusions from investigations of the effects of historical mining in the Boulder River watershed, Jefferson *Co*unty, Montana, *in* Nimick, D.A., Church, S.E., and Finger, S.E., eds., Integrated investigations of environmental effects of historical mining in the Basin and Boulder mining districts, Boulder River watershed, Jefferson County, Montana: U.S. Geological Survey Professional Paper 1652, p. 3-12. http://pubs.usgs.gov/pp/2004/1652/, accessed 9/27/13.

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SAMPLING MINE WASTE-ROCK PILES FOR PRIORITIZATION

Hillsboro District, New Mexico

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Purpose of case study: The purpose of this study was to develop a more cost effective approach to sampling mine-waste rock piles using a hand shovel and sampling to depths of 25 cm.

Brief site description and history: The Hillsboro mining district lies in the Animas Mountains in the eastern Black Range, New Mexico, and has been mined intermittently over the past 120 years. During the period 1877–1982 an estimated 270,000 ounces of gold, from lode and placer deposits, 78,000 ounces of silver, 24,000 pounds of lead, and 24 million pounds of copper were produced from the district. Abandoned mines with different types of deposits are present throughout the study area. Mine-waste rock piles from four different types of deposits are examined in this study. The placer gold waste-rock pile (Site A) is characterized by hematite, quartz, rock fragments, and trace pyrite. The Laramide polymetallic waste-rock piles (Sites B and E) contain pyrite, plagioclase feldspar, quartz, chalcopyrite, hematite, galena, and minor silver minerals. The Pb-Zn carbonate-hosted replacement waste rock pile (Site C) consists of galena, calcite, cerussite, anglesite, vanadinite, wulfenite, and manganese oxides. The Ag-Mn carbonate-hosted replacement waste rock pile (Site D) contains manganese oxides, rhodochrosite, and calcite. Mineralogical characteristics influence the level of impact the waste rock piles have on the environment (Munroe et al. 2000). Sulfides are typically unstable in the surface environment and, therefore, tend to be less resistant to weathering. Metals associated with pyrite, chalcopyrite, and galena therefore become more available to the environment. Secondary mineral oxidation rinds on sulfide and non-sulfide minerals can give insight into the probability of metal transfer from the mineral grains to the environment.

Define questions and objectives: Samples were collected to characterize the waste-rock piles.

Conceptual model: Different size fractions have different concentrations of metals and the size fraction with the largest concentrations will be utilized. Because the construction method of the waste rock piles can influence the distribution of the metal concentrations, the sampling grids and sampling densities were determined separately for each site (Figure A5.1 to A5.4). See Munroe (1999) and Munroe et al. (1999, 2000) for details on the different types of grids established and the rationale used. Geoenvironmental models are used to predict the environmental geochemistry of the four sites.

Costs and consequences of not sampling and monitoring: Mine-waste rock piles require sampling to establish if they are contributing to acid drainage and metals movement in the environment.

Data needs:

1. Determine which size fraction contained the highest metal concentrations. Samples were sieved in the field and only the <2 mm fraction was sampled. Particle size fractions 2-1

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mm, 1-0.5 mm, 0.5-0.25 mm, 0.25-0.125 mm, 0.125-0.063 mm, and <0.063 mm were subsequently sampled and analyzed (Figure A5.5).

2. Determine how many samples and where to collect from the surface of the waste rock piles. Grids and different sampling densities were established and tested (Figure A5.1 to A5.4; Munroe, 1999; Munroe et al., 1999, 2000).
3. Determine which analytical method, Flame Atomic Absorption Spectroscopy (FAAS) or X-ray Fluorescence Spectroscopy (XRF) was the best method for this project. FAAS involves a partial digestion process and measures only more soluble metal concentrations, whereas XRF is a direct measure of the total metal concentration.
4. Analyze for Cu, Pb, Zn, As, Fe, and Cd using both FAAS and XRF.
5. Quality assurance and quality control were maintained for accuracy and precision throughout the chemical analyses through the use of certified reference standards and in-house standards (Munroe 1999). Duplicate and triplicate samples also were prepared and analyzed. FAAS analyses typically were within ±15% of one another, whereas the XRF analyses were within ±2% (Munroe 1999).
6. Physical parameters of the waste rock piles sampled, such as construction method, size, lithology, grain-size distribution, terrain, physical dimensions, and weathering characteristics were examined (Munroe 1999; Munroe et al. 2000).
7. Examine the detailed mineralogy, texture, and mineral chemistry using electron microprobe.

Brief sampling plan: Samples were first collected from the mine-waste piles to determine the grain-size distribution and to obtain chemical compositions for different particle-size fractions in order to determine the optimum size fraction to be collected. The less than 0.25-mm size fraction was determined to be the fraction to be sampled (Figure A5.5; Munroe 1999; Munroe et al. 2000). This grain-size range was justified because it typically yielded the highest metal concentrations as discussed in Munroe (1999). Material was collected across the mid-slope of each of the waste-rock piles. The slope of each pile was divided into five zones and 1/5 of a 5-gallon bucket was filled for each zone. A small stainless-steel hand shovel was used throughout the sampling process. After the bucket was filled, the material was sieved to 2 mm and put into plastic sample bags. The >2-mm material was also bagged and saved. In the laboratory, the sample was sieved to obtain the <0.25-mm fraction for analyses.

One of the problems in sampling mine-waste rock piles is determining how many samples are needed and where in the pile they should be collected. In the second stage of this project, each of the four mine waste-rock piles was sampled using grids unique to each pile. Different grid patterns were used to determine the optimum grid. Samples were collected in three batches of 15 samples each. Each set of samples was collected from a unique grid system with no overlap in sample locations (Figures A5.1 to A5.4). Splits of samples were combined and analyzed in sets (i.e., 15, 30 and 45 samples) to determine the minimum number of samples necessary to adequately characterize a mine waste-rock pile geochemically. Sites A–D were sampled with sampling densities of 15, 30 and 45, each of which was combined to obtain three homogenized samples. The samples were mixed at least four times by cone and quarter methods until there was no longer a color variation. Once a single color was obtained the sample was split to form three subsamples which weighed approximately 1,000 g each. Each of the homogenized samples was ground for chemical analysis using a stainless-steel disc grinder to clay and silt size (<63 mm).

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Samples from Sites A, B, C, and D, and the composite/homogenized samples from each of the waste-rock piles were digested by dissolving 1 g in aqua regia (l mL HCl and 5 mL HNO_3). This procedure was repeated twice to dryness and then put into solution to a volume of 50 mL with 5% HNO_3. This dissolution process releases most metals bound in the sample, but the metals bonded in some silicates and other resistant minerals remain locked in the crystal lattice and are not completely recovered. Each sample was analyzed for Cu, Pb, Zn, and As by FAAS using an Instrumentation Laboratories Model Video 12 instrument according to manufacturer's specifications and XRF using a Phillips PW2400 instrument (Munroe 1999).

Pilot study: A preliminary study was conducted to test the procedures.

Analyze and manage data: Data were stored and graphed using Excel spreadsheets and RockWare Utilities.

Involvement of stakeholders: This was a thesis project and no stakeholders other than the mine owners were identified. The thesis committee consisted of three faculty members who reviewed the thesis and defense.

Decisions made: This work will be used in classifying existing and future waste rock piles at Hillsboro.

Conclusions:
Were the data quality objectives met? yes

Costs and benefits: The sampling procedures used in this study have proven to be an effective method in providing a first approximation of a waste-rock pile geochemical signature. The results also provide geochemical information that can be used to help prioritize subsequent characterization drilling. The exact costs were not determined, but included several days of field work, sample analyses, and data interpretation.

Lessons learned:

1. Typically, the smallest grain-size fraction sampled in each mine waste-rock pile contained higher concentrations of metals due perhaps to the mineralogical nature of the material (i.e., clay- and silt-size particles containing high cation-exchange capacity) and the higher surface area available for adsorption of ions (Figure A5.5; Munroe 1999). Therefore, in order to obtain the higher metal concentrations, the sampling strategy was to collect the <0.25 mm (250 mm) size fraction.
2. In some cases XRF metal concentrations were higher than FAAS, in others lower. However, trends for the two methods were similar.
3. Using grid patterns unique to each of the waste rock piles, three homogenized samples were obtained using sampling densities of 15, 30, and 45 sample cells. Cu, Pb, Zn, and As concentrations revealed that 15 and 30 samples per grid were similar to those for 45 samples per grid (Munroe et al. 1999). Therefore, mine waste-rock piles can be adequately characterized geochemically by homogenizing samples collected from a grid containing 15–30 sample cells.

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4. Secondary mineral oxidation rinds can coat a given mineral and prevent further metal release into the environment (Munroe et al. 2000).

Recommendations:

1. Develop different sampling procedures that account for the vertical mineralogical and geochemical heterogeneities within a mine waste rock pile.
2. Sample the different layers of different mineralogies or specific intervals regardless of mineralogy. This could provide insight into new methods for sampling mine waste-rock piles with or without a mineralogical sampling bias.

Bibliography:

Munroe, E.A., 1999, Geology and geochemistry of waste rock piles in the Hillsboro mining district, Sierra County, New Mexico: M.S. thesis, New Mexico Institute of Mining and Technology, Socorro, 144 p.

Munroe, E.A., McLemore, V.T., and Kyle, P., 1999, Waste rock pile characterization, heterogeneity and geochemical anomalies in the Hillsboro mining district, Sierra County, New Mexico: Journal of Geochemical Exploration, v. 67, p. 391-405.

Munroe, E.A., McLemore, V.T., and Dunbar, N.W., 2000, Mine waste rock pile geochemistry and mineralogy in southwestern New Mexico, USA; in ICARD 2000—Proceedings from the 5th international conference on acid rock drainage: Society for Mining, Metallurgy, and Exploration, Inc., Littleton, Colo., p. 1327-1336.

Figures:

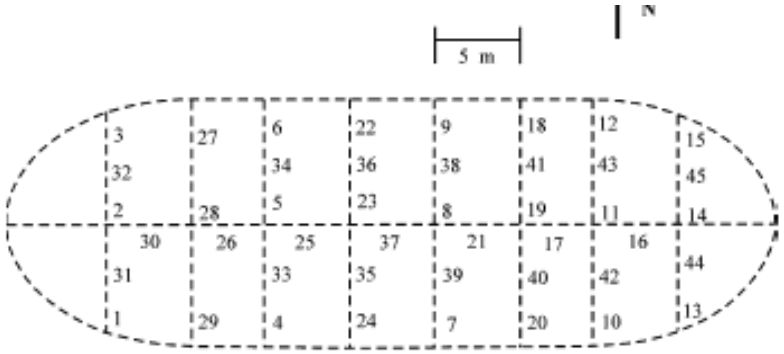


FIGURE A5.1 Sample locations for the placer gold waste-rock pile (site A). The numbers represent the sample numbers (Munroe, 1999).

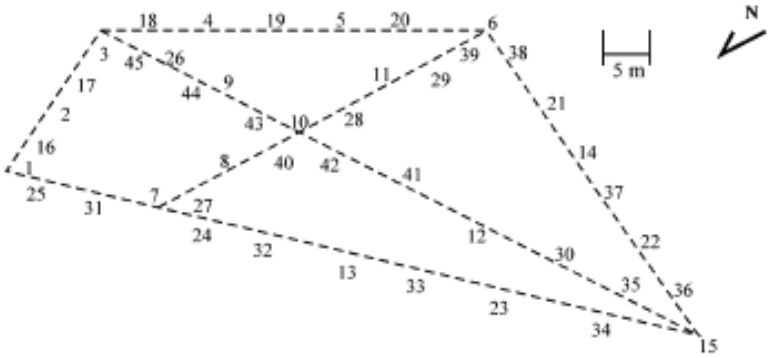


FIGURE A5.2 Sample locations for the Laramide polymetallic vein waste-rock pile (site B) (Munroe, 1999).

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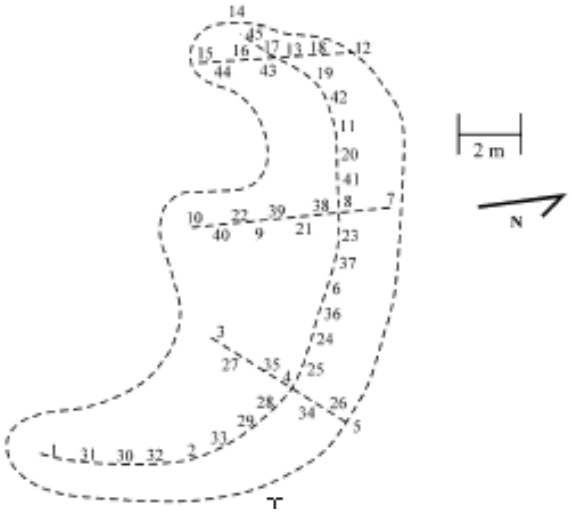


FIGURE A5.3 Sample locations for the carbonate-hosted Pb-Zn waste-rock pile (site C) (Munroe, 1999).

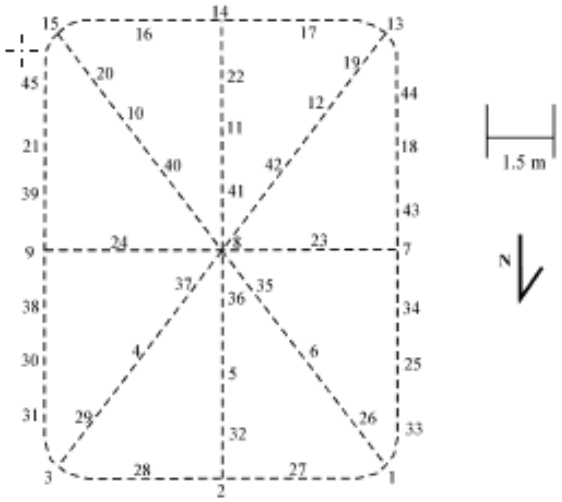


FIGURE A5.4 Sample locations for the carbonate-hosted Ag-Mn waste-rock pile (site D) (Munroe, 1999).

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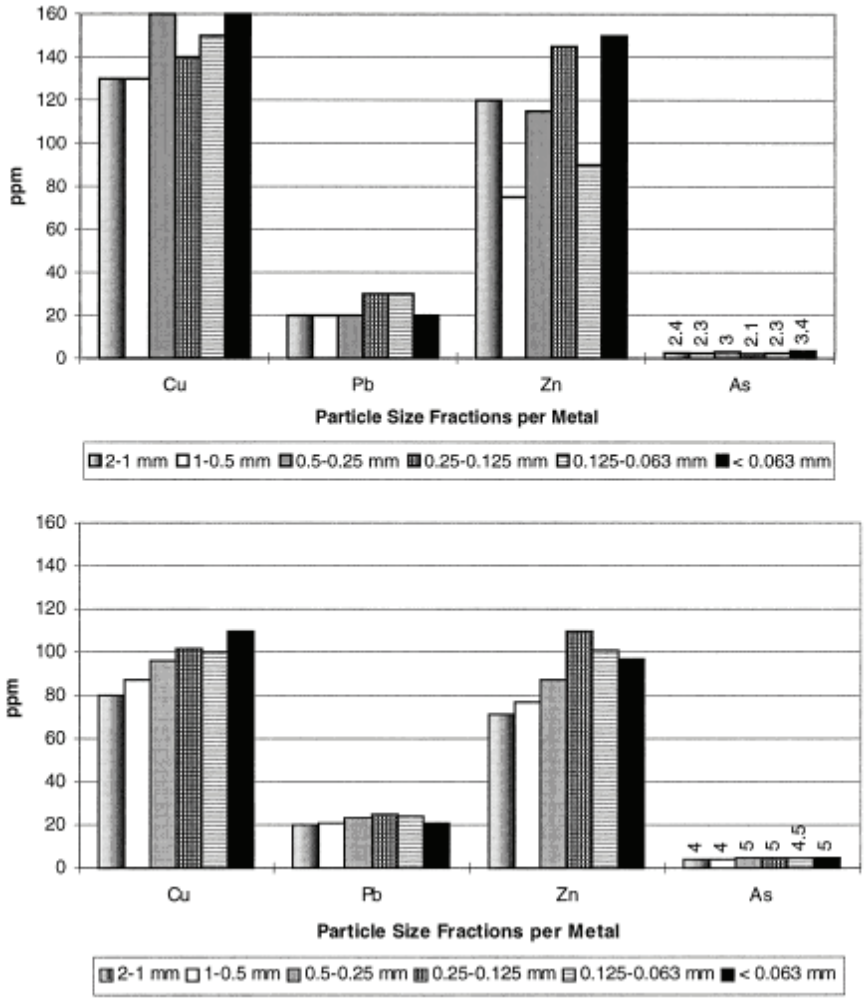


FIGURE A5.5 Chemical analyses of six size fractions of the gold placer waste-rock pile by (a) FAAS and (b) XRF. Other results are in Munroe (1999).

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SAMPLING AND MODELING TO DETERMINE AQUEOUS GEOCHEMICAL CONTROLS ON METAL TRANSPORT

North Fork Clear Creek in the Clear Creek Watershed, Colorado

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Purpose: The purpose of this case study is to present findings from a study that assessed aqueous geochemical controls on the spatial fate and transport of Cu, Fe, Mn, and Zn in a mining-impacted stream and to present findings from another study that evaluated effects of several parameters on the release of these metals from the streambed sediments in the absence of the mine-drainage inputs.

Site description and history: The North Fork of Clear Creek (NFCC), Colorado, is an acid-mine drainage impacted stream located approximately 48 km west of Denver, CO, is approximately 29 km long, and flows into the main stem of Clear Creek in Golden, CO (Butler, 2009; Butler et al., 2009). The entire Clear Creek watershed is a Superfund site, of which NFCC is Operable Unit 4. The stream is influenced by several mine-drainage sources resulting from extensive historical mining in the Central City–Idaho Springs Mining District in the Colorado Mineral Belt in the late 1800s. The primary metals of concern in the stream include Cu, Fe, Mn, and Zn. Seepage from waste and tailings piles along the stream also contributes to metal input. Immediately downstream from the primary mining inputs (Figure A5.6: NCC-SW-27 (Gregory Gulch); NCC-SW-20 (Gregory Gulch); and NCC-SW-17 (National Tunnel)), there is a visually apparent and rapid precipitation of iron oxyhydroxide (HFO) that travels downstream as particulates, forms loose coatings on rocks, and settles to the bed sediments. Hydrous manganese oxides (HMO) are seen to form hard coatings on rocks farther downstream, and aluminum oxyhydroxide (ALO) precipitation is observed during some months below Russell Gulch.

Define questions and objectives: Objectives of the two studies were to determine:

1. the primary controls on spatial fate and transport of Cu, Fe, Mn, and Zn; and
2. the primary controls expected on release of metals from streambed sediments to the stream water following removal/treatment of the mine-drainage inputs.

Data needs and sampling methods: Grab samples of water were synoptically obtained from locations along the stream reach (Figure A5.6) on May 21, 2002, June 3, 2004, September 14, 2004, and June 3, 2005. Water quality measurements included pH, alkalinity, ferrous iron, dissolved and total metals via ICP-AES (including Al, Ca, Cd, Cu, Fe, K, Mg, Mn, Na, Ni, and Zn), sulfate (calculated as 3 times the ICP-determined total S), and total organic carbon (TOC). Flow measurements were obtained by the Colorado Department of Public Health and Environment for most sites on each date, and calculated at other sites based on surrounding sites having measured values. QA/QC included replication at two sites for each spatial sampling date and a field blank.

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The absence of mine-drainage could result in increased dissolved organic carbon (DOC), decreased ionic strength, and increased pH relative to that originally observed in the mining-impacted stream water; each of these conditions might influence desorption of metals from existing streambed sediments. Aerobic sediment samples from the top 5 cm in 50 locations were collected from the region near NCC-SW-16 and NCC-SW-15 in April 2007 and combined for laboratory experimentation. Sediments were sieved at two size fractions (63 μm ≤ x < 2 mm; x < 63 μm) in the laboratory. A 2^4 x 10 factorial design assessed the influence of DOC, ionic strength, pH, size, and time on release of Cd, Cu, Fe, Mn, and Zn (measured by ICP-AES) from the sediments in batch tests. Individual units were prepared for each of 10 sampling times. QA/QC included replication of one time point in each of the 16 treatment combinations (sediment size plus treatment solution) and one blank for each of the 8 solutions tested.

Analysis and management of data: The spatial data were modeled with Visual-MINTEQ (www.lwr.kth.se/english/OurSoftware/Vminteq, accessed 9/27/13) to determine dominant controls on the fate and transport of the metals. Modeling included complexation with DOC, sorption to HFO, and sorption to HMO. Particulate iron and manganese were calculated from the difference in measured total and dissolved concentrations; each was assumed to comprise entirely its respective oxyhydroxide for input of mass of each solid available for modeling sorption processes. Measured TOC was assumed equivalent to DOC, which may result in an over-estimate of DOC complexation. Calculated percentages of particulate Cu and Zn from field samples were compared to model-predicted values.

Metals released from sediments were assessed using a general multivariate analysis of variance [MANOVA]. Particle size was determined to be such a strong factor in controlling metal release that assessment within each size fraction was required to determine the influence of the tested chemical factors.

Conclusions: QA/QC requirements were met with replicated spatial samples being within +/- 20% relative error and blanks having concentrations of analytes at least an order of magnitude below the lowest concentration in the stream water samples. Modeling generally over-estimated particulate Cu and under-estimated particulate Zn, with samples collected during lower flows having a closer comparison. Inclusion of DOC complexation improved comparisons between modeled and measured particulate Cu; inclusion of HMO improved comparisons between modeled and measured particulate Zn. One assumption in using equilibrium modeling is that the system is actually at equilibrium. This may not be true over all time and/or space within NFCC, especially during higher flow conditions or for all metals present. In a different study, both Cu and Zn were found to be at equilibrium at the farthest downstream site on a single date. In another study, Zn was found to be at equilibrium on a single date at a site in the mid-section of the stream reach, but Cu was not. Other assumptions made also may have influenced differences observed, such as all calculated particulate Fe and Mn being available for sorption processes. Under-prediction of Zn could indicate a sorbent is present in NFCC that was not measured or one that was not included in modeling. Whereas particulate Al was present during some dates below Russell Gulch, modeling did not include consideration of Al solids for sorption. It was desired to use a simple model without alterations to determine how closely observations could be predicted. Even with known limitations, Cu was predicted to within 1-54% of observed values and Zn was predicted to within 1-34%, over all sites and sampling dates.

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The majority of elements analyzed in the replicated batch sediment slurries were within the desired +/- 20% RPD; however some metals were outside this boundary for the 16 replicates analyzed (5 for Cd, 4 for Cu, 3 for Zn, and 11 for Fe). Large variations were observed in metals concentrations released as a function of time, even at short intervals of 1 and 10 minutes. It was not always possible to sample the replicates at exactly the same time, so small differences observed could have been due to the influence of time, or to small differences in ending pH values measured, especially for Fe. For data analysis, averages and standard deviations were used and errors propagated through interpretations. Blanks contained no metals above detection limits. Within each of the size fractions, each factor affected release of each metal differently; however, ionic strength and pH were seen to be most important for Mn and Zn, and DOC was most important for Cu. Overall, results suggested that relative to current conditions, a greater release of Cu and Fe and a lesser release of Cd, Mn, and Zn could be expected, if removal/treatment of the mine-drainage inputs results in an increase in DOC and a decrease in ionic strength.

Lessons learned: Fate and transport of metals in a mining-influenced stream depend on aqueous geochemical reactions and interactions, especially oxidation, precipitation, sorption, and complexation with organic carbon species. Each of these reactions and interactions between them is dependent to varying degrees upon pH, temperature, ionic strength, the concentrations of each ion and solid present, and kinetics. Similarly, release of metals from streambed sediments into the water column depend on these same factors, independently or interactively. These systems are inherently complex and may not be at equilibrium at all times and over all space. Probably the most important lesson learned is that metals behave differently, with controls on one not necessarily being controls on another, which leads to the importance of having sufficient data (type, quantity, and quality) to enable accurate simulation, interpretation, and understanding of a given metal's behavior.

Recommendations: It is important to understand what limitations exist for acquiring sufficient data and information for understanding complex mining-influenced water systems, both prior to and following remediation. An understanding of aqueous geochemistry is necessary in order to approximate what types of information are needed. It is recommended that as much relevant data as possible be collected within the typical time and cost constraints. Assumptions are often necessary, and these should be presented and their potential effect on interpretations made clear.

Bibliography:

Butler, B.A. 2009. Effect of pH, ionic strength, dissolved organic carbon, time, and particle size on metals release from mine drainage impacted streambed sediments. Water Research, 43(5):1392-1402.

Butler, B.A., Ranville, J.F., and Ross, P.E. 2009. Spatial variations in the fate and transport of metals in a mining-influenced stream, North Fork Clear Creek, Colorado. Science of the Total Environment, 407: 6223–6234.

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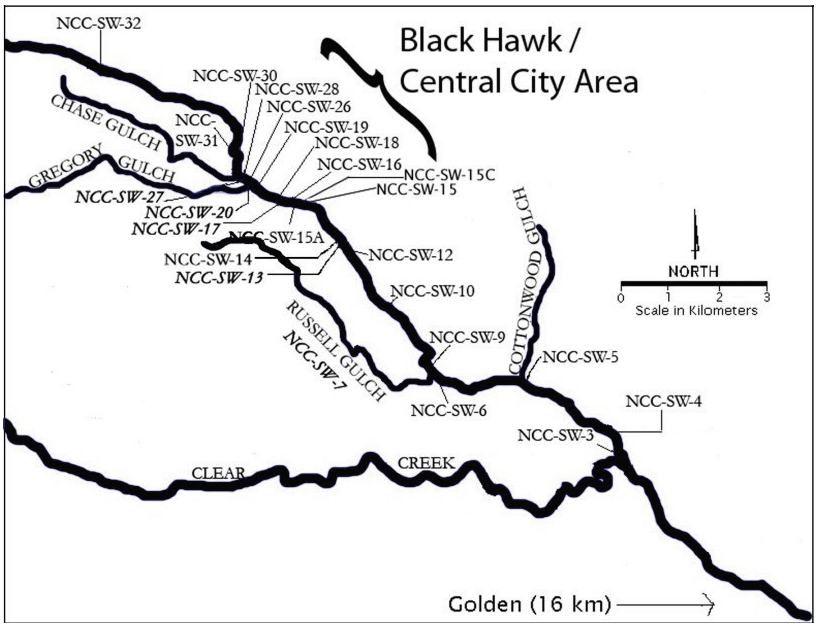


FIGURE A5.6 North Fork of Clear Creek, Colorado, site map and spatial sampling locations (Butler et al., 2009).

