

Chapter C

TOOLKIT FOR THE RAPID SCREENING AND CHARACTERIZATION OF WASTE PILES ON ABANDONED MINE LANDS

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INTRODUCTION

There are thousands of historical mine-waste piles present on inactive metal-mining sites, some of which are on Federal lands and have been abandoned. Assessment of metal mobility, acid-drainage production, and toxic effects from the weathering of historical mine-waste piles is an area of growing need as the environmental effects of inactive mine-waste sites across the country are being evaluated and mitigated. The U.S. Geological Survey Mine Waste Characterization Project has taken a multidisciplinary approach to assemble, develop, and refine methods and tools for characterizing and screening weathered solid-mine wastes. Researchers from a variety of disciplines, including geophysics, geochemistry, analytical chemistry, geology, mineralogy, remote sensing, and spatial modeling, have worked together at metal-mining waste sites in Colorado and New Mexico to develop an integrated "toolkit" for the rapid screening and characterization of historical mine-waste piles. Tools developed from this work can be used in ranking and prioritizing historical mine-waste piles. The following is a brief discussion of some of these tools.

SAMPLING AND GEOCHEMICAL SCREENING TOOLS

Sampling Strategy

A statistically based, cost-effective sampling strategy was developed that could provide the foundation for screening and prioritizing mine-waste piles on a regional or watershed basis. Because average properties generally are the emphasis in screening and prioritization, the sampling strategy entails collection of a composite sample from each waste pile. One 30-increment dump-composite sample collected using this sampling strategy contains as much information, relative to the average value, as 30 individual grab samples at $1/30$ of the analytical cost. When more detailed site characterization is required, other sampling strategies might be employed, depending on the objectives of the work.

A detailed discussion of the sampling strategy is given in K.S. Smith and others (2000). In order to minimize sampling errors, this strategy requires that a composite sample consist of at least 30 increments (subsamples). Hence, a mine dump is divided into at least 30 cells of roughly equal surface area. Multiple (>30) surficial samples of roughly equal mass are collected from each cell with a trowel, successively placed in a bucket, and mixed to create a mine-dump composite sample. The sample is air-dried and dry-sieved to < 2 mm. The resulting < 2 mm mine-dump composite sample should weigh at least one kilogram. For screening and prioritizing historical mine-waste piles, use of the < 2 mm size fraction generally should provide a worst-case scenario for metal leachability and appears to be a good choice to reduce the sampling error and reduce the sample size to one reasonable for reconnaissance field collection.

A main goal of this sampling strategy is to minimize sampling errors. Sampling errors can be categorized into seven major groups: fundamental error, grouping and segregation error, delimitation error, extraction error, preparation error, cycles, and trends. Pitard (1993) provides a detailed discussion of sampling errors and serves as the basis of this sampling strategy. The fundamental error (FE) is often the main source of sampling error and results from the compositional heterogeneity of particles. It cannot be eliminated, but it can be estimated prior to sampling. Based on estimates of FE, steps can be taken to minimize it and thus minimize the overall sampling error. Important factors in the FE include heterogeneity, particle size, and sample mass. If the population is very heterogeneous or the particle size is large, then more sample mass is required to minimize the FE. If the mass of the population (lot), M_L , is greater than ten times the mass of the sample, the FE can be estimated by the following equation:

$$FE^2 = \frac{clfgd^3}{M_S}$$

where

FE^2 is a relative variance

M_S is the sample mass (g)

c is the mineralogical factor

l is the liberation factor

f is the shape factor

g is the granulometric factor

d is the maximum particle size (cm)

The mineralogical factor, c , is the maximum heterogeneity generated by the constituent of interest in the target population and can be estimated by dividing the approximate density of the material (g/cm^3) by the average concentration of the constituent of interest (mg/kg). The liberation factor, l , is a correction factor for c that takes into account incomplete liberation of the constituent of interest, and ranges from 0, when there is no liberation, to 1, when there is complete liberation. Values for the shape factor, f , vary from 0.2 for flakes to 10 for needles, with a value of 0.5 representing a roughly spherical shape. The granulometric factor, g , is a correction factor when all the particles are not the same size, with noncalibrated material having a value of 0.25, and calibrated material having a value of 0.55.

