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Earth Mapping Resources Initiative Protocols—Sampling Hard-Rock Mine Waste and Perpetual Mine Water Sources

Scientific Investigations Report 2025–5068

U.S. Department of the Interior
U.S. Geological Survey

Cover. A Colorado Geological Survey staff member uses the Earth Mapping Resources Initiative mine waste sampling protocols to collect a composite sample at the Montezuma Mine, Colorado (Photograph by Kate Campbell, U.S. Geological Survey).

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By Kate M. Campbell, Robert R. Seal, Nadine M. Piatak, Jaime S. Azain, Jean M. Morrison, Sarah Jane White, Andrew H. Manning, Katherine Walton-Day, JoAnn M. Holloway, and Bronwen Wang

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Conversion Factors

International System of Units to U.S. customary units

Multiply	By	To obtain
	Length	
centimeter (cm)	0.3937	inch (in.)
millimeter (mm)	0.03937	inch (in.)
meter (m)	3.281	foot (ft)
meter (m)	1.094	yard (yd)
kilometer (km)	0.6214	mile (mi)
micrometer (µm)	0.00003937	inch (in.)
	Area	
square kilometer (km^2)	247.1	acre
square kilometer (km^2)	0.3861	square mile (mi^2)
	Volume	
liter (L)	0.2642	gallon (gal)
	Mass	
gram (g)	0.03527	ounce, avoirdupois (oz)
kilogram (kg)	2.205	pound avoirdupois (lb)

Temperature in degrees Celsius (°C) may be converted to degrees Fahrenheit (°F) as follows:

$$°F = (1.8 \times °C) + 32.$$

Supplemental Information

Specific conductance is given in microSiemens per centimeter (µS/cm).

Oxidation-reduction potential (ORP) is given in millivolts (mV).

Volumes are given primarily in milliliters (mL).

Dissolved oxygen is given in milligrams per liter (mg/L).

Abbreviations

~	about
-FA	filtered, acidified
-FALK	filtered, alkalinity
-FU	filtered, unacidified
-RA	raw, acidified
AP	acid generating potential
BPL	Big Pit Lake
DI	deionized
DO	dissolved oxygen
Earth MRI	Earth Mapping Resources Initiative
ETS	Example Tailings Site
GIS	geographic information system
GPS	global positioning system
ICP–MS	inductively coupled plasma mass spectrometry
ICP–OES	inductively coupled plasma optical emission spectroscopy
ID	identification
NP	neutralization potential
NNP	net neutralization potential
ORP	oxidation-reduction potential
PPE	personal protective equipment
QA/QC	quality assurance and quality control
SC	specific conductance
T	temperature
USDA	U.S. Department of Agriculture
USGS	U.S. Geological Survey

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Abstract

Supporting the overarching goal to evaluate critical minerals nationwide, the mine waste characterization effort in the U.S. Geological Survey (USGS) Earth Mapping Resources Initiative has created a series of protocols to standardize sampling carried out under this effort by the participating State geological surveys and their cooperators. The protocols are based on published, reviewed methods that can be deployed in the field. The protocols include (1) collecting and processing composite samples of mine and mill waste, including tailings, waste rock, gangue, heap leach piles, ore stockpiles, slag, or other mineralized and processed materials and (2) collecting and preserving water samples from perpetual or long-term mine water sources. The protocols also specify information to document on field sheets and detail the collection of geospatial data. The analytical methods used by the USGS and USGS contract laboratories are described in this report, including the data delivery pathway for USGS-derived data.

Introduction

The U.S. Geological Survey (USGS) Earth Mapping Resources Initiative (Earth MRI) is an effort to modernize mapping of the Earth's surface and subsurface across multiple types of data, including geologic, geophysical, geochemical, and topographical data (https://www.usgs.gov/earth-mapping-resources-initiative-earth-mri). Critical minerals are materials essential to the economic and national security of the United States with possible vulnerability to supply chain disruption (Nassar and others, 2020). Areas across the United States with the potential to host subsurface and surface deposits of critical mineral resources ("focus areas") were identified by the USGS and their State geological survey collaborators (Hammarstrom and others, 2023). Earth MRI focus areas are based on mineral systems, deposit types, and known and potential critical mineral commodities. The mineral systems classification links large mineral systems that may contain multiple deposit types to the mineral commodities associated with each deposit type (Hofstra and Kreiner, 2020). Earth MRI takes a mineral systems approach for prioritizing data collection efforts for aboveground and belowground critical mineral resources. One aspect of Earth MRI is to understand the distribution of critical minerals in aboveground mine waste.