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WATER AND SEDIMENT SAMPLING FOR MERCURY AND BASE METALS TO EVALUATE REMEDIATION EFFORTS

Pecos Mine and Alamitos Canyon Mill, New Mexico

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Purpose of case study: The Pecos River originates in northern New Mexico and flows into the Rio Grande in Texas. Unreclaimed mining and milling areas in the upper Pecos drainage, active in the 1920s and 1930s, had the potential to affect water quality which could have regional, state and international concerns. This study was undertaken in order to determine mercury and trace metal transport in water and sediments as well as general water quality along the Pecos River from the Pecos Wilderness in northeastern New Mexico to Brantley Dam in southeastern New Mexico. The Lisboa Springs Fish Hatchery (New Mexico Game and Fish Department) located north of the village of Pecos with inlet from and outfall to the Pecos River had experienced several fish kills, which might have been caused by contaminated water. The study was begun in 1992 and continued until 2000.

Brief site description and history: The study area covers the southern Pecos Wilderness to south of Brantley Lake and includes many tributaries and the main stem of the Pecos River. The headwaters of the Pecos River are in the southern Sangre de Cristo Mountains, with additional drainages from the Pedernal uplift, Capitan Mountains, Sierra Blanca, and Guadalupe Mountains. Elevations range from 1000 m to over 3600 m. The river flows southward through the villages, towns and cities of Pecos, Santa Rosa, Fort Sumner, Roswell, Artesia, and Carlsbad into west Texas and finally enters the Rio Grande at the international border with Coahuila, Mexico. The Pecos River flows through the Santa Fe National Forest and the Pecos National Historical Park (U.S. National Park Service) and several state parks. Water from the Pecos River is stored in three main reservoirs: Santa Rosa, Sumner, and Brantley Lakes. Eastern New Mexico is predominantly a rural agricultural area, but the larger cities have some light industry. Petroleum and potash production is important in southeastern New Mexico near Artesia and Carlsbad. There is currently no metal mining within the Pecos drainage basin. Reclamation of the campgrounds, roads, and Alamitos Canyon mill site began in the early 1990s and was nearly completed as of 1999. Reclamation of the Pecos mine site began in 1999 and was ongoing in 2000. It was completed in 2004. Areas were reclaimed by the New Mexico Highway Department and private contractors under the oversight of the New Mexico Environment Department. The Lisboa Springs Fish Hatchery closed, temporarily, in 2000 because the fish were diagnosed with Whirling disease, which was not related to metals contamination from the Pecos mine.

Define questions and objectives: Three main sources of potential contamination were identified:

- Pecos mine
- Alamitos Canyon mill (El Molino site)
- Material from the mine waste pile that had been used as fill in the campgrounds and roads north of Terrero

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Twenty-three sample sites were chosen along the Pecos River from the Wilderness area to Brantley Dam north of Carlsbad. Water and sediment samples were taken from each site to investigate mercury and base metal (Cd, Cr, Cu, Pb, and Zn) transport. Field measurements of pH, conductivity, temperature, and alkalinity were taken to define basic water quality.

Conceptual model: Spring runoff and heavy summer thunderstorms in the upper Pecos combined with the oxidation of mine and mill waste can produce acidic conditions, which could result in metal transport down the river and could also be responsible for fish kills at the hatchery. Municipal discharges and irrigated agriculture could also be affecting the water quality on the lower Pecos.

Costs and consequences of not sampling and monitoring: Long-term monitoring of the geochemistry of surface water and stream sediment of the Pecos River were needed in order to determine if multiple uses of the river were causing water quality problems. Also, sample sites above and below potential mine waste runoff were chosen to establish whether they were contributing to acid drainage and metals movement in the environment.

Data needs: Acidified water samples were required for trace metal analysis and non-acidified samples for general water quality. To reduce the number of samples taken back to the lab, field analyses were done for general water quality parameters. Trace metals in the water samples were determined by graphite furnace atomic absorption. Mercury was analyzed by the cold vapor-atomic absorption method. Stream sediment samples were collected at each site and lake bottom samples were taken from Santa Rosa, Sumner, and Brantley Lakes. Lake bottom samples were collected using a bottom-grab sampler that sampled only the upper lake sediment. Water depths and bathymetry were not known. Sediment samples were sieved in the laboratory into two size fractions: <75 μm (suspendable sediment) and >75 μm to 2 mm (bottom sediment). Both fractions were analyzed for base metals by flame atomic absorption. Mercury was determined by the silver wool-atomic absorption method. See references below for more details on the sampling and analytical techniques.

Brief sampling and monitoring plan: In September 1992, the New Mexico Bureau of Mines and Mineral Resources (NMBMMR) sampled and analyzed water and stream sediments from the Pecos River at sites near the Pecos Wilderness boundary southward to below Brantley Lake, north of Carlsbad (McLemore et al. 1993). Subsequently, the U.S. Bureau of Reclamation, in cooperation with the New Mexico Institute of Mining and Technology (NMIMT) and NMBMMR, initiated a three-year multi-disciplinary study of the headwaters of the Pecos River in order to identify and prioritize point and non-point sources of contamination so that measures could be taken to protect water supplies for municipal, irrigation, and recreational use and to protect wildlife habitat (Brandvold et al. 1995; Brandvold and McLemore 1999; McLemore et al. 1995a, b; Popp et al. 1996). In September 1996 and June 2000, the NMBMMR again sampled water and stream sediments from the Pecos River from the Pecos Wilderness southward to below Brantley Lake. The 2001 report presents a summary of the first nine sampling trips in 1992-1995 (McLemore et al. 1993; Popp et al. 1996) and presents new data from the sampling trips in 1996 and 2000 (McLemore and Brandvold 2001).

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Pilot study: A reconnaissance study was done in 1992 to test sample sites and analytical methods, and gather data before reclamation.

Analyze and manage data: Data were stored, graphed, and analyzed using Excel spreadsheets.

Involvement of stakeholders: Stakeholders were not involved in the project, however five papers were published on the results and were available for use by stakeholders.

Decisions made: Although metal concentrations in the immediate vicinity of the mine have decreased, concentrations remain higher below the Pecos mine than above it, suggesting that metals continue to be eroded and leached from the Pecos mine waste piles. The overall metal concentrations have dramatically decreased in stream sediments below Pecos village, mostly due to dilution of sediment downstream (McLemore et al. 1995a, b, 2001; Carey et al., 2001).

Conclusions:
Were the data quality objectives met: Yes.

Costs and benefits: Costs were shared across the university by the NMBMMR and the NMT Chemistry and Biology Departments, and were not quantified. The nine year study resulted in hiring and training of undergraduate as well as graduate students and multiple publications, which expanded the knowledge base of the Pecos River water quality (see cited references).

Lessons learned:

1. Although sediment Cu, Pb and Zn concentrations were high in the immediate vicinity of the mine and mill in 1992, these metals were not elevated in downstream sediments due to the large input of sediment at points below the village.
2. Since reclamation of the Pecos mine, Alamitos Canyon mill, campgrounds and roads began in 1990-1991, Cu, Pb and Zn concentrations have decreased overall.
3. The overall metal concentrations from 1992-2000 have dramatically decreased in stream sediments along Alamitos Creek and at the confluence with the Pecos River due to the mill reclamation and below Pecos village due to dilution of sediment downstream.
4. Although metal concentrations in the immediate vicinity of the mine have decreased from 1992-2000, concentrations remain higher below the Pecos mine than above it, suggesting that metals continue to be eroded and leached from the Pecos mine-waste piles. The reclamation at the mine site had not been completed in 2000, however; so there may no longer be higher metals concentration below the mine site.

Recommendations: Reclamation of the roads and campgrounds was completed by 1994; reclamation at the El Molino mill site was completed by 1999 and reclamation of the immediate Pecos mine area was completed in 2004. Additional study could be done along the same length of the river to further document the effects of reclamation and continued river utilization by agriculture, industry and public utilities.

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Bibliography:

Brandvold, L.A. and McLemore, V.T., 1999, A study of the analytical variation of sampling and analysis of stream sediments from mining and milling contaminated areas: Journal of Geochemical Exploration, v. 64, pp. 185-196.

Brandvold, L. A., McLemore, V. T., and O'Connor, C., 1995, Distribution and partitioning of copper, lead and zinc in stream sediments above and below an abandoned mining and milling area near Pecos, New Mexico: The Analyst, v. 120, p. 1485-1495.

Carey, E.A., Brandvold, L.A., and McLemore, V.T., 2001, Base metal and trace element concentrations in stream sediments along the Pecos River, from the southern Pecos Wilderness to Brantley Dam, north of Carlsbad, eastern New Mexico: American Society for Surface Mining and Reclamation, 18th annual meeting, 2001 proceedings volume 1, p. 246-254.

McLemore, V.T., Brandvold, L.A., and Brandvold, D.K., 1993, A reconnaissance study of mercury and base metal concentrations in water, and stream- and lake-sediment samples along the Pecos River, eastern New Mexico. In Carlsbad Region (New Mexico and West Texas): New Mexico Geological Society Guidebook 44, p. 339-351.

McLemore, V.T., Brandvold, L.A., Brandvold, D.K., Kirk, K., Popp, C. Hansen, S., Radkte, R., Kyle, P.R., and Hossain, A.M., 1995a, A preliminary summary of multidisciplinary studies in the upper Pecos River area, Santa Fe and San Miguel Counties, New Mexico. In Geology of the Santa Fe Region, New Mexico: New Mexico Geological Society, Guidebook 46, p. 331-338.

McLemore, V. T., Brandvold, L. A., Hossain, A. M., and Pease, T. C., 1995b, The effect of particle size distribution on the geochemistry of stream sediments from the upper Pecos River, San Miguel County, New Mexico. In Geology of the Santa Fe Region, New Mexico: New Mexico Geological Society, Guidebook 46, p. 323-329.

McLemore, V.T. and Brandvold, L.A., 2001, Long-term monitoring of the geochemistry of surface water and stream-sediment samples from the upper Pecos River, from the southern Pecos wilderness to Brantley Dam, north of Carlsbad, eastern New Mexico: http://geoinfo.nmt.edu/staff/mclemore/projects/pecos/home.html, accessed 9/27/13.

McLemore, V.T., Brandvold, L.A. and Carey, E.A., 2001, Long-term monitoring of the geochemistry of surface water and stream-sediment samples from the upper Pecos River, from the southern Pecos Wilderness to Brantley Dam, North of Carlsbad, eastern New Mexico; year nine. In Geology of the Llano Estacado: New Mexico Geological Society Guidebook 52, p. 321-332. http://nmgs.nmt.edu/publications/guidebooks/downloads/52/52_p0321_p0332.pdf, accessed 9/27/13

Popp, C. J., Brandvold, D. K., Kirk, K., Brandvold, L. A., McLemore, V. T., Hansen, S., Radtke, R., and Kyle, P., 1996, Reconnaissance investigation of trace metal sources, sinks, and transport in the upper Pecos River Basin, New Mexico: U.S. Bureau of Reclamation, Report No. 3-FC-4D-13830, 224 p.

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SAMPLING TO DETERMINE GEOCHEMICAL CONTROLS ON SELENIUM TRANSPORT AND FATE

Wooley Valley Phosphate Mine, Unit 4 Waste Pile, Southeastern Idaho

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Purpose of case study: Selenium (Se) toxicity is a known, serious threat to wildlife, waterfowl and livestock (particularly sheep and cattle) in the western United States. Much of the grazing and agricultural land in this region is underlain by Permian and Cretaceous-aged marine shales that exhibit high concentrations of Se. Mining in these areas also can enhance the release of Se to the environment, resulting in bioaccumulation of Se in food sources (e.g., browse forage and benthic invertebrates). Animals that consume high-Se foods may suffer from toxicosis and teratogenesis, leading to their death. This cycle of Se release and bioaccumulation in forage has been implicated in deaths of sheep and cattle in southeastern Idaho, an area where black marine shales are mined for phosphate.

Brief site description and history: Idaho phosphate mines occur in black, organic-rich shale and carbonate rocks of the Meade Peak Member of the Permian Phosphoria Formation. These shales are known to contain high concentrations of trace metals/elements including Ag, As, Cd, Cr, Cu, F, Mo, Ni, Sb, Se, Tl, U, V, and Zn. A typical deposit in the Meade Peak Member consists of a steeply dipping upper chert bed (>200 ft), which overlies an upper ore zone (10-15 ft), middle waste zone (80-120 ft), lower ore zone (20-40 ft), and basal carbonate rocks. The middle waste zone is a black shale with high concentrations of trace metals and, as part of the mining process, is discarded in a large waste pile. The Unit 4 waste pile from the Wooley Valley Mine (WVU4) was created in the 1970s, has a volume of approximately 10 million yd^3, and is sited in a valley in a low mountainous terrain with approximately 22 inches of rain/year. Percolation of surface and groundwater through the waste pile will leach trace metals from the solid phase. When this water emerges at the base of the pile (as seepage or from drains) then potentially toxic trace elements are transported into the downstream environment. A wetland has formed from seepage at the base of WVU4, and this environment has served as a natural laboratory to study metals transport from the waste rock and uptake by sediments and vegetation (Figure A5.7).

Problem definition:

Questions and objectives:

1. To understand the rate of Se release from the waste dump materials, and
2. To understand the role of the wetland processes (surface water flow and interaction with pore water, Se adsorption and co-precipitation with Fe oxides, oxidation/reduction in pore waters and sediments, wetland vegetation) in Se cycling and transport from the mine property.

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Conceptual model: Mineralogical studies have shown that the most common form of Se in the solid phase waste material is elemental Se or Se(0) (Hein 2004, Ch. 8). This is a secondary form of Se, created from bacterial oxidation of the primary form of Se, which is unknown but likely to be selenides associated with sulfide minerals and organic compounds. Oxidation of Se(0) due to exposure to the atmosphere or dissolved O_2 in pore water converts Se(0) to the soluble forms of Se(IV) and Se(VI), which are transported out of the waste pile by groundwater flow. This flow discharges to the natural wetland formed at the base of the waste pile.

Data needs: To address objective (1) it is necessary to have information on the kinetics of Se release from the waste-rock materials. Objective (2), assessing Se cycling and transport, requires knowledge of the water flow to and from the site, and concentrations of Se in the surface water, sediments and vegetation.

Sampling and monitoring plan: Data were collected from May through October of each sampling year to measure water flow to and from the wetland, and the chemistry of surface water, sediments, and plants in the wetland (Figure A5.7).

Water flow measurements: Water flow was measured by two methods: first, using the velocity meter/cross-sectional area method, and second, using a water level data-logger and the rating curve (flow as a function of water level) constructed from method 1.

Surface water chemistry: Surface water samples were collected daily with automatic samplers. Samples were transported to the laboratory for filtration (0.45 μm MCE membrane) and acidification. Chemical analyses were performed by ICP-MS and ICP-AES, where detection limits for Se are 0.2 and 12 μg/L, respectively.

Sediments: Sediment cores were collected to a depth of 15 cm with a BMH-53 piston-type core sampler or, for coarse sediment samples, with a stainless steel scoop. Each sample was air-dried and sieved through a 32-mm stainless steel sieve to remove coarse fragments. Selective extractions were performed to determine the Se concentrations associated with the exchangeable, carbonate, Mn oxide, non-crystalline Fe oxide, crystalline Fe oxide, and organic matter + sulfide fractions. Each extract was analyzed for total Se by ICP-AES. Total recoverable Se in each sediment sample was determined by digestion in HNO_3 + $HClO_4$ followed by ICP-AES analysis.

Plants: A sampling transect with points 30-50 m apart was constructed longitudinally within the wetland, following the flow path outlined by the beaver ponds. The above-ground portion of each sampled plant species was clipped, bagged, and taken to the lab for identification and pre-analysis preparation. Samples were dried in a mechanical convection oven at 60°C for 48 hr and ground to <20 mesh in a stainless steel grinding mill. Selenium was determined by hydride generation atomic-absorption spectroscopy (HG-AAS).

Analysis and management of data: Samples were collected and analyzed as described above.

Objective 1: Se concentration in seepage water from the waste dump (location e, Figure A5.7) was collected for the years 2002, 2006, and 2008. Plots of Se concentration vs. time were modeled with a linear form of a 1st order kinetic rate equation, which provided an average rate constant for Se release of 3.47e-3 hr^{-1} (Stillings and Amacher 2010).

Objective 2: Measurements of water inflow and outflow to/from the wetland were collected from May through September 2002. The inflow at the main seep (point e, Figure A5.7) measured ~0.002 m^3s^{-1} in May, but quickly dropped to 0 m^3s^{-1} by the end of June, characteristic of the pulse of snowmelt observed in a seasonal hydrograph. At the outflow to the wetland (point

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t, Figure A5.7) flow ranged from 0.0002 m^3s^{-1} in May, to 0.003 m^3s^{-1} at the end of September. This steady increase in outflow over 4.5 months was due to a diffuse groundwater discharge to the wetland.

Se concentration at the inflow was measured at 5 mg L^{-1} in May, 2002, and dropped to 0.2 mg L^{-1} by the end of June. At the outflow Se concentrations were constant from May through September 2002, at 0-0.04 mg L^{-1}. These flow and concentration data were used to calculate Se flux. Influx was high in early May, ~90 g day^{-1}, due to the initial, high concentration of Se. Influx values dropped to 0 g day^{-1} over time, as both Se concentration and discharge decreased. Se outflux was low throughout the season, ranging from ~0.05 to 2.0 g day^{-1}, and showed a slight increase in September with increasing discharge volumes.

Results of the sediment analysis showed bulk Se concentration ranged from 10 to 700 mg kg^{-1}, with the greatest concentration found close to the main seep (site e, Figure A5.7). At this location the bulk of the Se resided in the organic + sulfide fraction, although there was a significant concentration in the non-crystalline iron oxide fraction. Bulk Se concentration in the sediment decreased with increasing distance downstream from the main seep. In the samples collected from the middle of the wetland to the outlet, the bulk of the Se was found in the non-crystalline Fe fraction.

Plant tissue collected from the wetland contained Se concentrations from 5 to 80 mg kg^{-1}, with concentrations in legumes > trees > grasses > shrubs > forbs. Plant Se dropped exponentially along the wetland flow path, to fall below 5 mg kg^{-1} at approximately 150 m from the inlet seep.

A mass balance on Se was constructed for the wetland for the period of May through September 2002. A mass of 695 g of Se was delivered to the wetland from the main seep at the base of the mine dump during this period. A total of 84 g of Se left the wetland through the surface outflow. The difference between these numbers shows that 611 g of Se were retained within the wetland (Figure A5.8) via sequestering by sediments and plants.

Conclusions:

Were the data quality objectives met? The laboratory QA/QC data were met as evaluated by submitting sample blanks, duplicates, and blind standards. The more important question is whether all necessary data were collected, and whether the data analysis, as performed, yielded the most useful results.

Lessons learned: The published results from objective 2 were produced from only one season of monitoring, and in this year the inflow water ceased in June, when the main seep dried out. The reason for the short period of inflow was that the previous winter had been dry with minimal snowpack. In regions of the western U.S. characterized by recurring drought it would be important to conduct a monitoring program over a period of several years to assess the full variability of the hydrologic cycle.

Recommendations: A monitoring program ought to be conducted for more than one hydrologic season. To reduce costs, collection frequency for water samples could be decreased from daily to weekly or even monthly, especially once the initial pilot project has finished. A decrease in frequency means less data resolution, but this might be acceptable, depending upon the project objectives.

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The one Se flux that was not measured was release of volatile Se to the atmosphere. Preliminary data (Hein 2004, Ch. 15) suggest that small, but measureable, concentrations of Se (30-90 ng m^{-3}) occurred in the near-ground atmosphere. Concentrations of this magnitude may or may not affect the mass balance illustrated in Figure A5.8. Se flux measurements would be needed to fully assess this contribution to the mass balance.

Bibliography:

Hein, J.R., ed., 2004. Life Cycle of the Phosphoria Formation: From Deposition to Post-Mining Environment. Handbook of Exploration and Environmental Geochemistry, vol. 8, Elsevier B.V., Amsterdam. See chapters 8, 15, 17.

Stillings, L.L., and Amacher, M.C., 2010. Kinetics of selenium release in mine waste from the Meade Peak Phosphatic Shale, Phosphoria Formation, Wooley Valley, Idaho, USA. Chemical Geology, v. 269, p. 113-123.

Stillings, L.L., Willard, P.J., Amacher, M.C. 2007. Geochemical Controls on Se: A comparison of two wetlands. In Bullen, T.D., and Wang, Yanxin, eds. Water-Rock Interation-12, vol 2, p. 1271-1275.

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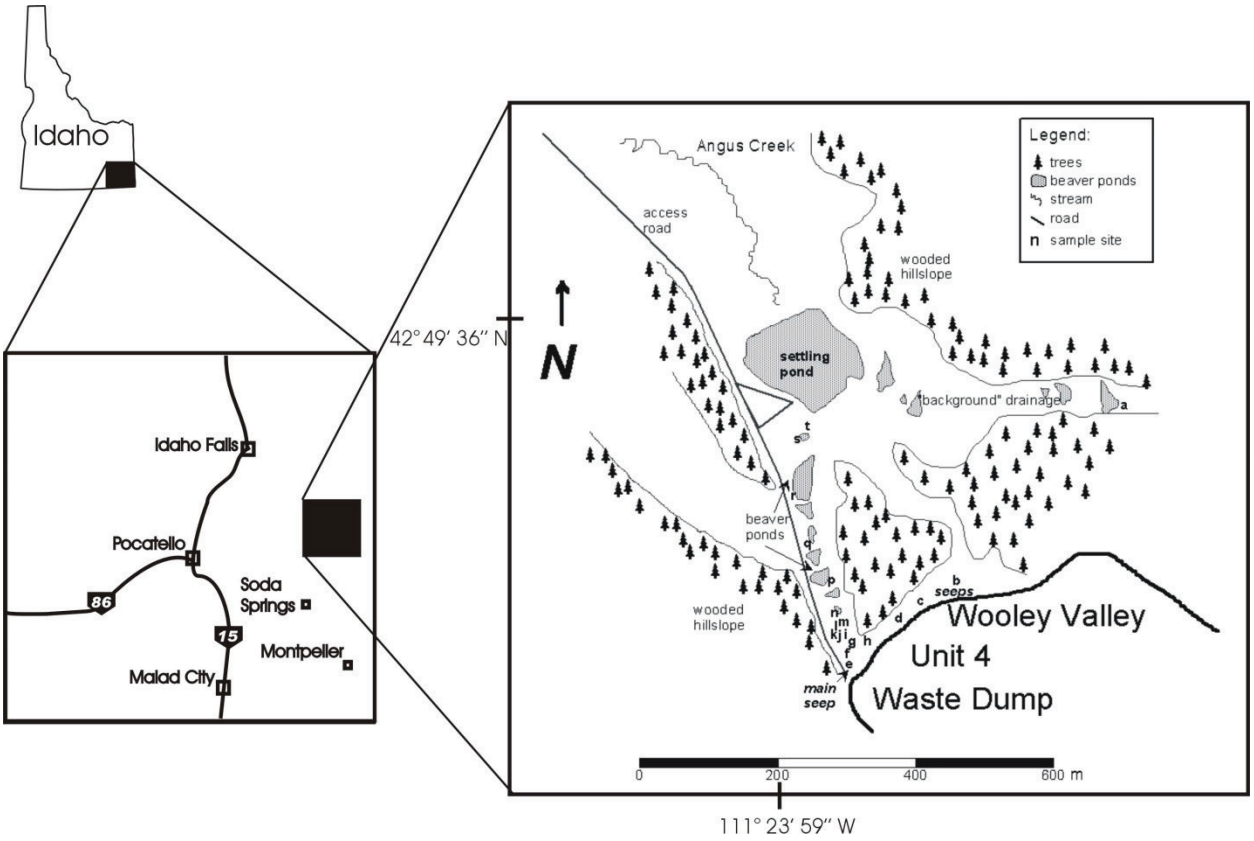


FIGURE A5.7 Location and schematic map for the natural wetland that developed from seepage from the Wooley Valley Unit 4 waste dump. Surface water in the wetland flows northward from the main seep following the line of beaver ponds. It flows into the settling pond and then to Angus Creek. Sample locations are marked by letters (after Hein, 2004).

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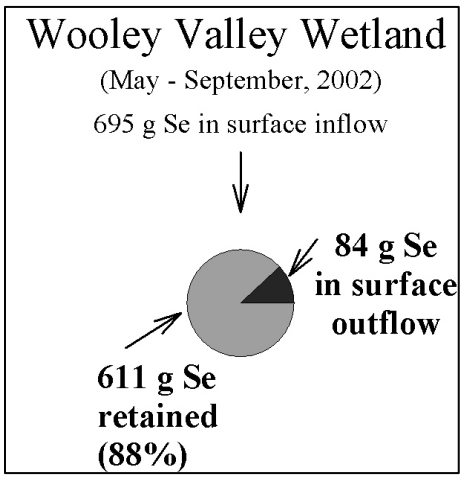


FIGURE A5.8 Mass balance of Se in the Wooley Valley Wetland, May-September 2002 (after Stillings et al. 2007).

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ASSESSING THE IMPACT OF HISTORICAL MINING AND LITHOLOGY ON AQUATIC ECOSYSTEMS

Central Colorado Assessment Project, Colorado

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Purpose of case study: To determine how natural geologic processes and historical mining interact to control the geochemistry and ecology of headwater streams in the Rocky Mountains of Central Colorado.

Brief site description and history: Central Colorado was heavily mineralized during the Larimide orogeny by emplacement of plutons and associated sulfide mineral deposits of various types (Tweto and Simms 1963). Recent surveys of mountain streams in Colorado suggest that up to 25 percent are degraded by elevated metal concentrations (Clements et al. 2000). Generally, this degradation is assumed to result from the mineral extraction economy that began in central Colorado in 1859 (Chronic and Chronic 1972). It is largely unknown to what extent weathering has released metals that affect aquatic ecosystems in this region.

Problem definition:

Define questions and objectives: We evaluated the extent to which historical mining versus hydrothermal alteration (Robb 2005) and ore deposit type (Cox and Singer 1986) influenced the availability of metals to aquatic ecosystems in the streams of central Colorado (Church et al. 2009; Schmidt et al. 2009, 2012; Wanty et al. 2009). Two hypotheses were tested: (1) that elevated concentrations of metals in water and sediment that impair aquatic invertebrate communities were restricted to historically mined catchments (a presumption made in many biomonitoring studies), and (2) that water and sediment toxicity to aquatic insect communities are controlled by hydrothermal alteration and mineral deposit type. This approach facilitated the development of lithology specific geochemical and ecological baselines. These baselines could be used to better understand the role that geologic sources of metals play in structuring aquatic ecosystems in mineralized regions and allow for a more quantitative assessment of the effect of historical mining on aquatic ecosystems.

Data, information, and risk evaluation:

Data needs: The goal was to sample a large number of catchments underlain by a simple geology. Enough samples from each geologic classification were needed to develop statistically robust geochemical and ecological baselines for each lithology. Fewer sites were sampled for invertebrates because the sampling effort is greater than for geochemistry and it was necessary to target streams with similar habitat qualities to limit the number of confounding variables influencing the data (Schmidt et al. 2009, 2012).