The other main sampling error of concern is the grouping and segregation error (GSE). To minimize the grouping factor of the GSE, it is necessary to collect as many small increments as practically possible, assuming that sample collection and preparation are properly carried out. (An increment is a group of particles collected from a population with a single operation of the sampling device.) Pitard (1993, p. 187) states that a sample should be made up of at least 30 increments. Minimizing the segregation factor of the GSE is much more difficult because it involves aspects such as differences in particle density, particle size, particle shape, mine-dump construction, etc. The segregation factor should be kept in mind when collecting samples.

Other sources of sampling error generally are less important than the FE or GSE. Delimitation error and extraction error are both related to the choice and use of sampling tools; collectively they are termed the materialization error. Preparation errors take place after sample collection and before analysis. These errors encompass such factors as sample preservation, contamination, loss, sieving, etc. Both cycles and trends relate to changes in the concentration of an analyte of interest with respect to time or space.

Metal Leachability Determination --A Field Method

Leaching tests are one of the main screening procedures used to evaluate and prioritize mine-waste piles. A leaching test has been developed that can be performed easily in the field, can provide on-site pH and conductivity information (for field prioritization), and can furnish samples for elemental analysis. Hageman and Briggs (2000) describe the Field Leach Test (FLT) in detail. It is based on the premise that the most chemically reactive material in weathered mine waste consists of relatively soluble components in the fine fraction (< 2 mm) of the waste. In general, higher concentrations of chemical constituents are leached from the smaller size fractions in the weathered mine-waste piles studied. The choice of the < 2 mm size fraction may tend to slightly overestimate the leachability of the mine-waste material as a whole, but this size-fraction cutoff does not appear to "miss" any readily leachable phases (K.S. Smith and others, 2000).

The FLT involves combining 50.0 grams of a < 2 mm mine-dump composite sample with 1,000 grams of deionized water in a capped one-liter polyethylene bottle. The mixture is vigorously shaken for five minutes, and then allowed to settle for 10 minutes. Specific conductance and pH can be measured in the field, and subsamples can be filtered and preserved for analysis of chemical constituents. Hageman and Briggs (2000) showed that results from the FLT reveal geochemical trends that correlate favorably with results from the Synthetic Precipitation Leaching Procedure (SPLP, EPA-1312, U.S. Environmental Protection Agency, 1994). Table 1 and figure 1 provide a comparison of leaching results from the FLT and the SPLP.

Net Acid Production Determination

There are a variety of methods to determine the potential of a mine-waste material to generate acid. For historical metal-mining waste piles that consist of weathered material, it is necessary to adopt a method that incorporates the potential acid production of both primary and secondary minerals, and the potential acid-consuming capacity of host-rock minerals (e.g., carbonates, chlorite, biotite). Primary minerals constitute the original ore and gangue assemblages of the mineral deposit, and secondary minerals are those that form upon oxidation and weathering of the primary minerals.

Table 1. Comparison of results from the Field Leach Test and the Synthetic Precipitation Leaching Procedure for specific conductance and pH in leachates of < 2 mm fraction mine-dump composites from sites in Colorado and New Mexico. Data are from Hageman and Briggs (2000). [SPLP = Synthetic Precipitation Leaching Procedure; FLT = Field Leach Test]

Mine-Dump Site	pH		Specific Conductance ($\mu\text{S}/\text{cm}$)	
	SPLP	FLT	SPLP	FLT
Venir	2.7	2.9	1,080	760
Sunday #2	2.8	3.0	810	620
Yukon	3.1	3.2	660	530
Mayday	3.5	3.6	200	120
Carlisle	5.4	5.1	500	390
Tucson	7.0	6.7	850	550
Main Iron Incline	8.5	7.8	230	160
Petroglyph	8.8	9.5	85	41