Brief sampling plan: Hydrothermal alteration was mapped and characterized across the study area primarily using mineral maps derived from analysis of Advanced Spaceborne Thermal Emission and Reflection Radiometer (ASTER) remote-sensing data (Rockwell 2013). Databases from the State of Colorado (from the United States Forest Service Abandoned Mine Land

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Inventory Project, http://geosurvey.state.co.us/water/Abandoned%20Mine%20Land/Pages/UnitedStatesForestServiceAbandonedMineLandInventoryProject.aspx, accessed 9/27/13) and the Federal Government (USGS Mineral Resources On-Line Spatial Data, http://tin.er.usgs.gov/mrds/, accessed 9/27/13) were used to determine deposit type and disturbance by mining (Church et al. 2009; Schmidt et al. 2009, 2012). Site selection controlled for elevation, precipitation, basin area, and discharge in such a way that these factors were poorly related with ecological and geochemical endpoints (Schmidt et al. 2010). Five unique lithologies were targeted (Schmidt et al. 2009, 2012). Sampling occurred during summers of 2004-2007. Approximately 10% of locations were sampled annually to capture annual variability in geochemistry and invertebrate community character. Greater than 250 sediment (wet sieved to <2 mm on site) and water (filtered and unfiltered) samples were collected and analyzed for major anions and cations (Church et al. 2009; Schmidt et al. 2009, 2012). Invertebrate communities were collected from approximately half these sites and analyzed to determine differences in diversity and density (number of individuals per 0.1 m^2) (Schmidt et al. 2009, 2012).

Conclusions:

Hypothesis (1) That elevated concentrations of metals in water and sediment that impair aquatic invertebrate communities were restricted to historically mined catchments.

Findings:

1. Elevated concentrations of metals in water and sediment were not confined to mined catchments.
2. Some unmined catchments produce sediment and water so toxic as to exclude aquatic life.

Hypothesis (2) That hydrothermal alteration and mineral deposit type interact with historical mining disturbance to control aquatic community character.

Findings:

1. Hydrothermal alteration and ore deposit type strongly affect the extent to which historical mining resulted in the elevation of metals in water and sediment.
2. Unmined quartz-sericite-pyrite alteration and Climax-style porphyry Cu-Mo deposits produced water and sediment toxic to aquatic life.
3. Propylitic altered polymetallic veins released less metal and acid and supported a more robust aquatic community as compared to other alteration styles or ore deposit types.

Strengths of approach: The approach can develop quantitative baselines for geochemical and ecological characteristics of streams derived from local conditions. Because the site classification scheme is related to how metals are distributed in the environment, there is less variability in the geochemical and biological data than is typically observed when classifying sites by the presence/absence of mining disturbance alone.

Weakness of approach: The approach requires technical expertise about geologic mapping and remote sensing technologies. Because the remote sensing technology cannot classify the mineralogy of vegetated parts of the catchment or below ground, there is a limit to how quantitative the remote sensing data can be. In order to achieve a statistically robust study design, the number of sampling sites can be high along with the cost of sampling.

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Lessons learned:

1. Using upstream/downstream comparisons to measure the ecological effect of a mine release may not be appropriate for all situations. Lithology above and below the mine may be different such that baseline for the mined lithology could be substantially higher in metal and acid producing minerals as compared to the lithology upstream of the mine.
2. Sampling analog catchments (catchments unmined but with similar lithologic characteristics) can provide a baseline to which comparisons may be made, which is quantitatively more accurate than other assessment approaches.
3. Invertebrate communities differed substantially among alteration styles, ore deposit type, and the presence/absence of mining disturbance.
4. Some unmined catchments never supported aquatic life, suggesting in some cases pre-mining geochemical baselines could have precluded aquatic communities.
5. Baselines for some lithologies had sediment and water metal concentrations well below aquatic life standards. Invertebrate communities from these locations were more diverse and abundant than in catchments where water and sediment metal concentrations were near aquatic life criteria. As a result, utilizing aquatic life standards as reclamation targets may not result in an invertebrate community as diverse and abundant as that observed at other undisturbed catchments.

Recommendations: The influence of lithology ought to be taken into account in ecological assessments of the effect of mining on aquatic ecosystems. Employing least-disturbed (Stoddard et al. 2006) or reference locations or upstream/downstream comparisons can result in over-estimating the effect of mining on the ecosystem by not accounting for the elevated metal content of mineralized rocks associated with mined locations. Analog basins are recommended to develop geochemical and biological pre-mining baselines (Runnels et al. 1992).

Bibliography:

Chronic, J., and H. Chronic. 1972. Prairie Peak and Plateau. A guide to the geology of Colorado. Colorado Geological Survey Bulletin 32, Colorado Geological Survey, Denver, Colorado, USA.

Church, S. E., Fey, D. L., Klein, T. L., Schmidt, T. S., Wanty, R. B., DeWitt, E. H., Rockwell, B. W., and C.A. San Juan. 2009. Environmental effects of hydrothermal alteration and historical mining on water and sediment quality in central Colorado. Pages 85-95 *in* R.M.T. Webb and D.J. Semmens, eds. Planning for an uncertain future—Monitoring, integration, and adaptation. Proceedings of the Third Interagency Conference on Research in the Watersheds: U.S. Geological Survey. Scientific Investigations Report 2009-5049. http://pubs.usgs.gov/sir/2009/5049/, accessed 9/27/13.

Clements, W. H., Carlisle, D. M., Lazorchak, J. M., and P.C. Johnson. 2000. Heavy metals structure benthic communities in Colorado mountain streams. Ecological Applications v. 10, p. 626-638.

Cox, D.P., and Singer, D.A., eds., 1986, Mineral deposit models: U.S. Geological Survey Bulletin 1693, 379 p., http://pubs.usgs.gov/bul/b1693/, accessed 9/27/13.

Robb, L. 2005. Introduction to ore-forming processes. Blackwell Publishing, Malden, Massachusetts. USA.

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Rockwell, B.W. 2013. Comparative mineral mapping in the Colorado Mineral Belt using AVIRIS and ASTER remote sensing data: U.S. Geological Survey Scientific Investigations Map 3256, 8 p. pamphlet, 1 map sheet, scale 1:150,000, http://pubs.usgs.gov/sim/3256/, accessed 9/27/13.

Runnels, D. D., Shepherd, T. A., Angino, E. E. 1992. Metals in water, determining natural background concentrations in mineralized areas. Environmental Science and Technology. 26: 2316-2323.

Schmidt, T.S., Church, S.E., Clements, W.H., Mitchell, K., Fey, D.L., Wanty, R.B., Verplanck, P.L., San Juan, C.A., DeWitt, E., Rockwell, B., and Klein, T.L. 2009. Geologic processes influence the effect of mining on aquatic ecosystems. Pages 195-205, *in* R.M.T. Webb and D.J. Semmens, editors. Planning for an uncertain future—Monitoring, integration, and adaptation. Proceedings of the Third Interagency Conference on Research in the Watersheds: U.S. Geological Survey. Scientific Investigations Report 2009-5049. http://pubs.usgs.gov/sir/2009/5049/, accessed 9/27/13.

Schmidt, T.S., Clements, W.H., Wanty, R.B., Verplanck, P.L., Church, S.E., San Juan, C.A., Fey, D.L., Rockwell, B.W., DeWitt, E.H., and Klein, T.L. 2010. Development of a new toxic-unit model for the bioassessment of metals in streams. Environmental Toxicology and Chemistry. 29:2432-2442.

Schmidt, T.S., Clements, W.H., Wanty, R.B., Verplanck, P.L., Church, S.E., San Juan, C.A., Fey, D.L., Rockwell, B.W., DeWitt, E.H., and Klein, T.L. 2012. Geologic processes influence the effects of mining on aquatic ecosystems. Ecological Applications. 22:870–879.

Stoddard, J. L., Larsen, D. P., Hawkins, C. P., Johnson, R. K., Norris, R. H. 2006. Setting expectations for the ecological condition of streams: The concept of reference condition. Ecological Applications. 16:1267-1276.

Tweto, O., and P. K. Sims. 1963. Precambrian ancestry of the Colorado mineral belt. Geological Society of America Bulletin 74:991-1014.

Wanty, R. B., Verplanck, P. L., San Juan, C. A., Church, S, E., Schmidt, T. S., Fey D. L., DeWitt, E. H., and T.L. Klein. 2009. Geochemistry of surface water in alpine catchments in central Colorado, USA: Resolving host-rock effects at different spatial scales. Applied Geochemistry 24:600-610.

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DEVELOPING A GEOCHEMICAL CHARACTERIZATION PROGRAM TO GUIDE RECLAMATION DECISIONS

Zortman-Landusky Mine Reclamation, Phillips County, Montana

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Purpose of case study: Use of geochemistry data to develop and support reclamation of a mine site following bankruptcy of a mining company.

Brief site description and history: The adjacent Zortman and Landusky mines are located in the Little Rocky Mountains, Phillips County, Montana (U.S. Department of the Interior, Bureau of Land Management and State of Montana Department of Environmental Quality, Hard Rock Bureau 1996; U.S. Department of the Interior, U.S. Bureau of Land Management and State of Montana Department of Environmental Quality (DEQ) 2001). The mines produced gold and silver from a mineralized syenite intrusion. Although mining in the area began over 100 years ago, the most extensive production was from open pit mining and heap leach cyanide processing that occurred from 1977 until 1998 when the operator declared bankruptcy and the site was taken over by the USBLM and the Montana Department of Environmental Quality (MDEQ).

The Little Rocky Mountains are an elliptical dome formed by the emplacement of Tertiary syenite porphyries into older metamorphic and sedimentary rocks ranging in age from Precambrian to Cretaceous. The syenite porphyry consists of potassium feldspar and sodic plagioclase, with lesser amounts of quartz and mafic minerals, principally hornblende.

The emplacement of the syenite stocks domed up the surrounding sedimentary rocks. Faulting and jointing associated with the emplacement of the syenite had a major influence on the localization of mineral deposits, as well as the development of acid rock drainage (both pre-mining and mining related). Following emplacement of the syenite porphyries, a lower temperature hydrothermal system channeled along the structural trends in the porphyry and caused widespread alteration and emplacement of gold, silver, and associated minerals such as pyrite.

In the 1970s there was renewed interest in mining in the Little Rockies. In 1979 the State of Montana approved an Environmental Impact Statement for large scale open pit heap leach mining by both Zortman Mining Company (Gulf Resources) and Landusky Mining Company (Wharf Resources). In 1981 Pegasus Gold Corporation became the operator of both mines following the merger of Pegasus Limited and Arco Gold Corp. The USBLM and the Montana Department of State Lands, now Montana Department of Environmental Quality, have coordinated on administration of the mine's Plan of Operations since 1981. Between 1979 and 1989 there were 11 amendments to the approved Plan of Operations generally expanding operations at both minesites. The initial mine planning and permitting envisioned only mining oxide ores. The minimal work done on the sulfide mineralization present deeper in the deposits suggested there was potential for acid rock drainage only if mining proceeded to the deeper portions of the deposit.

The extensive fracturing discussed above was an important factor influencing why the deposits were economic, as they required only blasting and leaching (no primary or secondary crushing was needed). This extensive fracturing was also a contributing factor in both the development of sedimentary paleo-ferricrete deposits and the recent decline in Swift Gulch water

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quality (see below). Gold values ranged from 0.75 to 0.96 grams per tonne. Much of the early mining at both Zortman and Landusky focused on the oxidized portion of the ore deposits. The early emphasis of mining of oxide ore and the relatively low percentage of pyrite (< 0.3%) in unoxidized ore led early workers to believe that the deposits had a low potential to generate acid rock drainage (Williams et al. 2009). The initial realization that acid rock drainage was an issue at the mine sites occurred in the early 1990s, when decreasing pH values in the headwaters of several drainages were noted. Initial attempts to mitigate acid rock drainage via lime amendments of waste rock were ineffective. In January 1998, Pegasus Gold Corporation filed for Chapter 11 bankruptcy.

Problem definition: Following the Pegasus bankruptcy there were substantial shortfalls in the bond coverage needed to perform reclamation. This shortfall meant characterizing material for reclamation would require innovative technologies and methods. A geochemical evaluation was undertaken with two primary objectives: first, to identify the location, extent, and probable current and future contaminant loads from the various facilities on the sites to prioritize for reclamation efforts (e.g., high priority, medium priority, low priority); second, to identify candidate materials on site for use as construction materials in cover and remediation purposes.

Data needs: The geochemical characterization program comprised an assessment of historical information, a field reconnaissance survey, a drilling campaign, and laboratory testing. Confirmation laboratory testwork conducted included paste pH and paste conductivity measurements on the as-received 'fines,' modified acid base accounting (ABA) tests, inorganic carbon and leach extraction analyses, forward acid titration (of Acid Buffering Characteristic Curves), multi-element ICP, net acid generation (NAG) tests, and sieve analyses.

Conclusions and lessons learned: The characterization program clearly showed that the rock with total sulfur contents greater than 0.2% had become acidic when weathered, with field paste pH values less than 5.0. Very little neutralization or buffering capacity was available in the associated rock types, other than what had been added to the heap leach pads in the form of lime and/or caustic soda. Acid Potential or AP values were relatively low (<25 Kg/Tonne $CaCO_3$; however, Neutralization Potential (NP) values were also negligible (<20 Kg/Tonne $CaCO_3$), with the result that any acidity production from oxidation of pyrite was immediately translated into runoff with low pH and high dissolved metal concentrations.

The results were used to map the ARD/Metal Leaching (ML) potential for various facilities and to provide a mass-loading evaluation of these facilities. Reclamation efforts have been focused on reducing infiltration and, therefore, contaminant loading from the various waste facilities, while relying on and optimizing water management, collection and treatment. The only drainage on Landusky that did not have an existing seepage collection system at the time of these evaluations was Swift Gulch, where a semi-portable water treatment plant is now operational.

Bibliography:

U.S. Department of the Interior, Bureau of Land Management and State of Montana Department of Environmental Quality, Hard Rock Bureau, 1996. Final Environmental Impact Statement, Zortman and Landusky Mines Reclamation Plan Modifications and Mine Life Extensions. Report prepared March, 1996.

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U.S. Department of the Interior, U.S. Bureau of Land Management and State of Montana Department of Environmental Quality (DEQ), 2001. Final Supplemental Environmental Impact Statement for Reclamation of the Zortman and Landusky Mines, Phillips County, Montana. December 2001.

Williams, R.D., Shaw, S., Jepson, W., Gammons, C. Kill Eagle, J. 2009. Zortman-Landusky: Challenges in a decade of closure. In Proceedings of the 8th International Conference on Acid Rock Drainage (ICARD), Skelleftea, Sweden.

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EVALUATING THE EXTENT OF URANIUM CONTAMINATION AND POTENTIAL HUMAN EXPOSURE

Blue Creek, Spokane Indian Reservation, Washington

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Purpose of case study: We were tasked to evaluate the extent of contamination by historical uranium (U) production in the Blue Creek watershed, the only fishery on the Spokane Indian Reservation. Fear of the downstream extent of contamination by radioactive mine waste resulted in a significant loss-of-use of valuable areas on the Spokane Indian Reservation used for substance living and tribal ceremonies.

Brief site description and history: Uranium was discovered in 1954 at the headwaters of a small tributary of Blue Creek. The deposit was formed during an Eocene intrusive event when hydrothermal circulation leached U from the intrusive rocks (zircon) and subsequently deposited U in a carbonaceous schist in a roof pendent (Barlow et al. 1958; Sheldon 1959; Becraft and Weis 1963; Nash 1977; Ludwig et al. 1981). Production of ~10M lbs U between 1956 and 1981 resulted in a large disturbed area containing radioactive mine waste (Dean et al. 1996). The mine site was cordoned off by a 6-ft chain link fence and resulted in loss-of-use by the tribe, not only of the area immediately surrounding the Midnite Mine site, but of the downstream drainage basin.

Problem definition: An extensive surface and subsurface drainage system and a permanent water-treatment plant were put in place at the mine site to remove contaminants from surface water and some groundwater (Sumioka 1991; Dean et al. 1996). Mining activity contaminated the creek bed of Blue Creek and the delta at the mouth of Blue Creek where it entered Lake Roosevelt. The tribe used the area near the mouth of Blue Creek for traditional ceremonial activities. The tribe had largely avoided these areas for a generation because of the uncertainty of the effects of radionuclide contamination on sediment, soil, water, and biota. Various Federal agencies needed to know the extent that their adjacent lands were affected by historical mining at this mine site to determine their potential liability.

Define questions and objectives: Previous studies by consultants and USEPA had failed to define the extent of the contamination in the Blue Creek drainage and delta that extended into Lake Roosevelt (Lockheed Martin 2005). The National Park Service possibly had liability for damages if the mine tailings could be traced off the reservation into Lake Roosevelt National Recreation area.

Conceptual model: The source of contamination was not in question. The fundamental question was to what extent had U contamination left the mine site and what levels of human activity could be safely carried out in the Blue Creek drainage, particularly at the mouth of the creek where traditional tribal ceremonies were held.

Data, information, and risk evaluation: Effects on aquatic life had been evaluated by the U.S. Fish and Wildlife Service (USFWS) and by ecologists from local universities. Extensive water-

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quality data were available from the USGS, USEPA, and Spokane Indian Tribe consultants. Risk evaluation was ultimately the responsibility of Federal agencies and the Spokane Indian Tribe.

Costs and consequences of not sampling and monitoring: Continued loss-of-use of a significant portion of the Spokane Indian Reservation by tribal members was the rationale for new sampling.

Data needs: Sampling of pre-mining sediment in traps in Blue Creek and in the Blue Creek delta was needed to show the extent of contamination from mining activities. ^{137}Cs data, water management records for Lake Roosevelt and historical records of production at the Midnite mine were used to define the temporal framework. Detailed geochemical data were used in conjunction with the established time lines to determine the nature and extent of contamination by the Midnite mine. The U mining signature was then separated from the environmental signature of other regional mining activities.

Brief sampling and monitoring plan: Complete sediment cores were taken in natural sediment traps created by the mining access road in Blue Creek and in the delta where it entered Lake Roosevelt (Church et al. 2008). Surface sediment samples were used to evaluate the current geochemical baseline and establish the extent of contamination today.

Pilot study: Sediment data on bulk samples collected by USEPA demonstrated the need for more detailed sediment data. Minus-80-mesh sediment samples were collected for comparison with previously collected samples from both the USGS and National Uranium Resource Evaluation (NURE) geochemical databases and sampling programs that occurred in the mid-1970s (Otton 1976; Ciminesi 1979). Bulk sediment samples collected by USEPA were inadequate because they contained large amounts of mineral dilutants in the form of coarse-grained potassium-feldspar phenocrysts (1x1x1.5 cm) and quartz derived from quartz monzonite, the major rock unit in the watershed. These large mineral fragments were not ingested by aquatic life and do not have active exchange sites where toxic metals may accumulate.

Analyze and manage data: Database available with publication (Church et al. 2008).

Involvement of stakeholders: Coordination with the Spokane Indian tribe was managed by the Natural Resource Damage Assessment and Restoration (NRDAR) oversight committee, which had representatives from all the Federal agencies having land-management interests at the site. A tribal representative was present during all sampling activities.

Decisions made: Data were sufficient to take the NRDAR contamination case forward.

Conclusions: Sediment geochemistry showed that pre-mining U concentrations in the Midnite Mine tributary immediately downstream of the mine site were strongly elevated relative to the crustal abundance of U (2.3 ppm). ^{137}Cs data, collected at 5-cm to 10-cm intervals depending upon the median grain size, from cores in Blue Creek (Church et al. 2008) and public records of production at the Midnite Mine site provided age control to document timelines in the sediment from the core immediately downstream from the mine site. Contamination of the sediment by U during the mining period is documented from the Midnite Mine in Blue Creek and into the Blue

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Creek delta. During the period of active mining (1956–1981), enrichment of base metals also occurred in the sediment of Blue Creek. These base metals were enriched in the sediment of Blue Creek delta by as much as 4-times the concentration of those same metals prior to mining. Cadmium concentrations were elevated by a factor of 10 and U by factors of 16 to 55 times pre-mining geochemical background determined upstream of the mine site. Post-mining metal concentrations in sediment are lower than during the mining period, but remain elevated relative to pre-mining geochemical background. Uranium contamination in surface sediment in the delta of Blue Creek cove extends at least 500 meters downstream from the mouth of Blue Creek, into Lake Roosevelt. The boundary between lands administered by the National Park Service (USNPS) and the Spokane Indian Tribe is defined as the 1,290-ft contour. Comparisons of the pre-mining geochemical background to sediment sampled during the period the mine was in operation, and to the sediment data from the post-mining period, are used to delineate the extent of contaminated sediment in Blue Creek cove along the thalweg of Blue Creek into Lake Roosevelt. Much of the base-metal contamination in the delta sediment was shown to have been derived from outside of the Blue Creek drainage and was not from the U mining activity (Church et al. 2008).

Were the data quality objectives met? Yes

Costs and benefits: The study was designed to (1) determine pre-mining geochemical baseline, (2) determine the extent and time-frames for geochemical contamination attributable to U production at the Midnite Mine, and (3) provide geochemical and radiologic data to determine risk of human exposure. These objectives were met in a timely fashion—the NRDAR case has proceeded to the next phase.

Lessons learned: Time-stratigraphic data derived using a multi-pronged approach provided useful constraints on the extent of contamination with time to limit the NRDAR case to contamination on the Spokane Indian Reservation.

Recommendations: U contamination from mining has been demonstrated. Much of the contaminants in sediment have been buried in sediment along the underwater trace of Blue Creek in the delta in Lake Roosevelt. Subsequent analysis of the data by the NRDAR risk-assessment team determined that fishing activities in the reservoir from the shoreline do not constitute a significant human-health risk on the basis of current radiometric data from sediment.

Bibliography:

Barlow, J.L., Hetand, D.L., Papulak, M.S., and Van Alstine, C.L., 1958, Preliminary report on uranium deposits of the Spokane Indian Reservation, Stevens County, Washington: U.S. Atomic Energy Commission RME-2067, Part I, 56 p.

Becraft, G.E., and Weis, P.L., 1963, Geology and mineral deposits of the Turtle Lake quadrangle, Washington: U.S. Geological Survey Bulletin 1131, 73 p.

Church, S.E., Kirschner, F.K., Choate, L.M., Lamothe, P.J., Budahn, J.R., and Brown, Z.A., 2008, Determination of premining geochemical background, Blue Creek Drainage, Stevens County, Washington: U.S. Geological Survey Scientific 9/27/13 2013.

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Ciminesi, F.J., 1979, Hydrogeochemical and stream sediment reconnaissance raw data report for Sandpoint NYMS quadrangle, Washington, Idaho, and Montana: Bendix Field Engineering Report GJBX 70(79), 18 p., 2 appendices.

Dean, N.E., Boldt, C.M.K., Schultz, L.E., Nilsen, D.N., Isaacson, A.E., Williams, B.C., Kirschner, F.E., Moore, B.W., Stroud, W.P., and Peters, D.C., 1996, Midnite Mine summary report: U.S. Bureau of Mines Report of Investigations 9604, 18 p.

Lockheed Martin, 2005, Final report Midnite Mine site ecological risk assessment Wellpinit, Washington: Report prepared for U.S. EPA, contract No. EP-C-04-032, 1,513 p. www.epa.gov/region10/pdf/sites/midnite_mine/ecological_risk_assessment_sept2005.pdf , accessed 9/27/13.

Ludwig, K.R., Nash, J.T., and Naeser, C.W., 1981, U-Pb isotope systematics and age of uranium mineralization, Midnite Mine, Washington: Economic Geology, v. 76, p. 89–110.

Nash, J.T., 1977, Geology of the Midnite uranium mine area, Washington—Maps, description, and interpretation: U.S. Geological Survey Open-File Report 77-592, 39 p.

Otton, J.K., 1976, Uranium and trace element data on stream sediments in the Midnite Mine area, Stevens County, eastern Washington: U.S. Geological Survey Open-File Report 76-447, 18 p.

Sheldon, R.F., 1959, Midnite Mine geology and development: Mining Engineering, v. 11, p. 531–534.

Sumioka, S.S., 1991, Quality of water in an inactive uranium mine and its effects on the quality of water in Blue Creek, Stevens County, Washington, 1984: U.S. Geological Survey Water-Resources Investigations Report 89-4110, 62 p.

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EVALUATION OF STREAM-SEDIMENT PORE-WATER SAMPLING AND CHARACTERIZATION METHODS

Ely Copper Mine Superfund Site, Vermont

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Purpose of case study: The purpose of this case study is to evaluate different strategies for the sampling and characterization of pore water in stream sediments to assess their toxicity. The equilibrium-partitioning sediment benchmark approach for assessing the toxicity of sediments is based on the assumption that toxicity is due to the effects of pore water in equilibrium with the sediment rather than from the sediment itself (Hansen et al. 2005). To evaluate this approach for assessing sediment toxicity at the Ely Copper Mine Superfund site, samples of sediment and pore water were collected for chemical analysis and laboratory toxicity testing (Seal et al. 2010). Knowledge of the chemical characteristics of pore water is also useful for evaluating the migration of contaminants in the hyporheic zone in gaining or losing reaches of streams. Three approaches were compared to insure representative sampling and characterization of pore-water chemistry at the Ely site.

Brief site description and history: The Ely Mine and its area of downstream impact constitute the Ely Copper Mine Superfund site. The site was placed on the USEPA National Priorities List (NPL) in 2001. The mine produced copper from 1821 to 1905 using underground mining methods from a tubular-shaped massive sulfide ore body that plunged to the northeast. Hand-sorted ore was roasted prior to smelting onsite. During World War I, an attempt was made to process dump material using a flotation circuit. Solid-waste material at the site includes sulfidic waste rock and minor tailings, beds of roasted ore, and slag (Figure A5.9). The mine site is drained by Ely Brook, which is acidic for most of its reach. Ely Brook discharges into Schoolhouse Brook, which flows 3.3 kilometers and discharges into the Ompompanoosuc River (Seal et al. 2010).

Problem definition: The sampling of pore water in stream sediment presents a number of challenges largely due to the limited extents of calmer depositional environments in higher energy streams. Fine-grained sediments are commonly limited in both areal extent and in thickness. Sampling using a push point with a peristaltic pump risks contamination of pore water with drawdown of overlying stream water if pumping rates are too high, especially in coarse sediments. Likewise, laboratory centrifugation or decantation of sediment risks contamination with overlying stream water during sampling as the bottle is being filled with sediment and removed from the stream.

Data needs: Data needs include the standard set of water-quality parameters. Measurements of unstable parameters such as pH, specific conductance, dissolved oxygen, and Eh were collected at the time of sampling—in the field for the push-point sampling, described below, or in the laboratory during extraction of waters with a centrifuge or gravity draining. Samples were analyzed for major ions and trace elements by the USGS Analytical Chemistry Services Group in

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Denver, CO. Major ions and trace elements were analyzed using ICP-AES and ICP-MS. Major anions were analyzed using ion chromatography, and alkalinity was determined by titration. Samples for trace elements and major ions included a less-than-0.45-µm filtered (dissolved) sample and an unfiltered (total) sample. Samples were analyzed for nutrients and dissolved organic carbon by the USGS National Water-Quality Laboratory in Denver, CO. A complete summary of the data used in this case study can be found in Seal et al. (2010).

Brief sampling plan: This case study is part of a larger study that was interested in comparing the aquatic habitat of epifauna found in riffle settings and that of infauna living in finer grained sediments in depositional settings of nearby pools (Seal et al. 2010). The sampling strategy described here targeted lower energy depositional settings (pools) in the small, high gradient streams within the watershed surrounding the Ely Mine. Samples were collected from undisturbed, continuously wetted, depositional zones in the stream channel. Three different approaches for sampling and characterizing stream-sediment pore waters were compared to evaluate representativeness:

1. Pore water was sampled using a push-point sampler connected to a peristaltic pump (Zimmerman et al. 2005). The sampler is a 6.4-mm stainless steel tube with a machined point and a 4-cm-long slotted interval near the tip. The narrow diameter minimizes disturbance of the sediment. The sampler was inserted to a depth of approximately 15 cm. Specific conductance of water was used to monitor gross chemical differences between surface water and pore water during sampling and to identify the presence of drawdown.
2. Sediment with pore water was collected in a bottle and centrifuged in the laboratory prior to using the water for toxicity tests. The top 10 cm of streambed sediment were sampled to obtain only the most recently deposited material. Composited samples were collected from 5 to 10 representative subsamples. Centrifugation in the laboratory was done at 68,647 m/s^2 (7,000 *g*) for 15 minutes in 250-mL conical polypropylene bottles.
3. Sediment with pore water was collected in a 2-L bottle, topped off with stream water, and "aged" or "equilibrated" in the laboratory for 28 days at room temperature prior to being collected for analysis through gravity draining. The 28-day incubation period was selected to coincide with the duration of other toxicity testing experiments being done for the larger study.

The representativeness of the three approaches described above was evaluated by comparing water-chemistry results with biological condition metrics derived from benthic macroinvertebrate data collected in depositional settings (Seal et al. 2010).

Analyze data: At the Ely Copper Mine Superfund site, Cu is by far the dominant contaminant in terms of exceedance of aquatic water-quality criteria in both surface water and pore water, followed by Cd and Zn, and locally Al and Fe. Collectively, the dissolved concentrations of Cu in the pore-water samples range from <0.5 to 2,140 µg/L. The methods of characterization agree well with strong correlations between the Cu concentrations of the three pore-water types (r^2 = 0.99) (Figure A5.10). Despite the strong correlations, comparison of the three methods indicates that the push-point samples invariably yield the lowest concentrations of dissolved constituents of the three methods. Of the 10 sample sites investigated, the laboratory equilibrated samples yielded the highest concentrations in seven samples, and the centrifuged samples yielded the highest concentrations in three samples (Figure A5.11).

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The three different types of pore-water chemical data were compared to biological condition metrics that characterized the macroinvertebrate community data collected in depositional zones. These macroinvertebrate data, designated as depositional-targeted habitat (DTH) data, were used to evaluate the sediment infaunal community structure, specifically metrics of organism abundance and the number of taxa present (i.e., richness). Variations in these metrics were compared to the ambient pore-water conditions. The DTH refers to the infaunal macroinvertebrate community living with fine-grain sediments found in low energy environments, such as pools, whereas the riffle-targeted habitat (RTH) refers to the epifaunal macroinvertebrates living in high-energy environments. The pore-water chemistry data were evaluated in terms of a hazard index, which is the sum of individual hazard quotients (the dissolved concentration of an element divided by its hardness-based chronic aquatic ecosystem criterion, or criterion continuous concentration, CCC) for the metals Cd, Cu, Ni, Pb, and Zn. A hazard index above 1 indicates problematic pore-water concentrations of these metals. The results of this comparison are shown in Figures A5.12a and A5.12b. Figure A5.12a shows the variation of hazard index values for the three pore-water samples with DTH abundance data. Figure A5.12b shows the same hazard index values varying with the number of DTH taxa. In general, all three pore-water sampling methods yield similar results, showing a greater abundance of organisms and more taxa present at sites with hazard indices below 1. However, the centrifuged sample associated with the highest DTH abundance and richness values is notably anomalous with a hazard index that is roughly 10 times the chronic limit.

Conclusions: All three pore-water sampling and characterization methods compared in this case study yielded similar results in terms of predicted toxicity in the stream depositional environment surrounding the Ely mine. Furthermore, the results were successful in predicting pore-water toxicity as reflected by the macroinvertebrate community characteristics at each of the sample sites. These similarities suggest that none of these methods are more prone to pore-water contamination from the overlying surface water than the others. Of the three methods described, the 28-day “aged” or “equilibrated” pore-water method was the simplest in terms of field or laboratory equipment requirements.

Bibliography:

Hansen, D.J., DiToro, D.M., Berry, W.J., Boothman, W.S., Burgess, R.M., Ankley, G.T., Mount, D.R., and McGrath, J.A., 2005, Procedures for the Derivation of Equilibrium Partitioning Sediment Benchmarks (ESBs) for the Protection of Benthic Organisms: Metal Mixtures (Cadmium, Copper, Lead, Nickel, Silver and Zinc): EPA/600/R-02/011, 121 p. (available online at www.epa.gov/nheerl/download_files/publications/metalsESB_022405.pdf, accessed 9/27/13)

Seal, R.R., II, Kiah, R.G., Piatak, N.M., Besser, J.M., Coles, J.F., Hammarstrom, J.M., Argue, D.M., Levitan, D.M., Deacon, J.R., and Ingersoll, C.G., 2010, Aquatic assessment of the Ely Copper Mine Superfund site, Vershire, Vermont: U.S. Geological Survey Scientific Investigations Report 2010–5084, 131 p. (available only online at http://pubs.usgs.gov/sir/2010/5084/, accessed 9/27/13)

Zimmerman, M.J., Massey, A.J., and Campo, K.W., 2005, Pushpoint sampling for defined spatial and temporal variations in contaminant concentrations in sediment pore water near the ground water/surface water interface: U.S. Geological Survey Scientific

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Investigations Report 2005–5036, 75 p. (available online at http://pubs.usgs.gov/sir/2005/5036/, accessed 9/27/13)

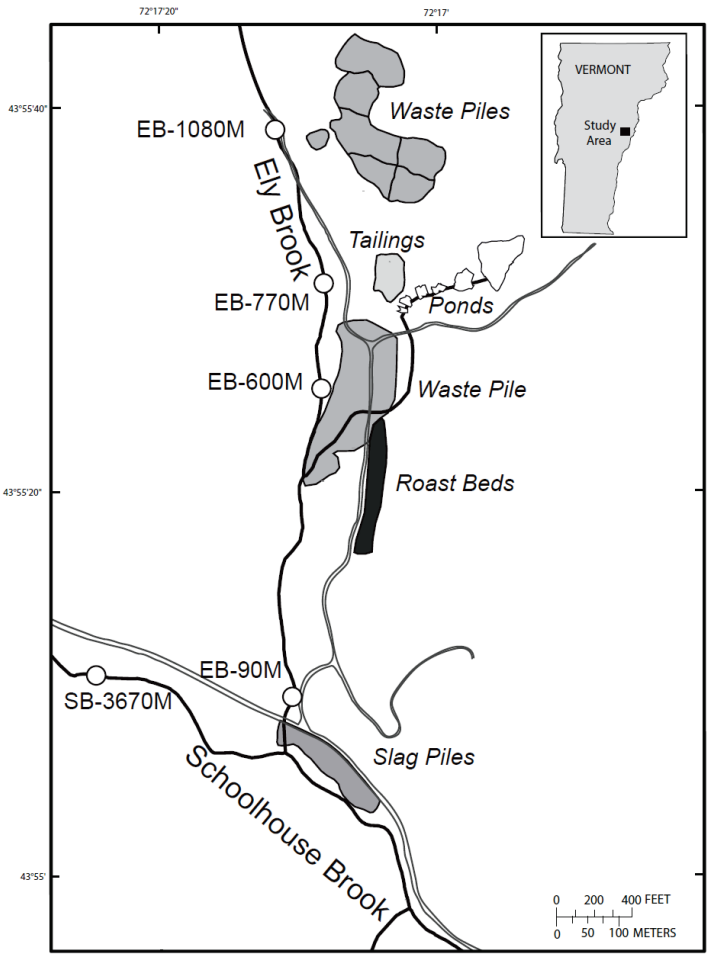


FIGURE A5.9 Map showing distribution of mine waste and sample sites at the Ely Copper Mine Superfund site, Vermont. Sample sites in Ely Brook (EB), Schoolhouse Brook (SB; not all shown), and the Ompompanoosuc River (OR; not shown) are labeled by river meter upstream of the next major confluence. For example, EB-90M is located on Ely Brook 90 m upstream of its confluence with Schoolhouse Brook. The confluence of Schoolhouse Brook with the Ompompanoosuc River is approximately located at OR-23650M.

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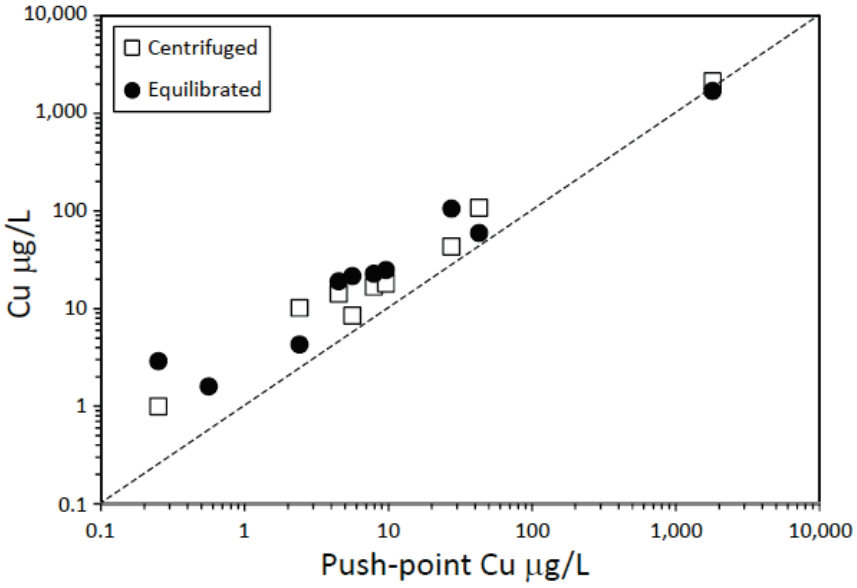


FIGURE A5.10 Comparison of copper concentrations of pore waters collected by various methods. Both the centrifuged and equilibrated samples are plotted against the push-point sample concentrations. The dashed diagonal represents perfect agreement among the sample types.

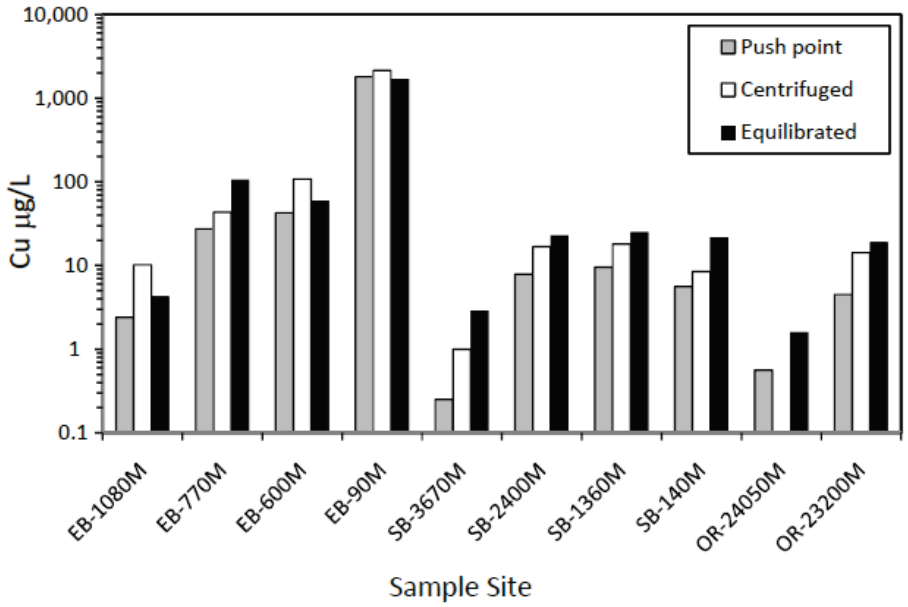


FIGURE A5.11 Comparison of dissolved copper concentrations from push-point, centrifuged, and equilibrated (aged) samples by sample site

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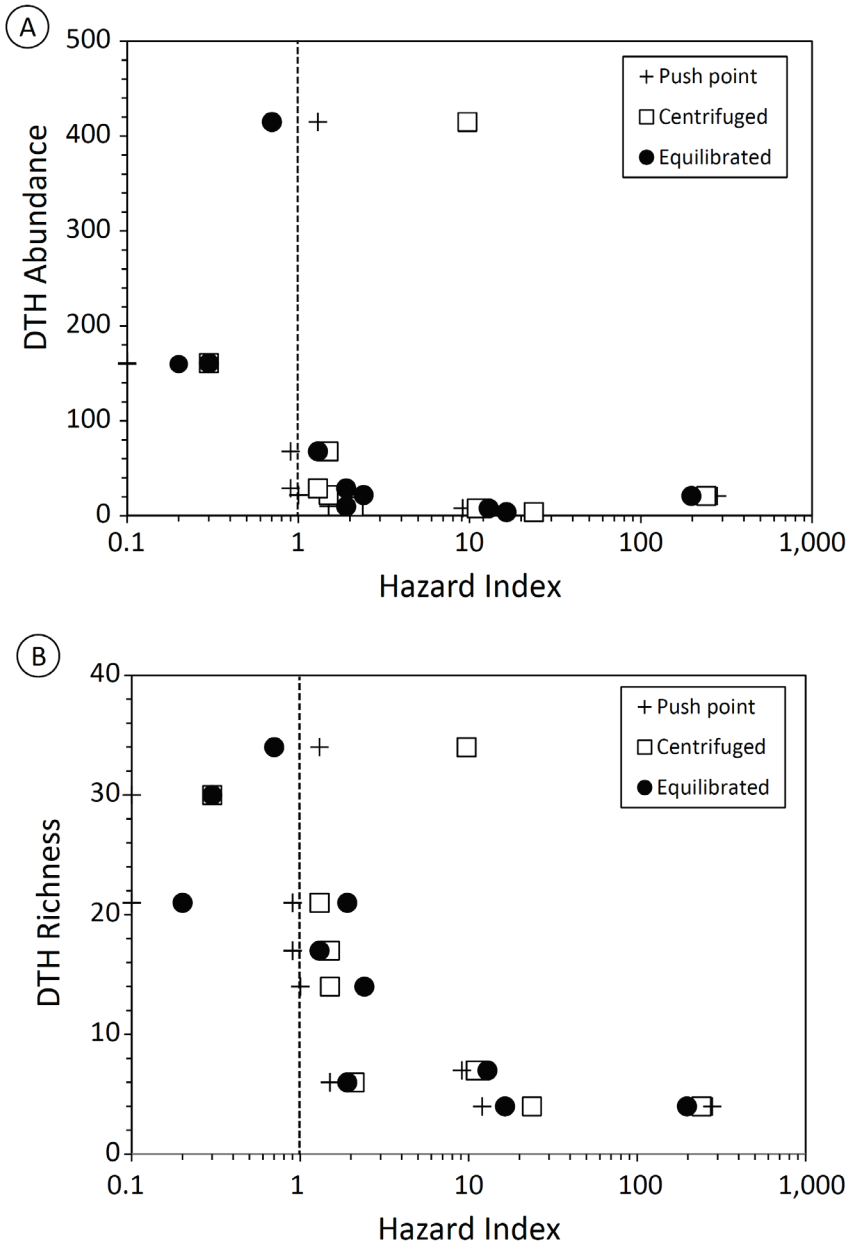


FIGURE A5.12 Variation of hazard indices of pore waters from push-point, centrifuged, and equilibrated (aged) samples with macroinvertebrate community data for depositional-targeted habitats (DTH). A. DTH abundance; B. DTH taxa richness.

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APPLYING MASS-LOADING ANALYSIS TO EXISTING DATASETS TO DEVELOP A CONCEPTUAL MODEL

St. Kevin Gulch Watershed Study Area, Colorado

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Purpose of case study: Demonstrating application of mass-loading studies in a watershed thought to have one dominant loading source.

Brief site description and history: St. Kevin Gulch is an upland catchment, originating at about 3,580 m, and draining to Tennessee Creek, which joins the Arkansas River south of the study area, near Leadville, Colorado (Figure A5.13). Bedrock in the catchment mostly includes Precambrian schist and granite (Singewald 1955). These rocks are only covered by a thin layer of soil in most places. The granite and quartzbiotite schist have little neutralizing capacity for acidic waters that come from weathering hydrothermally altered veins containing sulfide minerals. Mining in St. Kevin Gulch at the Griffin Mine occurred mostly in the years around 1915-1939, and included over 12,800 tons of ore for gold, silver, lead, and zinc (Singewald 1955). Most of the mine-waste was dumped near the mouth of the main portal to the mine and is referred to herein as the Griffin Mine waste pile. Water in St. Kevin Gulch has an ambient pH between 3 and 4, and elevated concentrations of iron, aluminum, copper, zinc, and manganese. St. Kevin Gulch degrades the water in Tennessee Creek and the Arkansas River during the high-flow periods (snowmelt runoff). Flow in the lower portions of St. Kevin Gulch is ephemeral and does not contribute to Tennessee Creek during low-flow periods.

Define questions and objectives: Determine primary locations of metal loading along St. Kevin Gulch and incorporate that information into a conceptual site model. Use existing data that were acquired using tracer injection and synoptic sampling method (Kimball et al. 2002).

Conceptual model: Waste and landscape disruption associated with previous mining activities contributes to water-quality degradation in St. Kevin Gulch. Based on the previous work at the site, springs located at the toe of the Griffin Mine waste pile are likely the largest source of metal and acidic loading to St. Kevin Gulch. However, other previously unidentified diffuse sources may also contribute substantial loads.

Costs and consequences of not sampling and monitoring: Continued seasonal impairment of the Arkansas River downstream from Tennessee Creek and St. Kevin Gulch after millions of dollars have been spent to clean up other downstream sources of mine drainage to the Arkansas River.

Data needs: Data for the mass-loading study were collected during low-flow conditions in August 1987. Twenty-eight stream samples and 10 inflow samples were collected and analyzed.

Brief sampling and monitoring plan: Mass-loading profiles were constructed for a 1.8 km reach of St. Kevin Gulch (Figure A5.13) using discharge and water-quality data. Discharge data

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were derived from the tracer-dilution method of measuring stream discharge (Kilpatrick and Cobb 1985). Water-quality samples were collected during a synoptic sampling event (all samples collected during one day) conducted during the tracer-dilution study. When used together, these techniques provide a description of the watershed that includes both discharge and concentration that were used to determine the mass loading associated with various sources of water (Kimball 1997; Kimball et al. 2002; Kimball et al. 2007).

Analyze and manage data: Database in P-Stat and Access.

Involvement of stakeholders: In 2007 the USEPA sponsored reworking of data for mass-loading analysis and development of the conceptual model.

Decisions made: Pending (2011).

Conclusions:
Were the data quality objectives met? Yes.

Costs and benefits: This study is an example of reworking previously collected data to achieve new objectives. The previous sample-collection methods were consistent with the data needs for a mass-loading analysis, but were not used originally for that purpose. The costs of reworking the old data were less than would have been needed to collect and analyze new data. As there has been no remediation in the area, changes from 1987 to 2007 are likely minimal and related to yearly climate fluctuation.

Lessons learned:

1. The greatest metal loading (aluminum, manganese, and zinc) along the study reach occurred at inflows to the stream in the vicinity of the Griffin Mine waste pile.
2. From about 20 to 50 percent of this load came into the stream not from the springs at the toe of the pile, but from groundwater entering the stream in the vicinity of the Griffin Mine waste pile.
3. Sources upstream from the Griffin Mine waste pile accounted for up to 50 percent of the copper load to the stream.
4. There were some downstream diffuse sources of loading to the stream, but these were minor sources of load in comparison to the loading that occurred in the vicinity of the Griffin Mine waste pile for manganese and zinc, but more significant sources for copper and aluminum.

Bibliography:

Kilpatrick, F.A. and E.D. Cobb, 1985. Measurement of discharge using tracers: USGS Techniques of Water-Resources Investigations Book 3 Chapter A16, U.S. Geological Survey, Denver, Colorado, 52 p. http://pubs.usgs.gov/twri/twri3-a16/pdf/TWRI_3-A16.pdf, accessed 9/27/13.

Kimball, B.A., 1997. Use of tracer injections and synoptic sampling to measure metal loading from acid mine drainage: USGS Fact Sheet FS-245-96, U.S. Geological Survey, Denver, Colorado, 4 p. http://ut.water.usgs.gov/publications/fs245-96/, accessed 9/27/13.

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Kimball, B.A., Runkel, R.L., Walton-Day, Katherine, and Bencala, K.E., 2002, Assessment of metal loads in watersheds affected by acid mine drainage by using tracer injection and synoptic sampling--Cement Creek, Colorado, USA: Applied Geochemistry, v. 17, no. 9, p. 1183-1207.

Kimball, B.A., Katherine Walton-Day, and R.L. Runkel, 2007. Quantification of metal loading by tracer injection and synoptic sampling, 1996–2000, Chapter E9. *In*: Integrated Investigations of Environmental Effects of Historical Mining in the Animas River Watershed, San Juan County, Colorado: USGS Professional Paper 1651, S.E. Church, P.B. von Guerard, and S.E. Finger (Editors). U.S. Geological Survey, Denver, Colorado, pp. 417-495. http://pubs.usgs.gov/pp/1651/downloads/Vol1_combinedChapters/vol1_chapE9.pdf, accessed 9/27/13.

Singewald, Q.D., 1955, Sugar Loaf and St. Kevin Mining Districts, Lake County, Colorado. U.S. Geological Survey Bulletin 1027-E: 299 p.

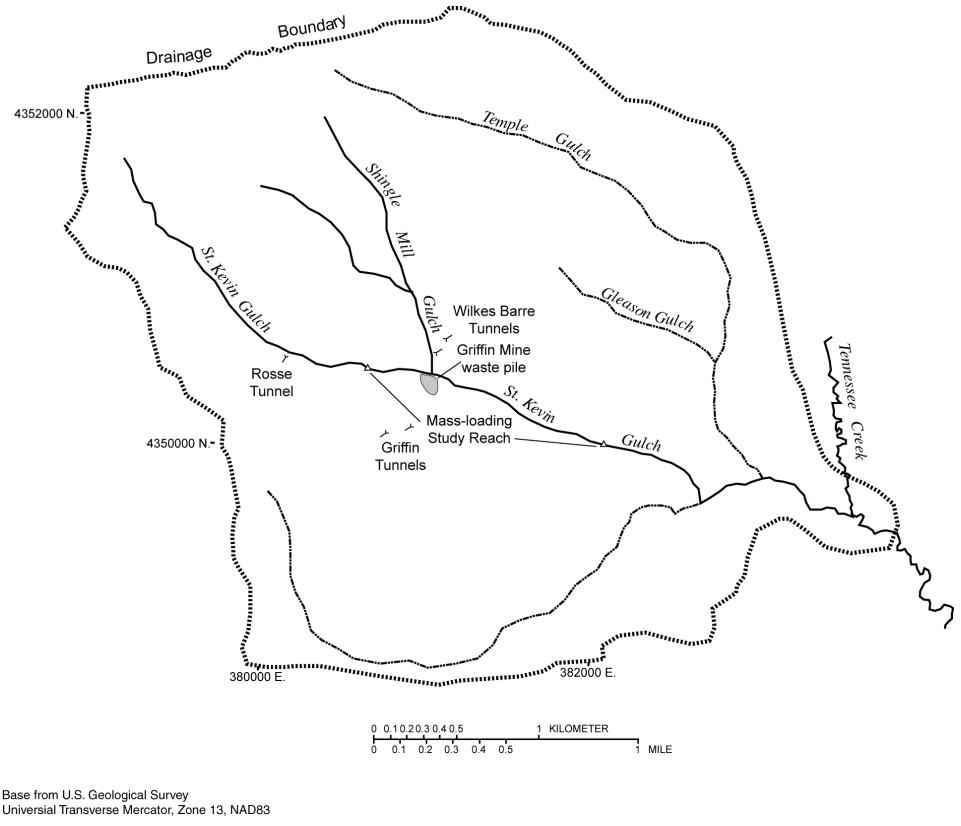


FIGURE A5.13 Location map of St. Kevin Gulch, Colorado showing location of mass-loading study reach and some mining features, including the Griffin Mine waste pile

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CHALLENGES OF QUANTIFYING METAL PARTITIONING WITHIN SOLID PHASES

An Example of Zinc in Slag from the Hegeler Zinc Smelter, Illinois

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Purpose of case study: A study of slag at an historical zinc-smelting facility in Illinois was conducted in order to assess the potential for the release of trace elements into the environment (Piatak and Seal 2010). One important aspect of this study is the partitioning of trace elements among the various solid phases. In this paper, we highlight the challenges associated with quantifying the abundances of solid phases based on X-ray powder diffraction (XRD), specifically for samples containing significant amorphous material, in this case, silicate glass. Quantitative mineralogy is then used in conjunction with mineral and glass chemistry from electron-probe microanalysis (EPMA) to calculate the amount of an element hosted by each phase. The relative weathering rate or solubility of the phases can be examined in relation to the amount of elements hosted in the phases to assess the potential for elements to be released into the environment. This paper focuses on this one aspect of the broader study published by Piatak and Seal (2010) that reported the mineralogy, mineral chemistry, major- and trace-element composition, and leachate chemistry of the Hegeler zinc slags and related these data to the release of trace elements into the environment.

Brief site description and history: The Hegeler zinc-smelting facility in Vermillion County, Illinois, was in operation from 1907 until approximately 1954 and produced Zn slab, rolled Zn products, sulfuric acid, and Cd. The resulting slag waste pile covers 24,000 m^2 and rises 16 m above grade. The slag is releasing potentially toxic trace elements (e.g., As, Cd, Pb, and Zn) into various media including groundwater, surface water, stream sediment, and nearby residential soils (from the use of slag in roadways). The site, listed on the USEPA National Priorities List (NPL) in 2005, is currently being evaluated for cleanup.