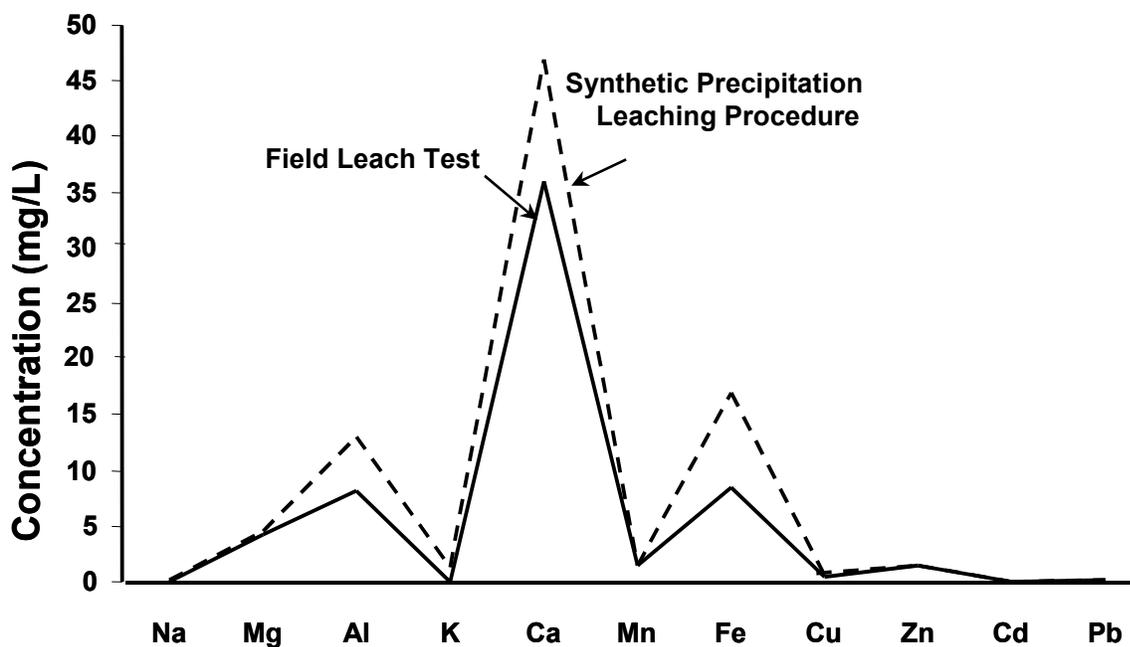


Figure 1. Comparison of leaching results of a < 2 mm fraction mine-dump composite from the Yukon mine near Silverton, Colorado. The dashed line represents results from the Synthetic Precipitation Leaching Procedure and the solid line represents results from the Field Leach Test. Modified from Hageman and Briggs (2000).

The net acid production (NAP) method of Lapakko and Lawrence (1993) has been slightly modified for use with weathered mine-waste piles. In this method, a 1.0-gram sample of pulverized (minus 200 mesh) waste material is digested with a heated solution of 30% hydrogen peroxide for one hour, cooled, and filtered. Acidic filtrates are titrated to pH 7 with NaOH, and NAP is calculated in terms of kilograms CaCO_3 per metric ton of waste. It is possible to perform this method in the field if necessary. Fey and others (2000) demonstrated that mine-waste piles

segregate into different groups when NAP is plotted against either the sum of five leachable metals (arsenic, cadmium, copper, lead, and zinc) or against leachable iron from a number of waste piles from polymetallic deposit types. The choice of leachable metals to plot against NAP can be adapted for the mineral deposit type(s) of interest. This approach can be used to rank the potential of mine-waste material to degrade water quality.