Problem definition: The objective of this work is to present a simplified method for assessing the potential for the release of trace elements from mine or mineral processing wastes into the environment. This case study on zinc slag highlights some of the challenges associated with this method. Figure A5.14 illustrates the various components of this method including empirical and calculated data, and validation.

Data needs: Several types of data are needed as input for the evaluation (Figure A5.14). The first type of data needed is an accurate estimate of the amount of each phase, including amorphous material, in the sample. The presence of small amounts of phases needs to be quantified because even trace phases can be the main host of an element. For example, the presence of even trace amounts of sphalerite can dominate the budget of zinc. The second type of data needed is detailed mineral chemistry including trace element concentrations for each phase. These two empirical inputs can be used to calculate the partitioning of trace elements in the solid phases. Lastly, published information on the weathering rates or solubilities of each phase can be used in combination with the distribution of elements in the phases to assess the potential for an element to be released into the environment. Overall, the data quality objectives are to represent

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accurately the amount of each solid phase in a sample type and determine an average concentration of elements in those phases.

Collecting and analyzing data: The first data need of quantifying crystalline and amorphous phases can be met using quantitative XRD or point counting using optical microscopy. This study uses quantitative XRD, due to the speed of the technique and recent advancements in its ability to quantify concentrations of amorphous material such as glass. Historically, determination of the accuracy of the technique has proven to be a challenge. Errors associated with the quantitative XRD results were examined by creating synthetic mixtures of known amounts of phases, including glass, with similar compositions to those found in the slag and analyzing these mixtures as unknowns (Figure A5.15). The amount and composition of the sample matrix, including the amorphous material, will influence the intensity and shapes of peaks and background in the XRD pattern; thus, the synthetic mixtures need to be similar in composition to the samples. The effect of the chemical composition of the glass on the resulting XRD pattern is illustrated in Figure A5.15B. Accurately quantifying the amount of amorphous material is especially difficult, but it can be estimated by adding a known amount of an internal standard to the sample. For the Hegeler slag samples, 10 wt % zinc oxide was homogenized with the sample and analyzed. An example of a synthetic mixture with and without zinc oxide is shown in Figure A5.15A, which also shows the effect of amorphous material on the background. The XRD pattern for samples containing the internal standard was analyzed using a Rietveld quantification method. The amount of amorphous material was determined by comparing the Rietveld-determined weight percent for the internal standard (>10 wt.%) to the amount known to be present (10 wt.%).

Quantifying phases that are present only in trace amounts can be challenging using XRD because of the high errors (usually greater than ±15 relative %) associated with the estimate. The detection limit for identifying phases using XRD is generally on the order of a few weight percent; phases can be identified in concentrations lower than this based on prior knowledge of the sample, including chemistry, and a few diagnostic XRD peaks. Some quantification software programs report phase concentrations down to hundredths of a percent, which is well below the instrumental detection limit. The main host for some trace elements may be in phases that are present below this instrumental detection limit. For example in the Hegeler slag, not all Zn is accounted for in the silicates and oxides as shown in Figure A5.16 as the difference between bulk chemistry (solid dot) and sum of host phase concentrations (bars); some Zn likely resides in the sulfides, which were identified by EPMA but are present in abundances too low to detect by XRD. If bulk chemical analyses indicate a missing fraction of an element, yet a known host phase is identified by EPMA, one option is to assume that a trace amount of a phase (e.g., 1 or 0.5 wt. %) is present to account for the element deficiency based on XRD. At Hegeler we used EPMA to identify the Zn-bearing mineral sphalerite, which was not identified using XRD.

The second data need is to determine the concentrations of major, minor, and trace elements in each phase in the samples. This need can be met using instruments such as EPMA or laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS). This study uses EPMA. One limitation of EPMA compared to LA-ICP-MS is higher detection limits; detection limits for the Hegeler study were in the hundreds of ppm range whereas LA-ICP-MS is generally reported in the ppb range. The need for this lower detection limit is arguable considering most trace elements will not be environmentally significant if they occur in such minute concentrations.

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Conclusions: Overall, the data quality objectives were generally met with some agreement between the sum of zinc in the phases compared to bulk chemical results (Figure A5.16); the R-squared value for this correlation is 0.9. This substantiates that the method may adequately describe which solid phases host this trace element in the Hegeler slag.

A lesson learned from this study was the importance of analyzing synthetic mixtures, including a glass, with compositions close to those found in the samples. This method validation step is required to overcome the challenge of accurately quantifying the abundance of crystalline and amorphous phases by XRD. Accuracy of the results can be evaluated by comparing the bulk chemistry results to the sum of the partitioning of an element in all the phases to determine if the partitioning calculations account for total element abundance. Also, the predicted potential for an element to be released into the environment can be validated by comparison to synthetic weathering experiments or groundwater or surface water chemistry.

Bibliography:

Piatak, N.M., and Seal, R.R., II, 2010, Mineralogy and the release of trace elements from slag from the Hegeler Zinc smelter, Illinois (USA): Applied Geochemistry, v. 25, p. 302-320.

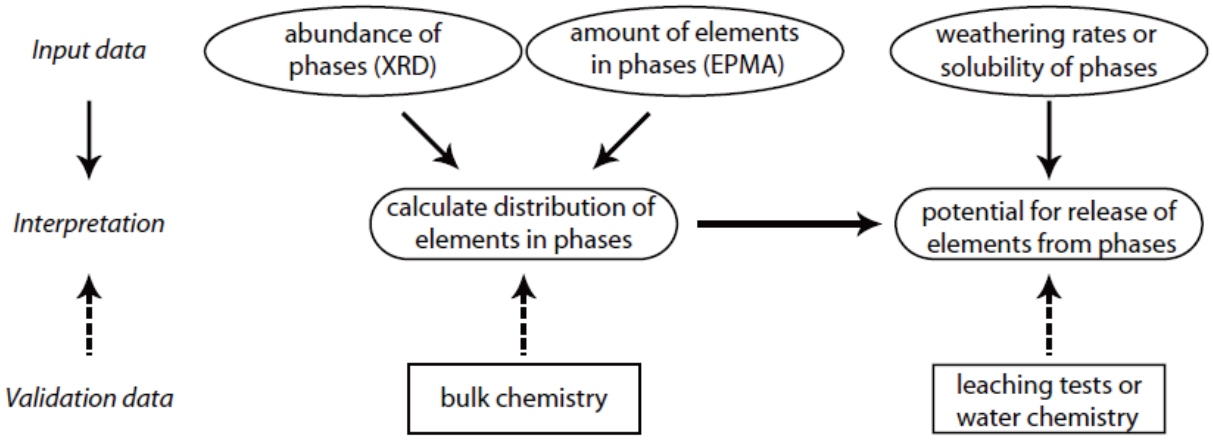


FIGURE A5.14 Diagram illustrating empirical data (input and validation data) and calculated data (interpretation) used when determining the partitioning of elements in solid phases and their potential for release in the environment

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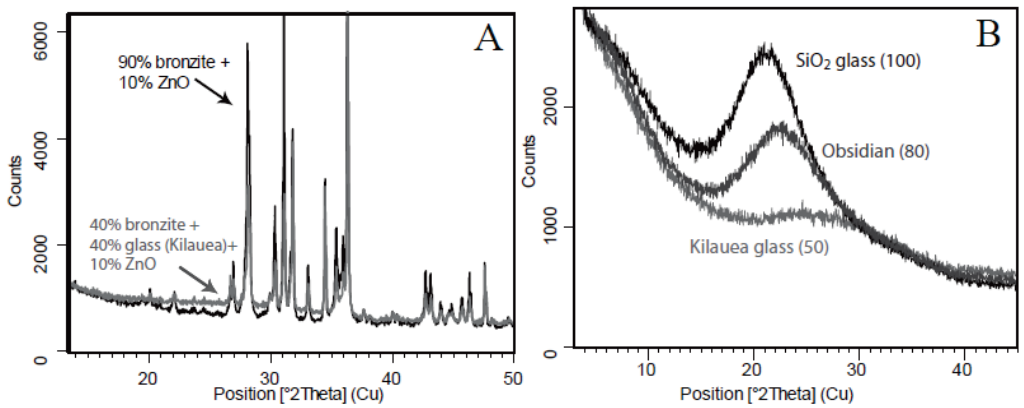


FIGURE A5-15 X-ray powder diffraction patterns for synthetic mixtures with and without a glass phase (A) and for glasses with various compositions (B). The approximate wt. % of SiO_2 in the glasses is given in parentheses.

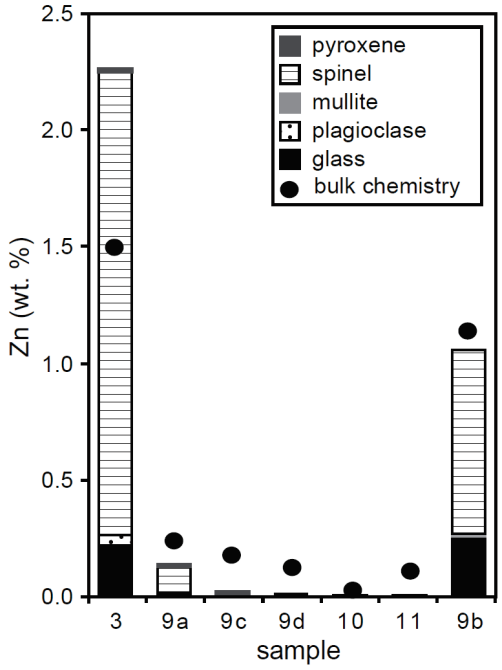


FIGURE A5-16 Bar chart of weight percent zinc calculated from quantitative mineralogy and average composition of each phase based on EPMA data. Black dots indicate zinc concentrations based on bulk chemical analysis (modified from Piatak and Seal 2010).

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PREDICTING POST-MINING WATER QUALITY USING HISTORICAL DATA AND LABORATORY TESTING

Characterizing Analogous Revett-style Cu-Ag Deposits, Montana

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Purpose of case study: Use of operational and exploration geochemistry data with mineralogy to develop a geochemical analogy between the previously unmined Rock Creek-Montanore and historically mined Troy Cu-Ag deposits of the Revett Formation. Case study emphasizes "mining" of historical data and comparison of laboratory and field-scale monitoring data to predict post-mining water quality.

Brief site description and history: The stratabound Cu-Ag deposit known as Rock Creek-Montanore hosted within the Proterozoic Revett Formation is separated by a fault into two blind, subsurface mining targets that lie beneath the Cabinet Mountain Wilderness Area in northwestern Montana. These targets are currently being permitted as the Rock Creek and Montanore mine projects, respectively. Downgradient water resources are of high quality, with very low concentrations of solutes, and therefore are sensitive to mine-related impacts. Consequences of inadequate sampling and monitoring, apart from degradation of downgradient water resources, include the inability to permit proposed mining operations and failure to comply with environmental regulations for existing mines. This analysis was developed in response to public concern from non-governmental organizations and has been made public as part of Environmental Impact Statement (EIS) and regulatory review proceedings.

The primary ore minerals at Troy and Rock-Creek Montanore, which include chalcocite (Cu_5FeS_4), bornite (Cu_2S), digenite ($Cu_{2-x}S$), and covellite (CuS), are encapsulated in quartzite that occurs in cyclic interbeds with siltite and argillite throughout the Revett Formation (Boleneus et. al. 2005). Limited archived drill core (from exploration programs that preceded wilderness area designation) is available for baseline environmental geochemistry analysis of the targeted ore zones. Rock would be mined from an ore zone of the Lower Revett with highly consistent lithologic and mineralogic characteristics that are very similar to the Upper Revett zone mined for 20 years at the Troy mine. At Troy, acid mine drainage has not developed, although some increases in mobility of trace metals including Cu, Ag, Pb, Mn, Fe, Zn, and Al have been observed in adit drainage, primarily in measurements of total rather than dissolved concentration (Enviromin 2007).

Problem definition: The prediction of future chemistry for mine drainage from the Rock Creek-Montanore deposit is constrained by the restricted availability of sample for kinetic testing of acid generation potential, coupled with the inherently scale-limited capacity of laboratory geochemical test methods in predicting future water quality. Reliance on long-term monitoring data from Troy as a field-scale geochemical analog for Rock Creek-Montanore offers a complementary method of predicting long-term drainage chemistry in underground workings where ore is exposed. Field-scale monitoring at Troy, together with laboratory geochemical analyses of core samples from Rock Creek-Montanore baseline studies, offers a more comprehensive basis for predicting future water quality. This case study compares the zones mined at Troy with those to be mined at Rock Creek-Montanore to establish the geochemical

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analogy that gives meaning to the use of Troy monitoring data in predictions for Rock Creek-Montanore. The fundamental question addressed in this case study is whether the ore beds to be mined at Rock-Creek from the lower Revett Formation are geochemically comparable to those mined from the upper Revett at Troy. Sampling objectives include spatially and lithologically representative sampling of the two zones, followed by detailed lithologic and mineralogic description, and geochemical analysis of key metal and sulfur geochemistry.

Data needs: Geological, mineralogical, and geochemical data were needed to evaluate and compare a representative population of samples within the targeted Revett ore zones. A sufficient number of samples were needed to adequately characterize the distribution of metal and sulfur concentrations. The most comprehensive and representative geological (drill logs, cross-sections, maps) and geochemical (assayed Cu and Ag) data were collected for exploration and metallurgical purposes, and validated for purposes of mine claim certification by U.S. Forest Service (USFS) mineral examiners in 1984 (Table A5.1). Mineralogy data collected in hand specimen during exploration were supplemented for this case study using petrographic, X-ray Diffraction (XRD), and Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS) methods to study 11 samples (Maxim 2003). These data support strongly consistent mineralogy and stratigraphic variation within the Revett Formation, with small-scale cyclic variation in frequency of quartzite, siltite, and argillite interbeds that are of minor importance at the mining scale due to strong silica encapsulation of ore minerals.

The Cu and Ag assay data used by the USFS to validate mineral claims for Rock Creek (n=347) and Montanore (n=213) were compiled for statistical analysis, along with several years of operational data from Troy (n=269), using standard spreadsheet and statistical software packages. Resulting distributions and summary statistics are summarized in Figure A5.17. Statistical analysis of variance for the Rock Creek and Troy data sets showed that these deposits are strongly similar to one another at a high level of confidence (Ag, $p=0.08$ and Cu, $p=0.35$, for $n=613$). Monitoring data from underground workings at Troy therefore offer a meaningful basis for field-scale prediction of future water quality for the Rock Creek-Montanore deposit.

Conclusions and Lessons Learned: Data quality for exploration geology and geochemistry data was certified using USFS mineral examination criteria. Mineralogical data were validated using standard analytical procedures for XRD and SEM-EDS methods (Maxim Technologies 2003). Data quality for historical operational assays of metal concentrations at Troy could not be validated independently for this study, but are assumed to be of high quality due to their central importance in successful metallurgical processing of mined ores. In this case, the value of historical data offering longer term and more comprehensive data was balanced against the inability to validate operational assay work. Laboratory analysis of select samples for environmental geochemical baseline characterization can be meaningfully complemented using field-scale monitoring of geochemical analogs. A basis for such comparison can be established using exploration and operations data to evaluate similarity in lithology, mineralogy, and geochemistry. “Mining” of historical exploration and metallurgical data can offer important data relevant to environmental geochemical baseline studies.

Bibliography:

Boleneus, D.E., L.M. Applegate, J.H. Stewart, and M.L. Zientek, 2005. Stratabound Cu-Ag Deposits of the Mesoproteozoic Revett Formation, Montana and Idaho. USGS Scientific Investigation Report, 2005-5231.

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Enviromin, 2007. Summary of Geochemical Data for the Rock Creek-Montanore Stratabound Cu-Ag Deposit: A Technical Report prepared for the Montanore Project EIS.

Maxim Technologies, 2003. Rock Creek Project Geochemistry Review. Third party review prepared for USFS Kootenai National Forest, May.

Table A5.1 Geological and geochemical data for Revett-Style Cu-Ag deposits, Montana.

	Montanore		**Rock Creek**		**Troy**	
	n	Mean	*n*	Mean	*n*	Mean
Assay Claim Validation	**		**		**	
Copper, ppm	213	5,400	347	6,700	269	7,100
Silver, ppm	213	45.7	347	52.2	269	44.5
Sulfur, weight %, calculated from Cu	213	0.14	347	0.17	282	0.18
Mineralogical Analysis		**	**		**	
Quantitative/analytical			10	nr	>100	nr
Feet drilled	1,500	nr	3,000	nr	11,429	nr
Mineralogy descriptions	1,000	nr	1,500	nr	4,798	nr
Assays	1,500	nr	7,255	nr	3,799	nr

After Enviromine (2007, Tables 3-1, 3-2, 3-3), ** Maxim Technologies (2003). n = number of samples nr = mean value not relevant

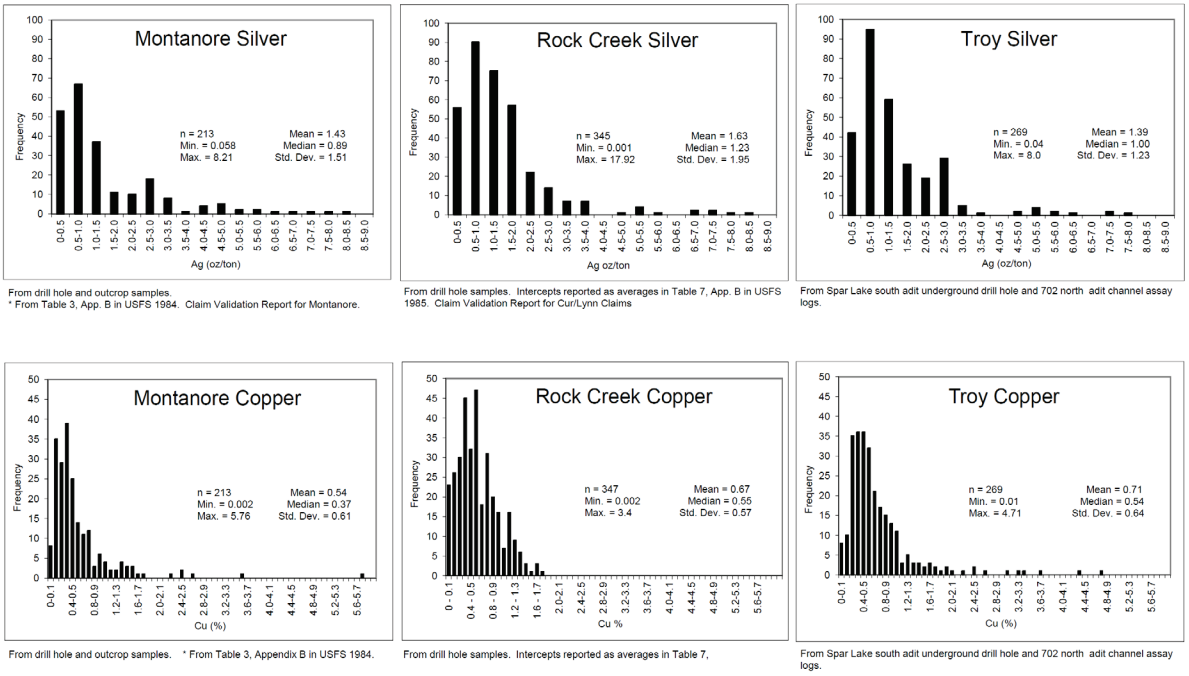


FIGURE A5.17 Comparison of assay data from Montanore, Rock Creek, and Troy.

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PREDICTING FUTURE GROUNDWATER QUALITY FROM A BACKFILLED PIT

Characterizing the Golden Sunlight Mine, Jefferson County, Montana

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Purpose of case study: Use of operational geochemistry data with mineralogy to develop and support a detailed analysis of the environmental impacts of pit backfill.

Brief site description and history: The Golden Sunlight Mine is a gold mine located approximately 50 kilometers east of Butte, Montana, in Jefferson County at the southern end of the Bull Mountains (Montana Department of Environmental Quality and Bureau of Land Management 2007). The mine is immediately north of Interstate 90, a major east-west thoroughfare and is highly visible from the highway. The mine is operated by Golden Sunlight Mines, Inc. (GSM), a wholly owned subsidiary of Barrick Gold Corporation.

The ore body is a breccia pipe in the late Precambrian host rocks of the Belt Supergroup (LaHood, Greyson and Newland formations). The breccia pipe is an irregular 250 meter cylinder and dips to the southwest. It is cut through and bounded principally by a highly complex series of east- and northeast-trending high-angle faults. The breccia pipe includes disseminated gold-bearing sulfide mineralization, including pyrite and minor tellurides. Stockworks extend more than 30 meters into the wallrock. The wallrock itself is hydrothermally altered containing sulfides, principally pyrite. Northeast trending gold-quartz veins are superimposed on the entire complex and are predominantly pyrite, with very rare galena, sphalerite, and barite. This highly complex geology and the presence of extensive sulfides, principally pyrite, highlight the environmental concerns regarding acid rock drainage (ARD) at the site. Waste rock at the site is highly acidic with negative to strongly negative acid-base accounting (ABA) numbers (-2 to -70 Kg/tonne $CaCO_3$) and pH values ranging from 2.0 to 3.0 in samples collected from pore waters in waste rock; sulfate values can be as high as 30,000 mg/L with associated high iron values. All reclamation alternatives envision long-term water collection and treatment of the acidic waters characteristic of effluent from the pit and waste dumps at GSM.

GSM mines and processes ore and waste from private land and Federal lands administered by the USBLM as well as State lands administered by the Montana Department of Natural Resources and Conservation. The mine is jointly permitted by the Montana Department of Environmental Quality (MDEQ) and the USBLM.

Early mining activity in the area was carried out in the late 1800s and early 1900s prior to extensive exploration in the 1960s and 1970s. GSM began major operations in 1982 following preparation of an Environmental Impact Statement (EIS) in 1981.

Problem definition: A March 2002 state court ruling, in response to a lawsuit filed by environmental groups, ordered MDEQ to have the mine begin implementation of the Partial Pit Backfill Alternative considered in a 1997 expansion, in accordance with state law.

Following a complex series of legal rulings, USBLM and MDEQ prepared a Supplemental Environmental Impact Statement (SEIS) evaluating the impacts of backfilling the pit at GSM with the reactive waste rock noted above. The ultimate quality and fate of groundwater in a backfilled pit was the major environmental concern evaluated in the SEIS.

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The first aspect of this issue is what will the water quality of groundwater in the backfilled pit be? The waste material from the east dump complex includes a wide range of weathered acidic material. There are extensive oxidation products from waste that have been in place for several years as well as material that is relatively fresh and can be expected to oxidize. Material in the backfilled pit will both continue to oxidize, driven both by oxygen and ferric iron, but also stored oxidation products would be subject to dissolution and mobilization as groundwater fluxes through the backfilled pit. Other than the time, estimated to be on the order of hundreds to thousands of years, required to (1) oxidize all the contained pyrite, and (2) remove oxidation products, both from continued pyrite oxidation and dissolution of preexisting byproducts, there are no mechanisms for improved water quality in the backfilled pit. Pore water in the backfilled pit would have all the characteristics of typical acid drainage: pH of 2.3, sulfate values in the range of 22,000 to 30,000 mg/L, and high in a variety of metals. Evaluation of alternatives such as using wells to dewater the pit concluded that because of the unstable and highly corrosive nature of the material and water in the backfilled pit, use of wells would not be successful.

The areas east and west of the mine consist of Tertiary valley fill sediments. Because the mine pit opens to the east, and the mine facilities are located below the pit, the geology east of the mine is of critical importance regarding potential ARD flow pathways. Historical ARD may have consumed potential neutralizing capacity along flow paths, which complicated environmental concerns. The effluent from the backfilled pit will most likely reactivate and utilize the preexisting flow paths from the pit area. The primary flow paths flow east and south, first through structures and then through valley fill before joining existing flow east of the pit. This flow path was identified as the colluvial aquifer.

Data needs: Geological, mineralogical, geochemical, and geohydrological information was needed to evaluate the environmental impacts of backfilling the pit with highly acidic waste rock on associated groundwater flows. The mine had extensive data for the geology, mineralogy, and geochemistry of the mine area and mine waste which had been developed throughout the operation of the mine. Data regarding the geohydrology, lithology, mineralogy, and geochemistry of the flow path in the colluvial aquifer required extensive re-evaluation of information collected below the minesite for a variety of purposes. This included re-evaluating monitoring well logs, monitoring well flow, and water quality information gathered earlier in the mine life cycle.

Conclusions and lessons learned: An analysis was made of the ability of the colluvial aquifer to attenuate ARD and it was determined that the amount of neutralizing capacity in the colluvial aquifer was not sufficient to neutralize the estimated 61 liters/minute effluent that would flow from the backfilled pit and mix with the existing Rattlesnake drainage flow. The existing Rattlesnake drainage flow of 190-400 lpm is impacted by natural mineralization, having a pH of 4.3, sulfate of 731 mg/L, aluminum of 6.5 mg/L, copper of 0.43 mg/L, zinc of 0.54 mg/L, and nickel of 13.03 mg/L based on existing GSM monitoring wells. Mixing of these two groundwater components was modeled using a two-dimensional (cross-section) flow-net analysis and would result in a 7- to 15-fold increase in sulfate concentrations and a 6- to 12-fold increase in copper concentrations. The ability of the colluvial aquifer to attenuate ARD contaminants was evaluated including acid/base reactions, silicate dissolution, sorption, ion exchange, oxidation-reduction reactions, and mixing. Much of the colluvial aquifer has an alkalinity of 30 mg/L or less, lacking

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the identified calcareous zones or carbonate cementation found in other Tertiary sediments east of the mine. Calculations show that groundwater quality standards within the Jefferson alluvial aquifer below the mine would be exceeded for copper (1.3 mg/L) and nickel (0.1 mg/L).

Bibliography:

Montana Department of Environmental Quality and Bureau of Land Management, 2007, Final Supplemental Environmental Impact Statement Golden Sunlight Mine Pit Reclamation, July, 2007.

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APPENDIX 6—APPLICATIONS AND EXAMPLES OF GEO-ENVIRONMENTAL MODELS (GEMs) AT MINE SITES

by Virginia T. McLemore

Reviewed by Ron L. Schmiermund and Robert R. Seal II

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APPENDIX 6. APPLICATIONS AND EXAMPLES OF GEOENVIRONMENTAL MODELS (GEMs) AT MINE SITES

Virginia T. McLemore

INTRODUCTION

The purpose of this appendix is to describe selected geoenvironmental models (GEMs) and how they could be used at mine sites. The basic objective of GEMs is to predict future and long-term geochemical and hydrological behavior and to identify potential environmental problems at a mine site by comparing the mine site to similar types of deposits in the world. However, very few case studies have been published that describe the use of GEMs, therefore several historical mine sites were selected for this appendix to apply the concept of GEMs, even though the selected site has produced and at some mine sites already closed. The mine sites discussed in this report were selected simply based upon available published data and known environmental issues. In most of these selected mine sites, GEMs typically were not used because the GEMs were not available to assist in identifying or preventing environmental issues at the time of production; some of these mine sites were not even required to have any environmental programs at the time of production.

GEMs were developed by the U.S. Geological Survey (USG)S to aid in environmental assessment of mineral deposits (Seal and Foley 2002; Seal and Hammarstrom 2003). A GEM is defined as "a compilation of geologic, geophysical, hydrologic, and engineering information pertaining to the environmental behavior of geologically similar mineral deposits (1) prior to mining, and (2) resulting from mining, mineral processing and smelting" (Seal et al. 2002). The key elements of a GEM include deposit type, deposit size, host rock, wall-rock alteration, mining and ore processing method, deposit trace-element geochemistry, primary and secondary mineralogy, topography and physiography, hydrology, and climatic effects (Table A6.1). GEMs are important to include in sampling and monitoring programs throughout the entire mine life cycle, because GEMs provide guidelines on the environmental characteristics of different types of mineral deposits that could be encountered at a mine or mineral processing site and can be used to determine the specific type of sampling and monitoring program required or identify specific media or constituents that should be sampled or monitored. GEMs are empirical data compilations that are best used as guidelines for the potential range of environmental impacts at a site and should not be used to predict absolute pH or metal concentrations that will develop at a site or in lieu of site mineralogical and chemical characterization (Plumlee 1999; Plumlee et al. 1999; Seal et al., 2002).

GEMs can support and predict:

- Establishment of pre-mining baseline or background conditions
- Exploration for new mineral deposits
- Mine planning and mine development
- Remediation
- Legacy issues (abandoned, inactive, historic mines)
- Natural altered areas that could contribute to adverse environmental effects
- Permitting, especially in the case of new districts or the first introduction of modern mining methods
- Rational assessments of operational and closure costs.

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GEMs can and should be used throughout the mine life cycle (Schmiermund and Ranville 2006), but in reality are not widely applied, probably because of several factors, including:

- Complexity of applying the GEMs
- Constraints due to project timing
- Unavailability of original exploration geologists and geochemists who have examined the deposit and adjacent areas in detail
- Communication barriers between the various teams developing the mine site.

During exploration, GEMs provide a means to anticipate geochemical environmental problems before they occur and aid in identifying the pre-mining baseline and background conditions. However, most exploration geologists are not familiar with specific environmental issues associated with mining and generally do not have the time or budget to address them. Furthermore, the exploration geologists and geochemists are not available during the permitting and designing of environmental programs since they have moved on to other exploration projects. During feasibility studies and permitting phases, GEMs can highlight and prioritize the types of environmental problems that could be encountered with the particular deposit type (i.e., acid drainage, presence of heavy metals, among others). However, during feasibility, development, and production stages, communication between the various teams is not always optimum. During the development and production stage, GEMs aid in identification of potential problems associated with specific mining methods and processing methods. Close interactions between the metallurgists and environmental geologists/geochemists/hydrologists is essential in understanding the subtle details of the metallurgical or beneficiation process being used and how to handle the resulting wastes efficiently with minimal effect to the environment. Finally, GEMs are useful in mine closure by providing a global perspective on pre-mining baseline or background conditions of similar deposit types, which are useful in establishing realistic closure goals.

GEMs have the greatest benefit when applied early in the mining process and in a collaborative environment. As the mine moves into production, the usefulness of GEMs diminishes because many of the decisions that depend upon application of the GEM have been made. However, applying GEMs throughout the mine life cycle can provide a reality check, but GEMs only provide guidance and should not blind the user to new and possible contradictory information. Table A6.2 summarizes some of the environmental issues that could have been predicted by GEMs at selected mines and deposits.

The Ficklin diagram is one example of the use of GEMs in the prediction of water quality at mine sites (Figure A6.1, A6.2). The Ficklin diagram is a scattergram of base metals (Zn, Cu, Cd, Co, Ni, Pb) plotted against pH and can be used to interpret variations in mine-drainage water chemistry among different deposits (Plumlee et al. 1994, 1999). These metals were selected because they are considered the most diagnostic parameters in differentiating between different geochemical processes that can lead to Mining Influenced Water (MIW) problems. Similar plots can be constructed using other metals and SO_4 for site specific mines or processing sites (INAP 2009).

TABLE A6.1. Geologic characteristics of mineral deposits that affect their environmental signatures (modified from Plumlee 1999).

Characteristic	Type of control	Explanation
Iron sulfide content	Chemical	Oxidation generates acid and supplies ferric oxide, an aggressive oxidant
Other sulfides	Chemical	Some sulfates can generate acid during

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Characteristic	Type of control	Explanation
		oxidation, depending upon the oxidant and chemistry of the minerals.
Carbonate, aluminosilicate, and other nonsulfide minerals	Chemical	Many of these minerals can consume acid. Iron and manganese carbonates can generate acid under specific conditions.
Mineral resistance to weathering	Physical	Function of the mineral and the texture and trace-element content of the mineral. Different minerals weather at different rates.
Secondary mineralogy	Chemical and physical	Soluble secondary minerals can store acid and metals that can be released when the minerals weather. Insoluble secondary minerals can armor reactive minerals and restrict access to weathering. Other minerals can store trace elements in insoluble forms.
Extent of pre-mining or pre-erosion weathering and oxidation	Chemical and physical	Pre-mining oxidation can reduce or enhance the potential for sulfide deposits to generate acid, depending on the site mineralogy and weathering processes.
Host rock lithology	Chemical and physical	Can consume or generate acid. Physical characteristics (porosity, permeability) control access of weathering agents.
Wallrock alteration	Chemical and physical	Can increase or decrease the ability of the host rock to consume or generate acid. Can increase or decrease the ability of the host rock to transmit water. Can increase or decrease resistance to erosion.
Major- and trace-elements in deposit and host rocks	Chemical	Elemental composition of deposit and host rocks typically reflected in environmental signatures and associations vary by deposit type.
Physical nature of ore body (vein, disseminated, massive, layered)	Physical	Controls access of weathering/oxidation agents. Influences mining method and amount of waste rock removal to access ore.
Porosity, hydraulic conductivity of host rocks	Physical	Controls access of weathering/oxidation agents.
Presence and openness of faults, joints	Physical	Controls access of weathering/oxidation agents.
Deposit grade and size	Physical and chemical	Controls magnitude of natural and mining impacts on surrounding environment.
Topography and physiography	Physical	Affect the position and shape of groundwater tables, which in turn control the extent to which mines or mineral deposits are exposed to significant groundwater flow.
Hydrology	Physical and chemical	Strongly controlled by geologic characteristics of deposits, including whether ore is present as veins or lenses, both of which can focus groundwater flow, or whether low-permeability barriers to groundwater flow, such as clay-altered wallrock are present.
Climatic effects	Physical and chemical	Affects the environmental behavior of mineral deposits, but its effects are often subordinate to those of deposit geology.

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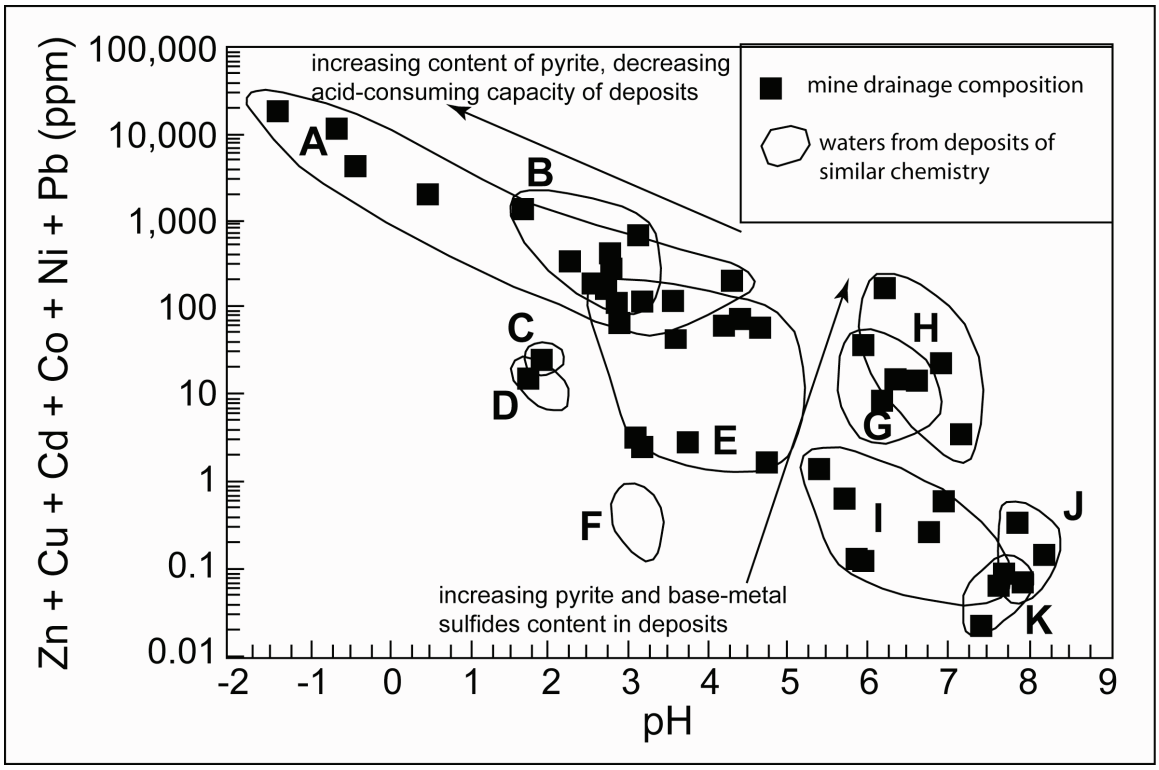


FIGURE A6.1. The Ficklin plot, shown here, is a plot of pH verses total metal content (the sum of dissolved Zn, Cu, Cd, Co, Ni, Pb concentrations) of waters draining from diverse mineral deposits (from Plumlee et al. 1994). The purpose of this plot is to illustrate the typical metal content of waters derived from specific types of mineral deposits which are identified by the letters in the diagram. The fields indicate waters from similar deposits. A=pyrite-rich massive sulfides, B=sulfide-rich ores with pyrite in altered wallrock, C=high sulfide, low base-metal hot spring ores in altered wallrock, D=high-sulfide, low-base metal porphyry Mo ores, E=pyrite- and base-metal-rich polymetallic veins and disseminations in wallrock with low acid-buffering capacity, F=pyrite-rich, base metal-poor veins and disseminations in wallrock with low acid-buffering capacity, G=pyrite- and base metal-rich polymetallic veins with carbonate or in carbonate wallrock, H=pyrite- and base metal-rich, polymetallic replacements and veins in carbonate-rich sediments, I=polymetallic veins with moderate to low pyrite and base metal content that are carbonate-rich or in carbonate wallrock, J=pyrite-poor polymetallic replacements in carbonate-rich sediments, K=pyrite-poor, Au-Te veins and breccias with carbonate gangue.

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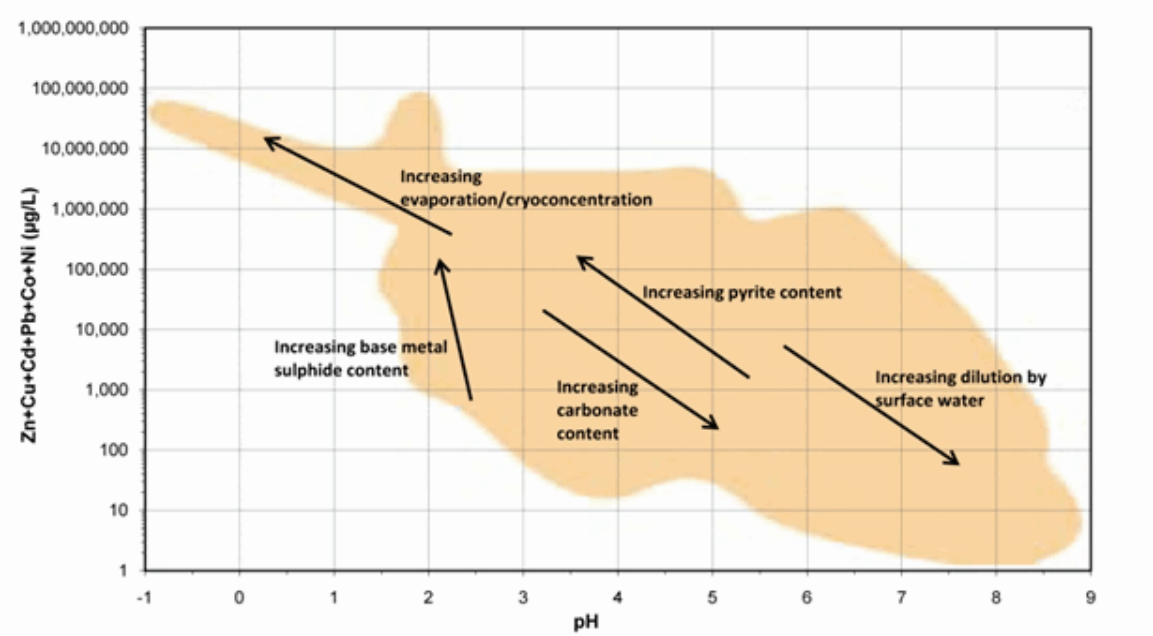


FIGURE A6.2. Revised Ficklin diagram. Arrows show likely path of water chemistry as a result of designated chemical processes (Revised from Ficklin et al. 1992; Plumlee et al. 1999; INAP 2009).

Environmental scientists realized that geologic and geochemical characteristics of mine and metallurgical processing sites play an important role in the understanding, prevention, and mitigation of MIW (Kwong 1993; Wanty et al. 1999; Odor et al. 1999; Hough et al. 2003; Maest et al. 2005). Price et al. (1995) compared water quality from various mines in Nevada and found that the water quality varies with the type of deposit. Plumlee et al. (1999) also compared water quality from various types of mineral deposits and came to the same conclusion. A project by Jung (2008) examining metals in mine wastes at mines in Korea also demonstrated that the type of mineral deposit was important in the resulting contamination.

Another project headed by James R. Kuipers and Ann S. Maest examined the performance of reclamation of major mines on U.S. federal land that were required to submit EA (Environmental Assessment) and EIS (Environmental Impact Statement) reports (Kuipers et al. 2006). Although, Kuipers et al. (2006) did not specifically identify the types of deposits or utilize the USGS GEMs, their report does recognize that geologic, geochemical and hydrologic characteristics are important in understanding water quality issues at mine sites and found that not all sites included detailed geologic descriptions. Eleven of their 25 mines selected as case studies exhibited inadequacies in geochemical characterization of the sites, including incorrect assumptions on the nature of ore deposits and surrounding areas, inadequate sampling, and failure to conduct the proper tests, wrong interpretations, or incorrect use of the proper models (Kuipers et al. 2006). Of the 71 mines studied, 23% of these sites had no or insufficient geological information in the EIS reports. The process of selecting and applying the relevant GEMs could have prompted the acquisition of the missing information and provided additional information and background.

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TABLE A6.2. Examples of mine and deposit sites with observed environmental issues that could have been predicted by GEMs.

Mine or deposit (commodities)	Geoenvironmental model (reference)	Environmental issue	Reference
Red Mountain, Alaska (base and precious metals)	Volcanicogenic-massive sulfide (Shanks et al., 2009) (Model 28a, 24a,b)	Presence of pyrite weathering has produced acid generation, high metal and rare earth elements content in surface waters. This deposit has not yet been mined.	Eppinger et al. (2007)
Questa, New Mexico	Climax molybdenum (Luddington and Plumlee, 2009) (Model 16)	Acid drainage in surface and groundwater from waste rock piles and tailings contains high metal content (Cd, As, Pb, Sb), F, and sulfate. Altered areas upstream of mine also contribute high metals, F, and acid drainage. Carbonate is present in some rock piles and neutralizes acid drainage locally.	Agency for Toxic Substances and Disease Registries (2005), Luddington et al. (2005), Nordstrom (2007), McLemore et al. (2008), see description in text
Hitura, Finland (Ni)	Magmatic-rich Ni-Cu (±PGM) sulfide (Schulz et al., 2010) (Model 7a)	Tailings-derived mine drainage water has variable pH (2.8-8.7), high electrical conductivity, and contains elevated Fe, Cd, Co, Cu, SO_4, Cl and Ni concentrations.	Heikkinen et al. (2002)
Greens Creek, Alaska	Volcanicogenic-massive sulfide (Shanks et al., 2009) (Model 28a, 24a,b)	Waste rock piles and tailings are potentially acid generating that could lead to acidic surface, see description below	Taylor et al. (1995), Kuipers et al. (2006), SRK Consulting Inc. (2009), see description in text
Golden Sunlight, Montana	Au-Ag-Te vein deposits (Model 22b)	Cyanide, ARD, and high copper in groundwater.	Kuipers et al. (2006)
Zortman and Landusky, Montana	Au-Ag-Te vein deposits (Model 22b)	Acid drainage and high metals from waste rock piles and heap leach.	Kuipers et al. (2006)
Jerritt Canyon, Nevada (Au)	Sediment-hosted Au (Model 26a)	Cyanide plume from tailings pond. Increase in SO_4 and TDS downstream of waste rock piles.	Kuipers et al. (2006)
Twin Creeks, Nevada (Au)	Sediment-hosted Au (Model 26a)	Perched groundwater high in cyanide, As, TDS, and other constituents near tailings.	Kuipers et al. (2006)
Spor Mountain, Utah (Be)	Volcanogenic beryllium (Foley et al., 2010)	Minimal environmental issues because the deposit contains little or no pyrite, low concentrations of betrandite, and arid environment. There are not any water data on uranium concentrations, which could be a concern.	Foley et al. (2010)

EXAMPLES OF THE USE OF GEOENVIRONMENTAL MODELS AT MINE SITES

GEM: Climax Molybdenum Deposits (USGS Model 16)

Climax molybdenum deposits typically are large (100 to 1,000 million metric tons of ore grading 0.6-1% Mo) porphyry-hosted deposits containing stockwork molybdenite-bearing veins and veinlets that are surrounded by highly altered host rock (Ludington and Plumlee 2009).

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Oxidation of pyrite found in the ore as well as altered host rocks can contribute to acid drainage in nearby streams and rivers, locally with pH of 1-3. Mining and milling can exacerbate acid drainage. Trace elements, such as Rb, Li, Cs, Be, Nb, U, Th, F and others can be present in elevated concentrations.

Application example: Questa mine, New Mexico

The Questa deposit is a Climax-type porphyry molybdenum (±tungsten) deposit, which is a large, low-grade (0.1–0.2% Mo) deposit that contains disseminated and stockwork veinlets of molybdenum sulfides and is associated with silica- and fluorine-rich porphyritic granitic intrusions. The Chevron Mining Inc. (formerly Molycorp, Inc.) Questa molybdenum mine is located on the western slope of the Taos Range of the Sangre de Cristo Mountains of the Southern Rocky Mountains near the edge of the Rio Grande rift in north-central New Mexico (McLemore 2009). Molycorp, Inc. mined some 81 million short tons of ore from their open pit at a grade of 0.191% MoS_2 between 1965 and 1982 (McLemore 2009). During the period of open pit mining (1969-1982) at the Questa mine, approximately 350 million short tons of overburden rock was removed and deposited onto mountain slopes and into tributary valleys forming nine mine waste rock piles surrounding the open pit. Underground block caving of ore commenced in 1982. Underground production from the Goat Hill orebody from 1983 to 2000 amounted to 21.11 million short tons of 0.31% MoS_2 (McLemore 2009).

Numerous environmental studies of the Questa mine are consistent with environmental effects predicted by the GEM. Predominant weathering reactions in the Goathill North (GHN) rock pile involve the oxidation of pyrite, dissolution of carbonate, and formation of sulfate minerals, mainly gypsum, jarosite, and soluble, efflorescent salts (depending on pH). Petrographic and geochemical analyses suggest higher abundances of jarosite, gypsum, Fe oxide minerals and soluble efflorescent salts (locally as cementing minerals), and lower abundances (to absence) of calcite, pyrite, and epidote in samples from the outer parts of the rock pile as compared to the interior (McLemore et al. 2009). The chemical composition of waters from the Questa rock piles (i.e., seeps and runoff waters from the rock piles, chemistry of leachate waters obtained by laboratory leaching of rock pile samples), contain high amounts of SO_4 (up to 19,000 ppm) and Al (up to 1690 ppm), implying that sulfide and silicate dissolution is occurring within the rock piles. Paste pH, which is related in part to the degree of weathering, is generally lower in samples from the outer parts of the rock pile as compared to samples from the interior (McLemore et al. 2009). The GEM predicted high F^- in the dump seepages and the dramatic influence of F-complexation on aluminum solubility, as is the case at the Climax mine, Colo. and Questa.

The evidence for weathering in the Questa waste rock piles is best illustrated by petrographic techniques and includes:

- Change in color
- Change in bulk texture
- Changes in mineralogy
- Changes in mineral texture
- Changes in grain textures.

Not all weathered samples exhibit all of these features. There are five principal lines of evidence that the clays in GHN are predominantly from the hydrothermal alteration to the host lithologies prior to excavation and emplacement in to the GHN rock pile and not formed by weathering (Donahue et al. 2007; Graf 2008):

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- Presence of fresh or lightly tarnished pyrite surrounded by clay minerals
- The unweathered drill core and samples from GHN have similar types and abundances of clay mineral groups based on X-ray diffraction (Xrd) analyses
- Chemical analyses from electron microprobe analysis reveal similar chemical compositions for the clays from the two environments (unweathered drill core vs. Weathered GHN rock pile material)
- Textural evidence gathered from electron microprobe examination
- Oxygen and hydrogen isotopic analysis.

Additional studies of the altered rocks associated with the Questa mine have established background data that can be used in other porphyry molybdenum sites (Nordstrom 2007). More than 20 naturally forming "alteration scars" are found along the mountain ridges of Red River between the towns of Questa and Red River. Alteration scars are natural, colorful (red to yellow to orange to brown), unstable weathering landforms that are characterized by steep slopes (greater than 25 degrees), moderate to high pyrite content (typically greater than 3%), little or no vegetation, and extensively fractured bedrock. Water-quality degradation of the Red River by the input of acid, sulfur, and other elements from surface water and groundwater derived from the alteration scars has been documented by past environmental studies (Allen et al. 1999; Shaw et al. 2003; Briggs et al. 2003; Lovetere et al. 2004; Maest et al. 2004; Ludington et al. 2005; Nordstrom et al. 2005; Naus et al. 2005; Nordstrom 2007). The high erosion rates on the bare slopes of the scars lead to denudation that continuously exposes additional pyrite-bearing outcrops. The dissolved constituents form acid drainage that mixes with surface water and groundwater and ultimately enters Red River. Paste pH of soils in the alteration scars averaged 2.5, and have high paste conductivities (greater than 1000 µS/cm) and sulfate concentrations that ranges from 1 to 11.5 percent (Shaw et al. 2003; Robertson GeoConsultants, Inc. 2000a, b; McLemore et al. 2009). Leach extractions of samples from the alteration scars show elevated concentrations of S, Cu, F, Bi, Sn, Mn, K, and Th (Robertson GeoConsultants, Inc. 2000a, b, Shaw et al. 2003). There does not appear to be any chemical distinctions among the various scars (Robertson GeoConsultants, Inc. 2000a, 2000b, Shaw et al. 2003; Briggs et al. 2003), which suggests that they were formed from similar rocks by similar processes.

GEM: Volcanicogenic-Massive Sulfide (VMS) Deposits (Models 24A-B, 28A)

Volcanicogenic-massive sulfide (VMS) deposits are lens-shaped to sheet-like bodies of sulfide-rich (at least 50% pyrite, chalcopyrite, galena, sphalerite, etc., with some precious metals), polymetallic, stratabound deposits associated with basaltic to rhyolitic volcanic rocks (Taylor et al. 1995; Sangster and Scott 1976; Seal et al. 2000; Seal and Hammarstrom 2003; Franklin et al. 2005; Shanks et al. 2009). Oxidation of pyrite and pyrrhotite found in the ore as well as altered host rocks can contribute to acid drainage in nearby streams and rivers, locally with pH of 1-3. Analyses of waters draining VMS deposits can plot in the extreme metal-high to ultraacidity fields (Figure A6.2). Host rocks typically have low buffering capacity. High concentrations of toxic metals can be present in some deposits, including As, Bi, Cu, Cd, Hg, Pb, Sb, and Zn (Taylor et al. 1995; Shanks et al. 2009).

Application example: Greens Creek mine, Alaska

The Greens Creek mine on Admiralty Island has been in production since 1984 and contains estimated reserves of 21.9 million metric tonnes of ore at 13.9% Zn, 5.1% Pb, 4.8 grams/metric tonnes Au, and 599 grams/metric tonnes Ag (Taylor et al. 2010a). Main ore

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minerals include sphalerite, galena, chalcopyrite, tetrahedrite, and gold with pyrite. Although, the Greens Creek deposit exhibits characteristics that are typical of VMS, sedimentary exhalative (SEDEX) and Mississippi Valley-type (MVT) deposits (Taylor et al. 2010b), for this paper it is best described as a VMS deposit.

Despite best management practices at the time, water quality was adversely impacted. Kuipers et al. (2006) points out that the predicted impacts to water quality as stated in the EIS/EA reports did not foresee the actual impacts to water quality. An independent audit by SRK Consulting Inc. (2009) concluded that the mine was "well managed with respect to oversight by company personnel and the agencies and in compliance with the majority of the applicable permits, plans, approvals and regulations." SRK Consulting Inc. (2009) summarized several water quality issues of concern that were known before the 2009 audit and were already being addressed. Applying the GEM could have provided data that could have avoided or minimized these adverse affects to water quality.

Environmental issues at the Greens Creek mine are consistent with those predicted by the VMS GEM (Taylor et al. 1995; Seal and Hammarstrom 2003; Kuipers et al. 2006; SRK Consulting Inc. 2009; additional information http://dnr.alaska.gov/mlw/mining/largemine/greenscreek/index.htm, accessed 8/23/13). Acid seepage from Production Rock Site 23/D contains some constituents above Alaska Department of Environmental Conservation (ADEC) water quality standards and is discharging into Greens Creek. See Kuipers et al. (2006) for a summary of the environmental geochemistry. The tailings are potentially acid generating, which could lead to acidic surface runoff, but placement of covers is expected to prevent any acid drainage. The geology and geochemistry of the deposit and alteration areas are well described for this mine (Taylor et al. 2010b) and the mine has provided data to the Alaska and U.S. agencies (http://dnr.alaska.gov/mlw/mining/largemine/greenscreek/index.htm, accessed 8/23/13), thus providing a data set that could be used to confirm and refine the VMS GEMs.