NON-INVASIVE SCREENING TOOLS

Imaging Spectroscopy

Imaging spectroscopy is a tool that can be used to map specific minerals by detecting distinctive chemical bonds within their structure. For remote sensing purposes, imaging spectroscopy can be used to map minerals contained in exposed surficial materials. One of the imaging spectroscopy sensors is the NASA/JPL AVIRIS system (Airborne Visual and Infra-Red Imaging Spectrometer). AVIRIS is flown on an ER-2 (U2) aircraft at 65,000 feet (19,800 meters) and yields a 17-meter pixel spacing with a 10.5-km swath (improvements in pixel spacing are expected soon). AVIRIS information can be analyzed using the USGS Tetracorder (Clark and others, 1995), which is an expert system that utilizes a database of more than 300 laboratory spectra and picks the best spectral match. Imaging spectroscopy also can be used for hand specimens and for on-site characterization using portable field spectrometers (spatial resolution in the millimeter to several meter range); future uses from satellites are planned. For additional information about imaging spectroscopy, see <<http://speclab.cr.usgs.gov>>.

Swayze and others (2000a, 2000b) have demonstrated that AVIRIS, combined with Tetracorder, can be used to map spectrally dominant iron-bearing minerals resulting from weathering of acidic mine-waste material. This approach is based on identifying iron-bearing secondary minerals, such as jarosite, goethite, and hematite (figure 2), that form on the surface of mine-waste piles as products of the oxidation of sulfide minerals. The best indicator mineral for acidic conditions appears to be jarosite. This approach can be used to screen large areas for potentially acidic mine-waste material and to identify sites that warrant closer examination. Figures 3 and 4 demonstrate how pH and specific conductance of leachates correspond to spectral zones along a traverse of a mine-waste dump. Hand-held spectrometers can be used for on-site work. Dalton and others (2000) have used imaging spectroscopy to map the distribution of acid-generating and acid-neutralizing minerals in a mineralized watershed.

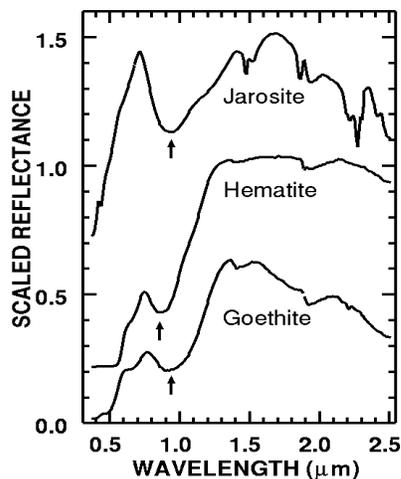


Figure 2. Spectra of some iron-bearing secondary minerals. Note the different shapes of the spectra, which are diagnostic for mineral identification. After Dalton and others (2000).

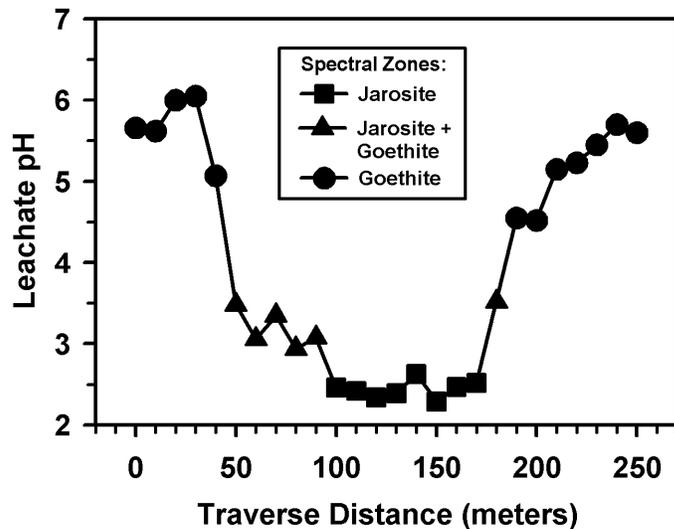


Figure 3. Surficial mine-waste leachate pH versus distance along a mine-waste pile traverse. Each spectral mineral zone is denoted by a different symbol. Note that the pH is lowest in the jarosite spectral zone. After Swayze and others (2000b).

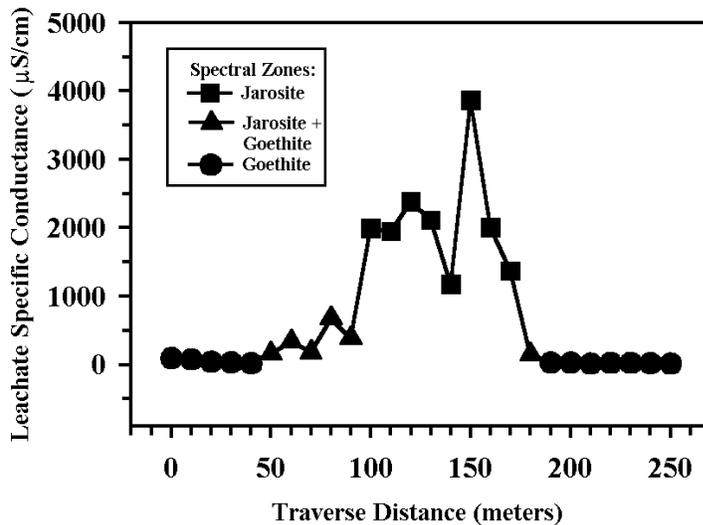


Figure 4. Surficial mine-waste leachate specific conductance along a mine-waste pile traverse. Note that jarosite zone leachates have higher specific conductance values than do leachates from the other spectral mineral zones. Specific conductance closely tracks sulfate concentrations in the leachates. After Swayze and others (2000b).

Geophysical Methods

Combining geological mapping with airborne geophysical surveying facilitates the task of screening large areas to locate historical mine dumps and assigning them initial priorities for further study. Airborne techniques include radiometric, magnetic, and electromagnetic mapping and can be used to map subsurface lithology, structure, and ground-water flow. B.D. Smith and others (2000) illustrated how airborne geophysical techniques can be applied at both a regional scale (e.g., state) and a local scale (e.g., watershed, mining district).

Geoelectrical methods, such as direct current resistivity, electromagnetic, and induced polarization, can be used to study conditions at depth within particular mine-waste piles. These methods can infer information about lithology, mineralogy (especially sulfide minerals), pore-water saturation, or location of pore water or groundwater containing high total-dissolved solids. Hence, these methods can be used to trace plumes of contaminated water and discern compositional variations, including pH, in mine-waste piles. Campbell and Fitterman (2000) and Campbell and others (1999) provided an overview of geophysical methods in mine-waste characterization. Table 2 summarizes the potential use of geophysical methods in mine-waste studies.

Table 2. Potential uses of geophysical methods in mine-waste characterization. Modified from Campbell and Fitterman (2000).

Method	Measures	Caused By	Mine-Waste Applications
EM (Frequency-Domain Electromagnetics)	Electrical conductivity (mS/m)	Ground water, lithology	Tracing AMD plumes
DC (Direct-Current Resistivity)	Electrical resistivity (Ohm-m)	Ground water, lithology	Shallow (<10 m) water tables in, and bottoms of, shallow (<20 m) waste dumps
TEM (Time-Domain Electromagnetics)	Electrical conductivity (mS/m)	Ground water, lithology	Deeper (10-30 m) water tables in waste dumps
CSAMT (Controlled-Source Audio Magnetotellurics)	Electrical resistivity (Ohm-m)	Ground water, lithology	Deeper (10-50 m) bottoms of waste dumps
IP (Induced Polarization)	Electrical chargeability (usually mV-sec/V)	Electrochemical reactions at grain surfaces	Concentrations of sulfides in waste dumps
SP (Spontaneous Polarization)	Ground voltages (mV)	Redox and streaming potentials	Experimental (no proven successes)
GPR (Ground-Penetrating Radar)	Speed of electromagnetic radiation (cm/ns)	Ground textures and included pore water	Possibly monitoring plume remediation
Magnetics	Magnetic field (nT)	Magnetization	Ferrous junk (e.g., rebar) in mine dumps (other mine-waste applications unproven)
Seismic	Acoustic wave velocity (m/s)	Compaction and ground water content	Tracing bottoms and edges of waste dumps, and basement under plume areas (all mine-waste applications are still in experimental stages)

SITE CHARACTERIZATION TOOLS

In the study of the potential impact of a mine-waste pile on the environment, it is important to understand the source(s) of possible metal contaminants, the processes controlling their release into the environment, and their transport mechanisms (Plumlee and others, 1999). These processes are related to the susceptibility and availability of the resident mineral phase(s) of the contaminant(s) to alteration, weathering, and dissolution reactions (Smith and Huyck, 1999). Understanding these processes is key to making informed decisions about mitigation, cleanup, disposal, and remediation of mined sites.