Application example: Red Mountain, Bonnifield district, Alaska

The Red Mountain (or Dry Creek) deposit is a VMS deposit in the Alaska Range in east-central Alaska that has never been mined, yet displays natural acid drainage and high metal and rare earth elements (REE) concentrations in the surface waters (Eppinger et al. 2007). Several mineralized massive to semi-massive sulfide horizons are found in and near the contacts between phyllic felsic metavolcanic schists and carbonaceous rocks. Primary quartz-sericite-pyrite alteration is extensive. Oxidation of pyrite and associated sulfides produced acid drainage and high metals concentration. Water samples from the quartz-sericite-pyrite alteration zone have the lowest pH (some below 3.5), highest specific conductance (some >2500 µS/cm), and highest metal concentrations. It is important to note that Red Mountain Creek and its tributaries within the quartz-sericite-pyrite alteration zone likely never supported aquatic life (Eppinger et al. 2007). The Red Mountain deposit provides an example of detailed pre-mining geochemistry of an unmined area that can be compared with known mined VMS deposits as well as provide some data on pre-mining conditions of VMS deposits and illustrate the strength of GEMs.

Application example: Pecos mine and mill, Santa Fe County, New Mexico

The VMS deposits at the Pecos mine are hosted by quartz-sericite, quartz-chlorite, and talc schists, which are interpreted to represent a metamorphosed, sheared rhyolite dome complex (Riesmeyer 1978; Riesmeyer and Robertson 1979). Numerous small lens-shaped ore bodies are

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concentrated into two subparallel, stratabound zones, named the Katydid and Evangeline deposits. The deposits strike northeast and extend for over 250 m along strike (Stott 1931; Krieger 1932a, b; Harley 1940). The mineralogy of the ore bodies is predominantly sphalerite, galena, and chalcopyrite with minor, and includes appreciable amounts of gold and silver in a gangue of quartz, sericite, pyrite, pyrrhotite, chlorite, actinolite, sericite, tourmaline, and calcite. The ore occurs in three textural types: (1) massive sulfide ore with at least 50% sulfides with massive, banded, or brecciated textures, (2) low-grade (less than 50% sulfides) massive or banded ore, and (3) stringer ore containing low-grade veinlets and stringers of sulfides (Krieger 1932a, b; Riesmeyer and Robertson 1979). The Pecos mine was mined during 1902-1904, 1927-1939, and 1943-1944 and was the largest lead-zinc producer in New Mexico from 1927 to 1939. Total production amounts to 2.3 million tons of ore grading 2.9% Pb, 9.2% Zn, 0.4% Cu, 2.4 oz/ton Ag, and 0.08 oz/ton Au worth over $40 million at the time of production (Harley 1940; McLemore et al. 1995). The Pecos mine is closed and has been reclaimed.

The Pecos mine and Alamitos Canyon mill (El Molino site 18 km south of the mine) were identified by state and federal agencies as point sources for metals contamination for Pb, Zn, Cu, Al, Se, Cd, and Cr (McLemore et al. 1993, 1995, 2001). The waste rock generated during mining activities was piled at the Pecos mine site and consists of several mine waste rock piles within an area of approximately 7.7 ha. Numerous discrete and diffuse seeps occur along the base of the waste rock piles. Material from the Pecos waste rock piles had been used as fill in the campgrounds and roads north of Terrero and these areas also were identified as point sources for metal contamination. Furthermore, the Lisboa Springs Fish Hatchery (New Mexico Game and Fish Department) experienced several fish kills from March 1991 through 1995 that was attributed to contamination from the Pecos mine (McLemore et al. 1993, 1995, 2001). Reclamation of the campgrounds, roads, and Alamitos Canyon mill site began in the early 1990s and was completed in 2002.

Geochemical analyses of surface-water samples prior to reclamation indicated that drainage from the Pecos mine did not significantly affect the composition of the surface water in the area, except in the immediate vicinity of the mine and mill site (Figure A6.3; McLemore et al. 2001). Water chemistry from seeps along the waste rock piles before reclamation is summarized in Figure A6.3. Elevated concentrations of Cu, Pb, Zn, Cd and other metals occurred in stream sediments below both the Pecos mine and Alamitos Canyon mill sites before reclamation began (Brandvold and McLemore 1999; McLemore et al. 1993, 1995, 2001) and had typical geochemical characteristics similar to VMS deposits. A decrease in metals concentrations in the stream sediments occurred with time since reclamation began, especially in the immediate vicinity of the Pecos mine (where Cu levels decreased from 310 to 92 ppm; Cd from 17 to 4.7 ppm; Pb from 300 to 160 ppm; and Zn from 3100 to 2080) and below the confluence of Alamitos Creek (Cu decreased from 270 to 30 ppm; Pb from 590 to 93 ppm; and Zn from 990 to 287 ppm). Collectively, multi-disciplinary studies suggest that Cu, Pb, Zn, and other metals in sediments were eroded and leached from the Pecos waste rock piles and Alamitos Canyon mill tailings, which migrated into the Pecos River (McLemore et al. 1993, 1995, 2001). These studies confirmed a decrease in metal concentrations with time since reclamation began, especially in the immediate vicinity of the Pecos mine and Alamitos Canyon. Reclamation efforts at the Alamitos Canyon mill site were successful in that the metal concentrations in both surface water and stream sediments have decreased since reclamation. The overall metal concentrations dramatically decreased in stream sediments below Pecos village in all sampling trips, mostly due to dilution of sediment derived from the red bed sedimentary units. Therefore, the total metals

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addition from the Pecos waste rock piles and Alamitos Canyon mill tailings into the lower Pecos River is insignificant compared to sediment input from the Pecos Wilderness and drainages south of Pecos village, except in the immediate vicinity of the Pecos mine and mill site.

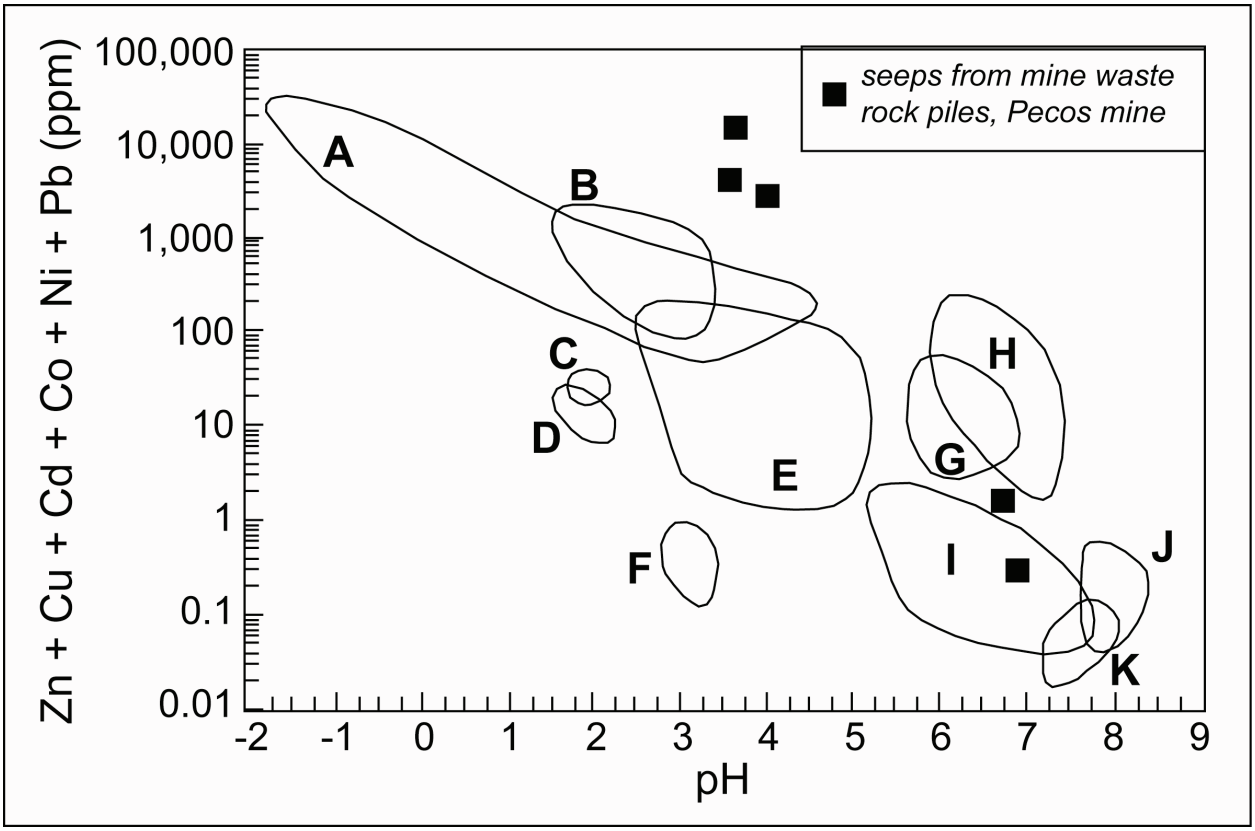


FIGURE A6.3. Ficklin plot of chemical analyses of water samples from seeps at the Pecos and Jones Hill mines (in ug/L). Analyses by NMBMMR Chemical Laboratory and reported in McLemore et al. (2001). The fields indicate waters from similar deposits, explained in Figure A6.1.

Porphyry Copper Deposits (Model 17)

Porphyry copper (molybdenum, gold) deposits are large, low-grade (<0.8% Cu) deposits that contain disseminated and stockwork veinlets of copper and molybdenum sulfides associated with porphyritic intrusions (Lowell and Guilbert 1970; Kesler 1973; Lowell 1974; John et al. 2010). Mineralization typically occurs in and around porphyritic diorite, granodiorite, monzonite, and quartz/monzonite plutons, which are surrounded by concentric zones of hydrothermal alteration (Lowell and Guilbert 1970). Predominant geoenvironmental characteristics of porphyry copper deposits include (1) potential for generating acid drainage due to presence of pyrite and other acid-generating minerals, (2) low acid-neutralizing potential provided by the presence of feldspars, and low amounts of carbonate minerals, and)3) presence of elevated acidity and elevated concentrations of Cu, Pb, Zn, Mo, and As in soils, sediments, groundwater, and surface water prior to mining (John et al. 2010). A report by the USEPA (1999) illustrated that dump leaching and solvent extraction-electrowinning operations could extract and concentrate soluble radioactive elements.

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Application example: Porphyry deposit in Peru

In this example, adequate sampling and analysis could not be completed prior to the first stage of a permitting process for a porphyry Cu-Au deposit in Cajamarca Department, Peru. By applying the principals of GEMs, similar deposits in British Columbia were used to develop a geochemical database as a surrogate for site-specific data. The deposit in Peru is an Au-rich, calc-alkaline porphyry Cu deposit (Schmiermund et al. 2006). Various similar deposits in British Columbia were characterized to support permitting of the Red Chris project (data are found at http://a100.gov.bc.ca/appsdata/epic/html/deploy/epic_project_doc_list_238_r_app.html, accessed 8/23/13), which also is an Au-rich, calc-alkaline porphyry Cu deposit, with similar geology to the deposit in Peru. This surrogate database from the Red Chris deposit was then used in modeling to approximate and define the environmental data for the deposit in Peru and to temporarily represent dump seepage and sulfidic wall rock runoff and estimate facility effluent and pit lake quality (Schmiermund et al. 2006).

Application example: Copper Flat, Hillsboro district, New Mexico

The Copper Flat porphyry copper deposit at Hillsboro in the Animas Mountains in central New Mexico is one of the older Laramide porphyry copper deposits in the Arizona-Sonora-New Mexico porphyry copper belt. The Copper Flat porphyry copper deposit in the Hillsboro district consists of Cu, Au, Mo, and Ag disseminated in a quartz-monzonite stock and in thin quartz veins (McLemore et al. 1999, 2000). Unlike the Santa Rita, Tyrone, and Morenci deposits, there is only a minor supergene enrichment zone at Copper Flat; Copper Flat is a low-grade hypogene deposit with low pyrite content. Much of the ore is concentrated within a breccia pipe that is 396 m long, 183 m wide, and has a vertical extent of almost 518 m. Predominant minerals include pyrite, chalcopyrite, chalcocite, azurite, malachite, and cuprite. Minor amounts of molybdenite, galena, bornite, tetrahedrite, sphalerite, and fluorite are also present. Chalcopyrite is the predominant copper mineral. Gold and silver occur as electrum. Molybdenite is the predominant molybdenum mineral. Oxidation is restricted to the upper 6-9 m of the sulfide zone. Overburden ranges from 0 to 6 m.

Quintana Minerals Corporation mined the property for three months in 1982 producing 7.4 million pounds of copper, 2,306 ounces of gold, and 55,966 ounces of silver. Mining activities ceased because of low copper prices. Since no mining activities have occurred since 1982, the site is an excellent field laboratory for studying the behavior of metals and sulfide minerals exposed with waste rock and tailings in the arid southwest. There is a 12.8-acre pit and pit lake on site that is located near the center of the breccia pipe. New Mexico Copper Corporation has recently applied for mining permits to begin production (http://www.emnrd.state.nm.us/MMD/MARP/PermitSI027RN.html, accessed 8/20/13).

The pit lake has been sampled at least 65 times between 1989 and 1997 (Raugust 2003; Raugust and McLemore 2005). The pH of the lake is typically neutral to alkaline, with exception occurring in 1992 and 1993, where the pH dropped as low as 4.4. At least one intermittent seep from the pit wall has been sampled and the results reported a pH of 2.64, a total dissolved solid concentration (TDS) of 12,770 milligrams per liter (mg/L), and a sulfate concentration of 790 mg/L. The andesitic host rocks surrounding the ore body and groundwater inflow have a high acid buffering capacity as shown by the partial dissolution of calcite and the precipitation of gypsum and goethite. The alkalinity of the groundwater and host rocks quickly neutralizes and dilutes acidic discharges into the pit lake. Groundwater samples collected from monitoring wells located down gradient from the pit lake indicate groundwater chemistry is similar before and

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after the excavation of the pit. Most of the water samples from the porphyry copper deposit are near-neutral, low metal waters, although a few pit lake samples are high metal, acid waters (Figure A6.4). The near-neutral, low metal waters probably represent increasing dilution by surface water (Figure A6.2). The GEMs model could provide critical data for application in any new permitting process.

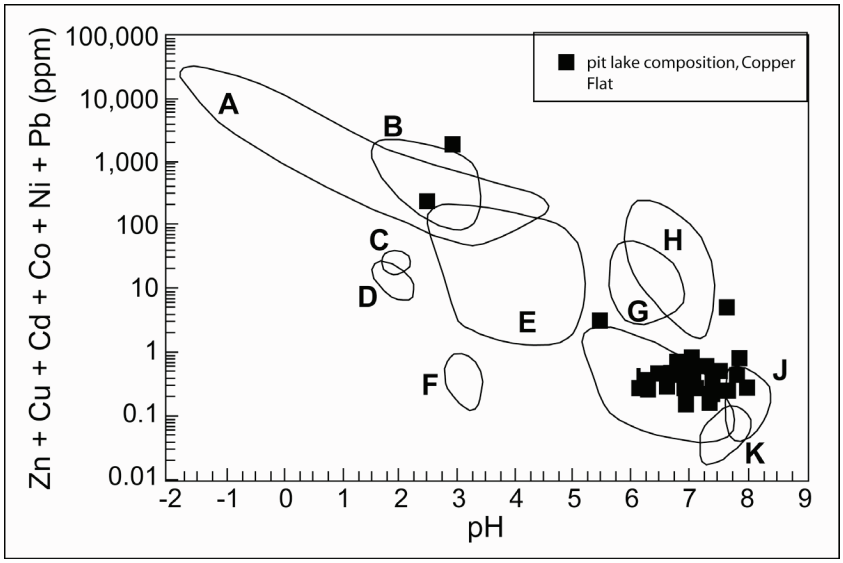


FIGURE A6.4. Ficklin plot of chemical analyses of water samples from surface water, groundwater, and pit lake samples from the Copper Flat porphyry copper deposit, Hillsboro district, New Mexico (data from Raugust, 2003).

Cu, Au, and Zn-Pb Skarn Deposits (Models 18b, c)

Skarn deposits are metasomatic magmatic deposits that are composed of Ca-Fe-Mg-Al silicate (referred to calcsilicate) minerals in replacement deposits in carbonate rocks. Skarns can be barren of economic minerals or contain various amounts of Cu, Au, Pb, Zn, and Ag minerals as well as Mn, Fe, Sn, and W (Einaudi et al. 1981; Hammarstrom et al. 1995). Sulfide minerals, including pyrite are common in mineralized skarns. Some of the environmental issues associated with Cu, Au, and Pb-Zn skarn deposits include (1) waters draining tailings, mine rock piles, or ponds that contain elevated Fe, Al, Pb, Co and Zn, (2) acid drainage, and (3) if these waters contaminate local streams and rivers, aquatic and plant communities can be adversely affected (Hammarstrom et al. 1995).

Application example: New World mine, Montana

The New World mine is 3 miles east of the northeast corner of Yellowstone National Park in Montana, where Au-Cu-Ag mineralization is found in three, carbonate-hosted, massive sulfide replacement deposits (Kirk and Kirk 2004). A porphyry Cu-Au deposit has been found by drilling at depth. Crown Butte Mines Inc. proposed to mine the Miller Creek and Homestake deposits (reported reserves of 7.9 million metric tons of 0.261 oz/short ton Au, 0.74% Cu, and 1.05 oz/short ton Ag) in 1996, but the U.S. federal government purchased the deposit to prevent development and possible contamination of nearby water sources. The federal buyout included funding to close and remediate historic mining in the district.

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Metals found in the district include variable amounts of Al, Fe, Cu, Pb, Cd, and Zn (Kirk and Kirk 2004). Carbonate-hosted deposits should have few if any acid water quality impacts, but the USGS model does predict that acid drainage and elevated concentrations of metals can and do occur in some deposits. Indeed in the New World mining district, same areas are acid generating and metals concentrations are locally elevated. Water samples from two creeks in the mining district are shown in Figure A6.5 and A6.6, and plot in the Cu-skarn ores field. Application of the GEM at the New World mine identified the Cu-skarns as primary sources for acid drainage and control of this drainage should improve water quality in the mining district (Kirk and Kirk 2004). Furthermore, Figures A6.6 and A6.7 illustrate that the dissolved metal content has been diluted by water derived from unmineralized carbonate rocks in water downstream in the lower reaches of the two creeks. Utilization of the Ficklin plot in this area has focused reclamation efforts as well as provided interested parties with the confidence that, in this case, natural dilution will aid in improving the water quality.

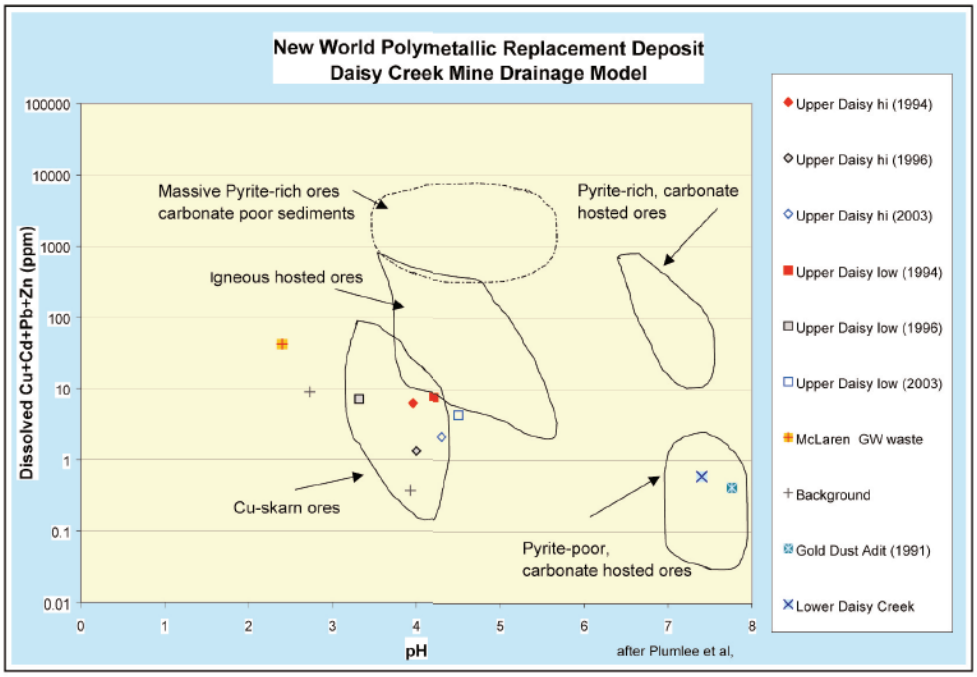


FIGURE A6.5. Water samples from the Daisy Creek plotted on a Ficklin plot (from Kirk and Kirk 2004).

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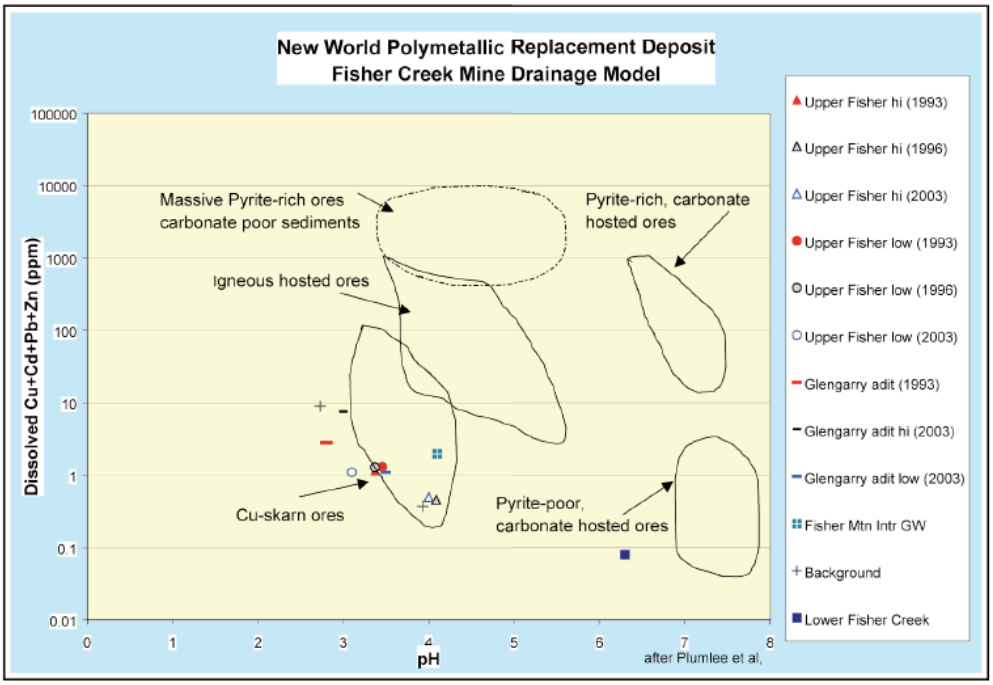


FIGURE A6.6. Water samples from the Fisher Creek plotted on a Ficklin plot (from Kirk and Kirk 2004).

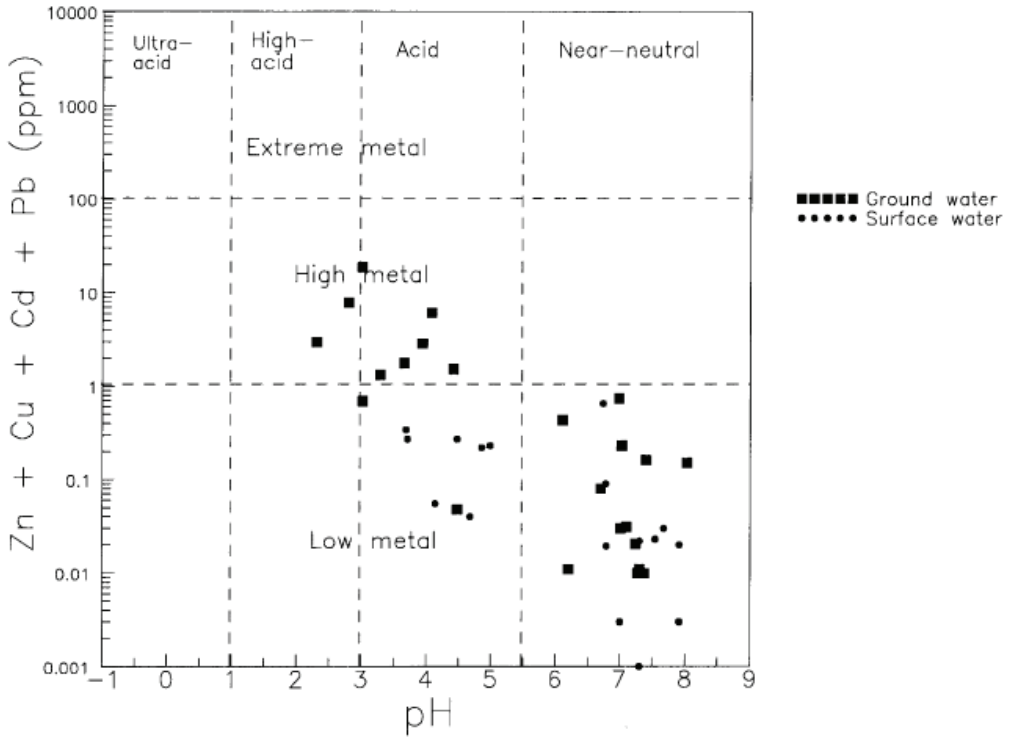


FIGURE A6.7. Ficklin plot of ground and surface water samples from the New World mining district (from Hamarstrom et al. 1995).

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Polymetallic veins

Application example: Hillsboro district, New Mexico

Polymetallic veins are associated with the Copper Flat porphyry copper deposit at Hillsboro in the Animas Mountains in central New Mexico. The Copper Flat quartz-monzonite porphyry stock (CFQM) intruded the vent facies of an andesitic volcanic/intrusive complex and hosts porphyry copper mineralization. Polymetallic veins are associated with latite/quartz latite dikes and both radiate outwards from the CFQM, and are flanked by distal carbonate-hosted Pb-Zn and Ag-Mn replacement deposits to the south and north.

The polymetallic veins are associated with latite/quartz latite dikes and consist of quartz, pyrite, clay, iron oxides, barite, malachite, pseudomalachite, chrysocolla, chalcopyrite, bornite, free gold, galena, and several additional minor minerals (McLemore et al. 1999, 2000). Some veins are as much as 1500 m long and 0.8-3 m wide. They are typically en echelon and pinch and swell. The veins locally grade along strike into an alteration zone of sericite and pyrite with little or no metals concentrations. The veins vary tremendously in chemical composition, but are typically enriched in Au, Ag, Cu, As, Bi, Pb, Zn, and Sb, and depleted in Mo relative to the porphyry copper and carbonate-hosted replacement deposits (McLemore et. al. 1999).

A petrographic and electron microprobe study by Munroe (1999) and Munroe et al. (1999, 2000) found that pyrite oxidation rinds preferentially partition arsenic and some galena grains were surrounded by cerussite, which armored the galena, preventing further oxidation and release of lead. Water samples draining from these vein deposits are near-neutral, low metal waters (Figure A6.8). Compare these analyses with the water samples from the Copper Flat porphyry copper deposit in Figure A6.4.