Mineralogy Determination by X-Ray Diffraction

X-ray diffraction (XRD) methods (described in the previous chapter) can be used to identify mineral phases in mine-waste samples. This information can be combined with bulk chemical analysis to try to account for residence phase(s) of chemical constituents. However, weathered mine-waste material commonly contains a significant portion (often as high as 40-50%) of non- or poorly crystalline material that is amorphous and undetectable by XRD techniques. This amorphous material likely contributes to the mobility of some constituents. Hence, it is often necessary to supplement XRD methods with other methods, such as sequential chemical extractions (see below) or microanalysis techniques (see previous chapter) that can identify and assess the amorphous phases in the mine-waste material.

The limit of detection for XRD is around 1 to 5%, depending on the mineral phase and sample composition. Many secondary minerals resulting from the weathering of sulfide minerals can comprise less than 1% of the whole rock, yet can be important in metal transport or acid generation. An example is efflorescent salts (see previous

chapter), which often are below the XRD limit of detection in whole-rock samples and which commonly have a large influence on drainage-water quality.

Metal Partitioning Determination by Sequential Chemical Extractions

In sequential chemical extractions, constituents are extracted from some or all of operationally defined solid phases through reaction with a sequence of solvents (Chao, 1984). The sequence of extractions exposes the sample to increasingly rigorous chemical treatments. This provides a means for evaluating the availability and potential mobility of metals extracted from the various phases. There are many operationally defined sequential-extraction schemes. Leinz and others (1999, 2000) have adopted the following scheme for weathered mine-waste material:

1. Water-soluble constituents -- A 0.25-g sample, with 0.25 g silica gel added, is extracted with 25 mL deionized water in a 50-mL centrifuge tube for two hours in a horizontal reciprocating shaker at ambient temperature.
2. Ion-exchangeable constituents -- The residue from step 1 is extracted with 25 mL of 1 M sodium acetate for one hour in a horizontal reciprocating shaker at ambient temperature.
3. Constituents associated with carbonates -- The residue from step 2 is extracted with 25 mL of 1 M sodium acetate buffered to pH 5 (using acetic acid) for two hours in a horizontal reciprocating shaker at ambient temperature. (This extraction mostly pertains to calcite and magnesite. Some carbonates, such as siderite, are minimally extracted.)
4. Constituents associated with manganese oxides and amorphous iron oxides -- The residue from step 3 is extracted with a 25 mL of 0.25 M solution hydroxylamine hydrochloride in 0.25 M hydrochloric acid for 30 minutes in a 50°C water bath.
5. Constituents associated with crystalline iron oxides -- The residue from step 4 is extracted with 25 mL of 4 M hydrochloric acid for 30 minutes at 94°C in a boiling water bath.
6. Constituents occurring as or in sulfide minerals -- The residue from step 5 is extracted with 2 g of sodium chlorate followed by the careful addition of 10 mL of concentrated hydrochloric acid. The extraction is allowed to continue for 45 minutes at room temperature. Following separation, the aqueous phase is diluted to 25 mL with deionized water and the residue is treated with 4 M nitric acid for 40 minutes in a boiling water bath. The two extracts are analyzed separately and the results combined.
7. Constituents occurring as or in silicate minerals -- The residue from step 6 is transferred to a Teflon beaker and digested with 10 mL each of concentrated nitric acid, perchloric acid, and hydrofluoric acid at 220°C, to a moist bead. The moist bead is treated with 25 mL of 4 M hydrochloric acid for 30 minutes at 100°C. The final volume is adjusted to 25 mL with deionized water.

Isolation and Enumeration of Iron- and Sulfur-Oxidizing Bacteria

Iron- and sulfur-oxidizing bacteria enhance the degradation of sulfide minerals. A determination of their numbers can provide an estimate of the contribution of biological activity to mine-waste decomposition and acid-drainage generation. Bacteria of the genus *Thiobacillus* have long been recognized as active agents in the formation of acid-mine drainage. At acidic pH, these bacteria can catalyze iron-oxidation reactions nearly 10^6 times faster than is possible through inorganic mechanisms alone.

Although generally considered ubiquitous in the acid-mine drainage environment, microbiological techniques must be used to confirm the presence of thiobacilli. Newer molecular-based techniques to isolate and identify microbes are rapidly being developed. However, most studies have relied on “classical” microbiological methods to identify the presence of thiobacilli in sediments and waters associated with mine wastes. Such methods employ media specially formulated to meet growth and nutritional needs of the microbes of interest. After the initial isolation of the microbes is complete, additional tests can be run to characterize the biochemistry of the organisms, and laboratory simulation experiments can be run to estimate microbial action upon the mine-waste material.

Thiobacillus ferrooxidans and *Thiobacillus thiooxidans* were isolated from the Mayday mine dump near Silverton, Colorado, using the MPN (most probable number) culture technique (Stanton, 2000). An initial set of MPN tubes using small volumes of inoculum was used to assess and select core samples for detailed study and enumeration of cell numbers. For Mayday drill core 1, an additional MPN test was run using inocula of 10, 1, and 0.1 mL into prepared media. Cell numbers of approximately 1000/g were obtained from some samples from the Mayday mine dump (Table 3).

Table 3. Results from microbiological most probable number tests and selected elemental concentrations for cored samples from the Mayday mine-waste pile near Silverton, Colorado. [NA = not analyzed; depth is the midpoint of the interval]

Sample Number	Depth (ft.)	Cu (ppm)	Pb (ppm)	Zn (ppm)	Total C (%)	Total S (%)	<i>Th. ferro.</i> per g	<i>Th. thio.</i> per g
H1-C1-T	0.8	100	2,200	730	0.17	1.5	210	2
H1-C1-B	2.4	40	400	90	0.10	0.54	1,100	2
H1-C2-T	4.3	30	270	230	0.73	0.36	9	460
H1-C2-M	6.0	870	32,100	27,100	1.08	4.34	1,100	4
H1-C2-B	7.64	4,600	8,600	5,900	0.14	9.14	210	75
H1-C3-T	9.75	25	60	160	0.02	1.91	21	9
H1-C3-B	11.12	50	100	150	0.05	1.09	1,100	210
H1-C4	15.1	45	130	130	0.05	2.0	NA	NA
H1-C5	17.5	160	1,450	880	0.15	1.02	NA	NA