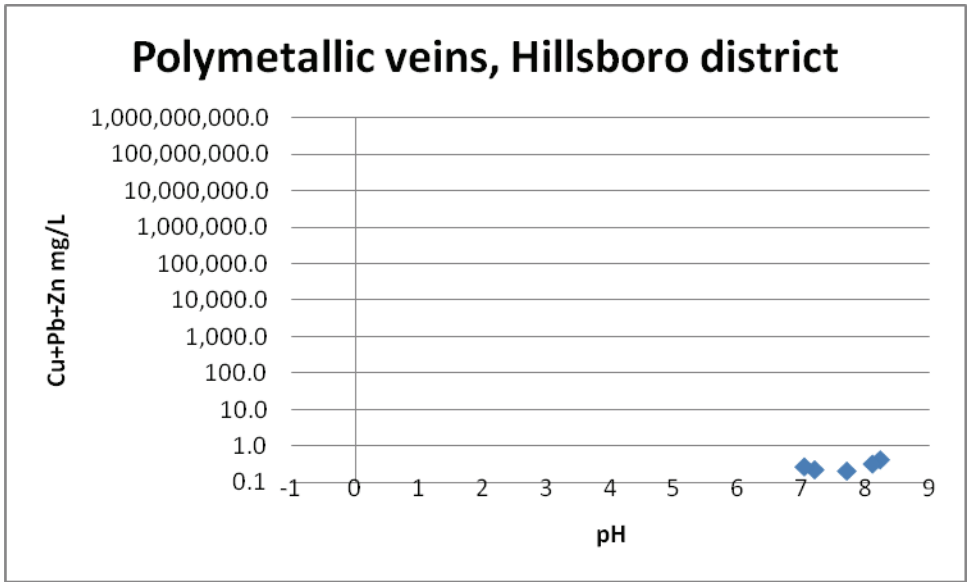


FIGURE A6.8. Modified Ficklin plot of ground and surface water samples from polymetallic vein deposits, Hillsboro mining district, New Mexico (data from Munroe 1999).

FINAL COMMENTS

The mining industry has never been under such scrutiny and oversight as today. As such, GEMs provide a first-order means of predicting environmental issues at mines. It would be helpful if mining companies and government agencies would identify the type of mineral deposit in their reports as well as completely describe the geology and geochemistry of the deposit so

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that the general public and others can easily apply GEMs. However, more research is needed in the application of GEMs. Some mines have provided extensive data that would be useful in refining some of the GEMs, but support and identified priorities are required to compile and interpret these data.

ACKNOWLEDGMENTS

This appendix is part of an on-going study of the mineral resources of New Mexico at NMBGMR, L. Greer Price, Director and State Geologist. Review and comments from Robert Seal and Ron Schmiermund greatly improved this appendix and are appreciated.

REFERENCES CITED

Agency for Toxic Substances and Disease Registries, 2005, Public health assessment for Molycorp, Inc., Questa, Taos County, New Mexico, EPA Facility ID NMD002899094: Agency for Toxic Substances and Disease Registry, Atlanta, 92 p.

Allen, B.D., Groffman, A.R., Molles, M.C., Jr., Anderson, R.Y., and Crossey, L.J., 1999, Geochemistry of the Red River Stream System Before and After Open-Pit mining, Questa Area, Taos County, New Mexico: Final report prepared for the New Mexico Office of the Natural Resources Trustees, Albuquerque, New Mexico 91 p.

Brandvold, L. A. and McLemore, V. T., 1999, A study of the analytical variation of sampling and analysis of stream sediments from mining and milling contaminated areas: Journal of Geochemical Exploration, v. 64, pp. 185-196.

Briggs, P.H., Sutley, S.J., and Livo, K.E., 2003, Questa baseline and pre-mining Ground Water Investigation: 11. Geochemistry of Composited Material from Alteration Scars and Mine-Waste Piles: U. S. Geological Survey, Open-file Report 03-458, 17 p., http://pubs.usgs.gov/of/2003/ofr-03-458/, accessed 8/23/13.

Donahue, K.M., Dunbar, N.W., and McLemore, V.T., 2007, Origins of clay minerals in the Molycorp Mine Goathill North Rock pile, Questa NM: Society of Mining, Metallurgy and Exploration Annual Convention, Denver, Feb. 2007, preprint no. 07-100, 8 p., http://geoinfo.nmt.edu/staff/mclemore/documents/07-100_19.pdf, accessed 8/23/13.

Einaudi, M. T., Meinert, L. D., and Newberry, R. J., 1981, Skarn deposits; *in* Skinner, B. J., ed., 75th anniversary volume: Economic Geology, El Paso, Texas, p. 317-391.

Eppinger, R.G., Briggs, P.H., Dusel-Bacon, C., Giles, S.A., Gough, L.P., Hammarstrom, J.M., and Hubbard, B.E., 2007, Environmental geochemistry at Red Mountain, an unmined volcanogenic massive sulfide deposit in the Bonnifield district, Alaska Range, east-centrl Alaska: Geocheistry: Exploration, Environment, Analysis, v. 7, p. 207-223.

Ficklin, W.H., Plumlee, G.S. and McHugh, J.B., 1992, Geochemical Classification of Mine Drainages and Natural Drainages, *in* Mineralized Areas: Proceedings, 7th International Symposium on Water-Rock Interaction, Park City, Utah, p. 381-384.

Foley, N.K., Seal, R.R., II, Piatak, N.M., and Hetland, B.R., 2010, An occurrence model for the national assessment of volcanogenic beryllium deposits: U.S. Geological Survey Open-File Report 2010-1195, 4 p., http://pubs.usgs.gov/of/2010/1195/, accessed 8/23/13.

Franklin, J.M., Gibson, H.L., Jonasson, I.R., and Galley, A.G., 2005, Volcanogenic massive sulfide deposits: Economic Geology 100th Anniversary Volume, p. 523–560.

Graf, G.J., 2008, Mineralogical and geochemical changes associated with sulfide and silicate weathering, natural alteration scars, Taos County, New Mexico: M. S. thesis, New Mexico Institute of Mining and Technology, Socorro, 193 p.,

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http://geoinfo.nmt.edu/staff/mclemore/documents/Gabriel_Graf_Thesis.pdf, accessed 8/23/13.

Hammarstrom, J.M., Kotlyar, B.B., Theodore, T.G., Elliott, J.E., John, D.A., Doebrich, J.L., Nash, J.T., Carlson, R.R., Lee, G.K., Livo, K.E. and Klein, D.P., 1995, Cu, Au, and Zn-Pb skarn deposits; *in* du Bray, E.A., ed., Preliminary compilation of descriptive geoenvironmental mineral deposit models: U.S. Geological Survey, Open-file Report 95-831, p. 90-111, http://pubs.usgs.gov/of/1995/ofr-95-0831/, ccessed 8/23/13.

Harley, G.T., 1940, The geology and ore deposits of northeastern New Mexico (exclusive of Colfax County): New Mexico Bureau of Mines and Mineral Resources, Bulletin 15, 104 p.

Heikkinen, P.M., Korkka-Niemi, K., Lahti, M., and Salonen, V.-P., 2002, Groundwater and surface water contamination in the area of the Hitura nickel mine, western Finland: Environmental Geology, v. 42, p. 313–329.

Hough, E., Kessler, H., Lelliott, M., Price, S.J., Reeves, H., and Bridge, D., 2003, Look before you leap: the use of geo-environmental data models for preliminary site appraisal; *in* H.M. Moore, H.R. Fox, and S. Elliott, eds., Land reclamation: extending the boundaries: Lisse, A.A. Balkema, p. 369-375, http://nora.nerc.ac.uk/5326/1/houloo.pdf, 8/23/13.

INAP (International Network for Acid Prevention), 2009, Global Acid Rock Drainage (GARD) Guide: International Network for Acid Prevention (INAP), http://www.gardguide.com/index.php/Main_Page, accessed 8/23/13.

John, D.A., Ayuso, R.A., Barton, M.D., Blakely, R.J., Bodnar, R.J., Dilles, J.H., Gray, Floyd, Graybeal, F.T., Mars, J.C., McPhee, D.K., Seal, R.R., Taylor, R.D., and Vikre, P.G., 2010, Porphyry copper deposit model, chap. B *of* Mineral deposit models for resource assessment: U.S. Geological Survey Scientific Investigations Report 2010–5070–B, 169 p., http://pubs.usgs.gov/sir/2010/5070/b/, accessed 823/13.

Jung, M.C., 2008, Contamination by Cd, Cu, Pb, and Zn in mine wastes from abandoned metal mines classified as mineralization types in Korea: Environmental Geochemistry and Health, v. 30, p. 205-217.

Kesler, S.E., 1973, Copper, molybdenum, and gold abundance in porphyry copper deposits: Economic Geology, v. 68, p. 106-112.

Kirk, L.B. and Kirk, A.R., 2004, A geoenvironmental mineral deposit model for the New World polymetallic replacement/skarn deposit: Society for Mining, Metallurgy and Exploration, Preprint 04-122, 6 p.

Kuipers, J.R., Maest, A.S., MacHardy, K.A., and Lawson, G., 2006, Comparison of predicted and actual water quality at hardrock mines: the reliability of predictions in Environmental Impact Statements: Kuipers and Associates and Buka Environmental, 228 p., http://pebblescience.org/pdfs/ComparisonsReportFinal.pdf, accessed 8/23/13.

Krieger, P., 1932a, Geology of the lead-zinc deposits at Pecos, New Mexico-Part I: Economic Geology, v.27, p. 344-404.

Krieger, P., 1932b, Geology of the lead-zinc deposits at Pecos, New Mexico-Part II: Economic Geology, v. 27, p. 450-470.

Kwong, Y.T.J., 1993, Prediction and prevention of acid rock drainage from a geological and mineralogical perspective: Canadian National Hydrology Research Centre Contribution CS-92054, 47 p.

Lovetere, S.H., Nordstrom, D.K., Maest, A.S., and Naus, C.A., 2004, Questa Baseline and Pre-Mining Ground-Water Quality Investigation. 3. Historical Ground-Water Quality for the

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Red River Valley, New Mexico. U.S. Geological Survey Water-Resources Investigations Report 03-4186, 127 p. Questa Baseline and Pre-Mining Ground-Water Quality Investigation. 3. Historical Ground-Water Quality for the Red River Valley, New Mexico, accessed 8/23/13.

Lowell, J.D., 1974, Regional characteristics of porphyry copper deposits of the southwest: Economic Geology, v. 69, p. 601-617.

Lowell, J.D., and Guilbert, J., 1970, Lateral and vertical alteration mineralization zoning in porphyry ore deposits: Economic Geology, v. 65, p. 373-408.

Ludington, S., Plumlee, G., Caine, J., Bove, D., Holloway, J., and Livo, E., 2005, Questa baseline and pre-mining ground-water quality Investigation, 10. Geologic influences on ground and surface waters in the lower Red River watershed, New Mexico: U.S. Geological Survey, Scientific Investigations Report 2004-5245, 46 p.

Ludington, S., and Plumlee, G.S., 2009, Climax-type porphyry molybdenum deposits: U.S. Geological Survey Open-File Report 2009-1215, 16 p., http://pubs.usgs.gov/of/2009/1215/, accessed 8/23/13.

Maest, A.S., Kuipers, J.R., Travers, C.L., and Atkins, D.A., 2005, Predicting water quality at hardrock mines: Methods and models, uncertainties, and state-of-the-art: Kuipers and Associates and Buka Environmental, 90 p., http://www.waterboards.ca.gov/academy/courses/acid/supporting_material/predictwaterqualityhardrockmines1.pdf, accessed 8/23/13.

Maest, A.S., Nordstrom, D.K., and Lovetere, S.H., 2004, Questa Baseline and Pre-Mining Ground-Water Quality Investigation. 4. Historical Surface-Water Quality for the Red River Valley, New Mexico. U.S. Geological Survey, Scientific Investigations Report 2004-5063.

McLemore, V.T., 2009, Geologic Setting and Mining History of the Questa mine, Taos County, New Mexico: New Mexico Bureau of Geology and Mineral Resources, Open-file Report 515, 29 p., http://geoinfo.nmt.edu/publications/openfile/details.cfml?Volume=515, accessed 8/23/13.

McLemore, V. T., Brandvold, L. A., and Brandvold D. K., 1993, A reconnaissance study of mercury and base metal concentrations in water, stream- and lake-sediment samples along the Pecos River in eastern New Mexico: New Mexico Geological Society, Guidebook 44, p. 339-351, http://nmgs.nmt.edu/publications/guidebooks/downloads/44/44_p0339_p0351.pdf, accessed 8/23/13.

McLemore, V. T., Brandvold, L. A., Brandvold D. K., Kirk, K., Popp, C., Hanson, S., Radtke, R., Kyle, P. R., and Hossain, A. M., 1995, A preliminary summary of multidisciplinary studies in the upper Pecos River area, Santa Fe and San Miguel Counties, New Mexico: New Mexico Geological Society, Guidebook 46, p. 331-338, http://nmgs.nmt.edu/publications/guidebooks/downloads/46/46_p0331_p0338.pdf, accessed 8/23/13.

McLemore, V.T., Brandvold, L.A. and Carey, E.A., 2001, Long-term monitoring of the geochemistry of surface water and stream-sediment samples from the upper Pecos River, from the southern Pecos Wilderness to Brantley Dam, North of Carlsbad, eastern New Mexico; year nine; in Geology of the Llano Estacado**:** New Mexico Geological Society Guidebook 52, p. 321-332, http://nmgs.nmt.edu/publications/guidebooks/downloads/52/52_p0321_p0332.pdf, accessed 8//23/13.

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McLemore, V.T., Dickens, A., Boakye, K., Campbell, A., Donahue, K., Dunbar, N., Gutierrez, L., Heizler, L., Lynn, R., Lueth, V., Osantowski, E., Phillips, E., Shannon, H., Smith, M., Tachie-Menson, S., van Dam, R., Viterbo, V.C., Walsh, P., and Wilson, G.W., 2008, Characterization of Goathill North Rock Pile: New Mexico Bureau of Geology and Mineral Resources, Open-file report 523, http://geoinfo.nmt.edu/publications/openfile/details.cfml?Volume=523, accessed 8/23/13.

McLemore, V., Heizler, L., Donahue, K., and Dunbar, N., 2009, Characterization of Weathering of Mine Rock Piles: Example from the Questa Mine, New Mexico, USA: Securing the Future and 8th ICARD, June 23-26 2009, Skelleftea, Sweden, conference proceedings, 10 p., http://www.proceedings-stfandicard-2009.com/pdfer/Virginia_McLemore_P_T2_Characterization-of-weathering-of-mine-rock-piles-example-from-the-Questa-mine-New-Mexico-USA.pdf, accessed 8/23/13.

McLemore, V. T., Munroe, E. A., Heizler, M. T. and McKee, C., 1999, Geochemistry of the Copper Flat porphyry and associated deposits in the Hillsboro mining district, Sierra County, New Mexico, USA: Journal of Geochemical Exploration, v. 66, p. 167-189.

McLemore, V.T., Munroe, E.A., Heizler, M.T., and McKee, C., 2000, Geology and evolution of the mineral deposits in the Hillsboro District, Sierra County, New Mexico. *In* J.K. Cluer et.al (ed). Geology and Ore Deposits 2000: The Great Basin and Beyond: Geological Society of Nevada Symposium, Reno/Sparks, May 2000, p 643-649.

Munroe, E. A., 1999, Geology and geochemistry of waste rock piles in the Hillsboro mining district, Sierra County, New Mexico (M. S. thesis): New Mexico Institute of Mining and Technology, Socorro, 144 p, http://www.ees.nmt.edu/outside/alumni/papers/1999t_munroe_ea.pdf, accessed 8/23/13.

Munroe, E. A., McLemore, V. T., and Kyle, P., 1999, Waste rock pile characterization, heterogeneity and geochemical anomalies in the Hillsboro mining district, Sierra County, New Mexico: Journal of Geochemical Exploration, v. 66, p. 391-405.

Munroe, E. A., McLemore, V. T., and Dunbar, N. W., 2000, Mine waste rock pile geochemistry and mineralogy in southwestern New Mexico, USA; *in* ICARD 2000—Proceedings from the 5th international conference on acid rock drainage: Society for Mining, Metallurgy, and Exploration, Inc., Littleton, Colo., p. 1327-1336.

Naus, C.A., McCleskey, R.B., Nordstrom, D.K., Donohoe, L.C., Hunt, A.G., Paillet, F.L., Morin, R.H., and Verplanck, P.L., 2005, Questa baseline and pre-mining groundwater quality investigation. 5. Well installation, water-level data, and surface- and ground-water geochemistry in the Straight Creek drainage basin, Red River valley, New Mexico, 2001-03: United States Geological Survey, Scientific Investigations Report 2005-5088, 228 p.

Nordstrom, D.K., 2007, Questa baseline and pre-mining ground-water quality investigation. 25. Summary of results and baseline and pre-mining ground-water geochemistry, Red River Valley, Taos County, New Mexico, 2001-2005: U.S. Geological Survey Professional Paper 1728, p. 183.

Nordstrom, D.K., McCleskey, R.B., Hunt, A.G., and Naus, C.A., 2005, Questa baseline and pre-mining groundwater quality investigation. 14. Interpretation of ground-water geochemistry in catchments other than the Straight Creek catchment, Red River valley, Taos County, New Mexico, 2002-2003: United States Geological Survey, Scientific Investigations Report 2005-5050, 94 p.

Odor, L., Wanty, R.B., Horvath, I., Fugedi, U., 1999, Environmental signatures of mineral deposits and areas of regional hydrothermal alteration in northeastern Hungary,

Copyright © 2014 Society for Mining, Metallurgy & Exploration Inc. All rights reserved. Electronic edition published 2014.

Geologica Hungarica, Tomus 24, p. 107-129. http://crustal.usgs.gov/projects/gem/pdfs/wanty3.pdf, accessed 8/23/13.

Plumlee, G.S., 1999, The Environmental Geology of Mineral Deposits; *in* G.S. Plumlee and M.J. Logsdon, Eds., The Environmental Geochemistry of Mineral Deposits, Part A: Processes, Techniques and Health Issues: Reviews in Economic Geology Vol. 6A, Society of Economic Geologists, Inc., p. 71-116.

Plumlee, G.S., Smith, K.S., and Ficklin, W.H., 1994, Geoenvironmental models of mineral deposits, and geology-based mineral-environmental assessments of public lands: U.S. Geological Survey, Open File Report 94-203, 9 p.

Plumlee, G.S., Smith, K.S., Montour, M.R., Ficklin, W.H., and Moiser, E.L., 1999, Geologic controls on the composition of natural waters and mine waters draining diverse mineral-deposit types; in L.H. Filipek and G.S. Plumlee, eds., The Environmental Geochemistry of Mineral Deposits, Part B: Case Studies and Research Topics: Reviews in Economic Geology Vol. 6B, Society of Economic Geologists, Inc., p. 373-432.

Price, J.G., Shevenall, L., Henry, C.D., Rigby, J.G., Christensen, L.G., Lechler, P.J., Desilets, M.O., Fields, R., Driesner, D., Durbin, B., and Lombardo, W., 1995, Water quality at inactive and abandoned mines in Nevada: Nevada Bureau of Mines and Geology, Open-file report 95-4, 73 p., http://www.nbmg.unr.edu/amr/amr.pdf, accessed 8/23/13.

Raugust, J.S., 2003, The natural defenses of Copper Flat, Sierra County, New Mexico, New Mexico Bureau of Geology and Mineral Resources Open File Report No. 475, New Mexico Bureau of Geology and Mineral Resources, Socorro, New Mexico, 485 p., http://geoinfo.nmt.edu/publications/openfile/downloads/OFR400-499/451-475/475/ofr_475.pdf, accessed 8/23/13.

Raugust, J.S. and McLemore, V.T., 2005, The Natural Defenses of Copper Flat, Sierra County, New Mexico: Reclamation Matters, Spring/Summer 2005, p. 18-23, http://www.asmr.us/Publications/ReclamationMatters/2005/SpringSummer2005.pdf, accessed 8/23/13.

Riesmeyer, W.D., 1978, Precambrian geology and ore deposits of the Pecos mining district, San Miguel and Santa Fe Counties, New Mexico (unpub. M.S. thesis): Albuquerque, University of New Mexico, 215 p.

Riesmeyer, W.D. and Robertson, J.M., 1979, Precambrian geology and ore deposits of the Pecos mine, San Miguel County, New Mexico: New Mexico Geological Society, p. 175-179, http://nmgs.nmt.edu/publications/guidebooks/downloads/30/30_p0175_p0179.pdf, accessed 8/23/13.

Robertson GeoConsultants Inc, 2000a. Interim Background Characterization Study, Questa Mine, New Mexico. RGC Report 052008/6.

Robertson GeoConsultants, Inc., 2000b, Background study data report, Questa Mine, New Mexico: Robertson GeoConsultants, Inc. Report 052008/12, January.

Sangster, D.F. and Scott, S.D., 1976, Precambrian, strata-bound, massive Cu-Zn-Pb sulfide ores of North America; *in* Wolf, K.H. ed., Handbook of strata-bound and stratiform ore deposits: Elsevier Scientific Publishing Co., Amsterdam, v. 6, p. 129-222.

Schmiermund, R.L., Lazo, C., and Parnow, C., 2006, Application of Geo-Environmental Models to Accelerated EIA and Permitting Processes for an Andean Porphyry Cu-Au Deposit: 7th International Conf. on Acid Rock Drainage: St. Louis, MO, 9 p., http://www.imwa.info/docs/imwa_2006/1903-Schmiermund-CO-1.pdf accessed 8/23/13.

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Schmiermund, R.L., and Ranville, J.F., 2006, Geo-Environmental and Related Models in the Life Cycle of Mine Development–A Flowchart for Integration. Poster w. Extended Abstract, 7th International Conf. on Acid Rock Drainage: St. Louis, MO.

Schulz, K.J., Chandler, V.W., Nicholson, S.W., Piatak, Nadine, Seall, II, R.R., Woodruff, L.G., and Zientek, M.L., 2010, Mamatic sulfide-rich nickel-copper deposits related to picrite and (or) tholeiitic basalt dike-sill complexes—A preliminary deposit model: U.S. Geological Survey Open-File Report 2010–1179, 25 p.

Seal, R.R. and Foley, N.K. (eds.), 2002, Progress on geoenvironmental models for selected mineral deposit types: U.S. Geological Survey Open-File Report 2002-195, 213 p., http://pubs.usgs.gov/of/2002/of02-195/, accessed 8/23/13.

Seal II, R.R., Foley, N.K., and Wanty, R.B., 2002, Introduction to Geoenvironmental Models of Mineral Deposits; *in* R.R. Seal II and N.K. Foley, Eds., Progress on Geoenvironmental Models for Selected Mineral Deposit Types: U.S. Geological Survey Open-File Report 02-195.

Seal II, R.R. and Hammarstrom, J.M., 2003, Geoenvironmental Models of Mineral Deposits: Examples from Massive Sulfide and Gold Deposits; *in* J.L. Jambor, D.W. Blowes and A.I.M. Ritchie, Environmental Aspects of Mine Wastes: Short Course Series Vol. 31, Mineralogical Association of Canada, p. 11-50.

Seal, R.R. II, Hammarstrom, J.M., Foley, N.K., and Alpers, C.N., 2000, Geoenvironmental models for seafloor base- and precious-metal massive sulfide deposits: ICARD2000 Proceedings from the Fifth International Conference on Acid Rock Drainage, p. 151-160.

Shanks, W.C.P., III, Dusel-Bacon, Cynthia, Koski, Randolph, Morgan, L.A., Mosier, Dan, Piatak, N.M., Ridley, Ian, Seal, R.R., II, Schultz, K.J., Slack, J.F., Thurston, Roland, 2009, A new occurrence model for national assessment of undiscovered volcanogenic massive sulfide deposits: U.S. Geological Survey Open-File Report 2009–1235, 27 p.

Shaw, S., Wels, C., Robertson, A., Fortin, S., and Walker, B., 2003, Background characterisation study of naturally occurring acid rock drainage in the Sangre de Cristo Mountains, Taos County, New Mexico; in 6th ICARD: Cairns, Queensland, Australia, July 12-18, p. 605-616, http://www.robertsongeoconsultants.com/papers/icard03back.pdf, accessed 8/23/13.

SRK Consulting Inc., 2009, Environmental audit of the Greens Creek mine, Final Report: report to State of Alaska, 225 p., http://dnr.alaska.gov/mlw/mining/largemine/greenscreek/pdf/gcaudit2009.pdf, accessed on 8/23/13

Stott, C.E., 1931, Geology of the Pecos mine: Engineering and Mining Journal, v. 131, p. 270-275.

Taylor, C.D., Philpotts, J., Premo, W.R., Meier, A.L., and Taggart, Jr., J.E., 2010a, The Late Triassic metallogenic setting of the Greens Creek massive sulfide deposit in southeastern Alaska; *in* Taylor, C.D., and Johnson, C.A., editors, Geology, geochemistry, and genesis of the Greens Creek massive sulfide deposit, Admiralty Island, southeastern Alaska: U.S. Geological Survey Professional Paper 1763, p. 8-64.

Taylor, C.D., Lear, K.G., and Newkirk, S.R., 2010b, A genetic model for the Greens Creek polymetallic massive sulfide deposit, Admiralty Island, southeastern Alaska; *in* Taylor, C.D., and Johnson, C.A., editors, Geology, geochemistry, and genesis of the Greens Creek massive sulfide deposit, Admiralty Island, southeastern Alaska: U.S. Geological Survey Professional Paper 1763, p. 417-429.

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Taylor, C.D., Zierenberg, R.A., Goldfarb, R.J., Kilburn, J.E., Seal, II, R.R., and Kleinkopf, M.D., 1995, Volcanic-associated massive sulfide deposits; *in* E.A. de Bray, ed. Preliminary compilation of descriptive geoenvironmental mineral deposit models: U.S. Geological Survey, Open-file Report OF 95-831, p. 137-144.

USEPA (U.S. Environmental Protection Agency), 1999, Technically enhanced naturally occurring radiaoactive materials in the southwestern copper belt of Arizona: U.S. Environmental Protection Agency Report EPA 402-R-99-002, 124 p., http://www.epa.gov/radiation/docs/tenorm/402-r-99-002.pdf, accessed 8/23/13.

Wanty, R.B., Berger, B.R., and Plumlee, G.S., 1999, Environmental models of mineral deposits-the state of the art: (1999) Geologica Hungarica, Tomus 24, pp. 97-106, http://crustal.usgs.gov/projects/gem/pdfs/wanty2.pdf, accessed 8/23/13.

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Management Technologies for Metal Mining Influenced Water

Sampling and Monitoring for the Mine Life Cycle

Volume 6

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