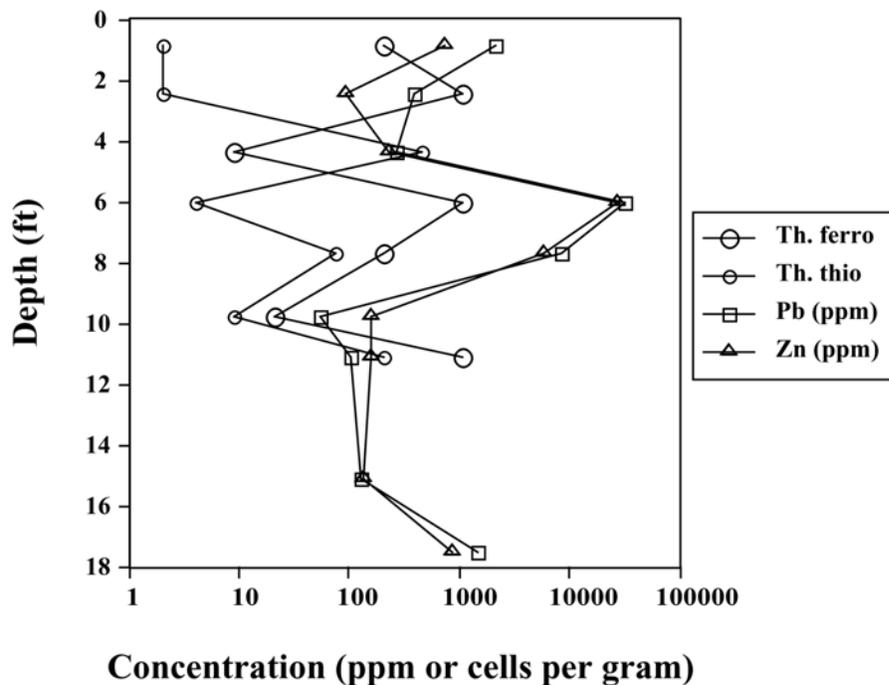


Figure 5. Plot of microbiological data (*Thiobacillus ferrooxidans* and *Thiobacillus thiooxidans* counts) and lead and zinc concentrations with depth in drill core 1 from the Mayday mine-waste pile near Silverton, Colorado.

Below 6 ft (2m), *Thiobacillus ferrooxidans* shows a strong relation of cell numbers to Pb and Zn concentrations whereas *Thiobacillus thiooxidans* shows lower cell numbers with respect to these elements (figure 5). Additionally, there appears to be an antagonistic effect between the two organisms in the mine dump above 6 ft. That is, when *Thiobacillus ferrooxidans* numbers are high, *Thiobacillus thiooxidans* numbers are low, and vice versa.

Use of Multidimensional Spatial Modeling

Multidimensional spatial modeling techniques can be used to integrate and synthesize disparate information about a mine site. Using these techniques, it is possible to simultaneously view two-dimensional and three-dimensional information. This approach can be a visually intuitive tool to examine spatial relationships and to view digital data. Yager and Stanton (2000) provided an example of spatial modeling by combining topographical, geophysical, and geochemical data for a mine-waste pile (Fig. 6).

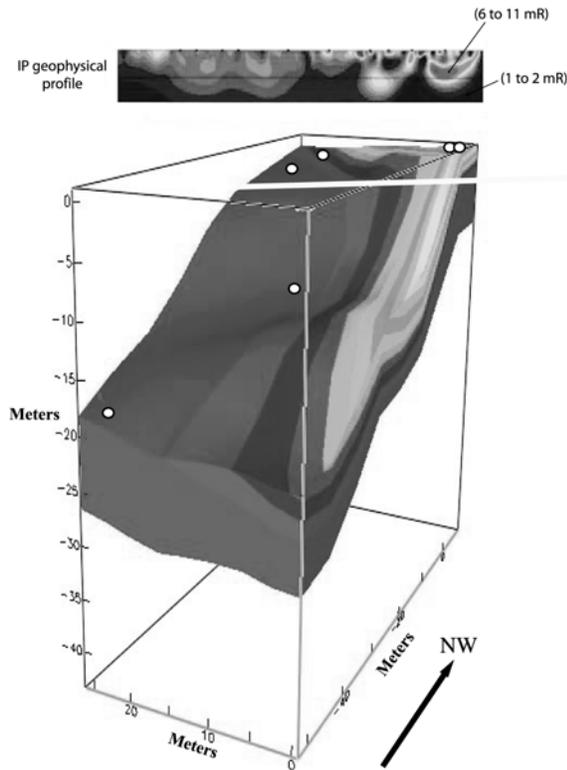


Figure 6. Synthesis of two-dimensional topographic data, three-dimensional geochemical gridded data for Zn (ppm), and geophysical induced polarization (IP) profile for the Mayday mine-waste pile near Silverton, Colorado. The white line across the face of the upper bench indicates the approximate IP profile location. Arrows point to relatively high IP signals (the arrow along the lower right edge of the block diagram indicates orientation of the waste pile). Note that high concentrations of zinc (lighter gray shades) in the upper right (northern) part of the pile coincide with a relatively high IP (6-11 milliradian) signal, although zinc alone is not responsible for the relatively high IP signal in the northern part of the pile. Modified from Yager and Stanton (2000).

ACKNOWLEDGMENTS

The U.S. Geological Survey Mineral Resources Program funded this work.

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