The Earth MRI mine waste characterization effort collects compositional data to help identify mine waste sites that potentially contain critical mineral resources. The data can be used to estimate the critical mineral endowment of mine waste in the United States. Additionally, the data can be used to identify potential mineral hosts of critical minerals and provide a cursory evaluation of the environmental characteristics of the mine waste to help delineate potential reprocessing, environmental management, reclamation strategies, and other attendant costs. Identification of sites with critical mineral potential is the first step toward fostering economic development in conjunction with environmental remediation. Whether mine waste is a potential resource, an environmental liability, or both will depend on the geochemical and mineralogical characteristics of the waste. In addition, compositional characterization of mine waste may inform the use of new technologies to reprocess mine waste more efficiently for the extraction of critical minerals in the future. This information will be useful when assessing the total costs and benefits of reuse, recycling, reprocessing, reclamation, and restoration of ecosystem services. Evaluation of the future recoverability of critical minerals from mine waste is beyond the scope of this report and will depend on a variety of factors, including prevailing commodity prices, technological innovations, and financial incentives. The goal of Earth MRI mine waste characterization effort is to develop a comprehensive and internally consistent national database of mine waste locations, volume and mass estimates, bulk geochemical composition, bulk mineralogical composition, and contained mineral commodities. To this end, this report describes the methods followed by participating State geological surveys and their cooperators to standardize sampling carried out by Earth MRI's mine waste characterization effort.

Purpose and Scope

This report is intended to supplement inperson guidance that the USGS provides to participating State geological surveys. The scope of these protocols encompasses sampling of solid mine waste and large perpetual mine-related water sources (for example, pit lakes and draining adits). This report presents an approach to sampling a variety of solid waste types, including tailings, waste rock, gangue, heap leach piles, ore stockpiles, slag, or other mineralized and processed materials. If other types of materials are present at a site (for example, efflorescent salts, secondary precipitates, or streambed sediments) and the opportunity to sample is available during the course of a study, consult with USGS Earth MRI personnel on methodologies for sampling. Optional field sheets that provide guidance on general field notes and geospatial data are provided in appendix 1.

Many mine waste sites may be vertically heterogeneous. Vertical profile sampling, in addition to composited surface samples, may be considered. These protocols include an approach to sampling drill core; methodologies for drilling, trench sampling, or other vertical sampling techniques are not discussed in detail in this report because they are highly site dependent.

Safety planning, including job hazard analysis, selection of personal protective equipment (PPE), and appropriate safety training, is the responsibility of the State agency conducting the work. The USGS is not responsible for providing equipment, safety training, or evaluating site hazards and risks. Work with the responsible organization's safety officer to determine and complete the necessary safety requirements to perform the proposed work in a safe manner. Required PPE is provided as a guide for sample collection requirements. Additional PPE may be needed to conduct the work safely.

Protocols for Sampling Solids

Sampling of solid mine waste is complex because of the varied nature of source material, waste management approaches, meteorological effects, and site accessibility. Sampling described in these protocols are primarily focused on surficial material (0–10 centimeter [cm] depth; exceptions are described in the "Defining Sample Units, Locations of Subsamples, and Depths of Subsamples" section) using a statistically based, relatively rapid, composite sampling approach based on Smith and others (2000) and Naftz and Walton-Day (2016). Before sampling, divide the site into appropriate sample units as described in detail in the "Defining Sample Units, Locations of Subsamples, and Depths of Subsamples" section. Within each sample unit, the area is subdivided into a grid or randomized distribution of at least 30 subsample locations. For statistical rigor, a minimum of 30 subsample locations is required; more than 30 subsamples are acceptable. After collecting subsamples of equal volume and combining them into a single composite sample that has a minimum combined dry weight of 1 kilogram (kg), the sample is sent to USGS Sample Control, where it will be crushed and homogenized prior to submission for analysis.

Defining Sample Units, Locations of Subsamples, and Depths of Subsamples

The overall objective of designating sample units is to differentiate parts of a site that potentially could have different material composition or to divide a very large area into smaller units to obtain a series of composite samples that are more representative of a site. The number of sample units will depend upon the size, topography, mine waste type, ore-processing history, geography of the site, and other objective criteria, such as color. For example, if the tailings pile has discrete benches, each bench may be considered as a separate sample unit. Before collecting samples at the site, it is valuable to identify the site features and boundaries using recent satellite, aerial photography, or other imagery. The features may be subdivided into sample units if the feature is large and has topographical or other distinguishing attributes that indicate a change in composition or site management (for example, benches, discrete piles, or tailings color). If the site is large with no clear distinguishing features, select sample units of approximately 0.04–0.06 square kilometers (km^2). If a pile has mixed types of waste or has areas that contain vastly different sizes of material, sample units can be drawn to reflect those differences. For example, if a pile has mixed fine-grained material and cobble-sized pieces on the top of the pile, and boulder-sized rocks with no fine material around the base of the pile, the top of the pile may be considered a separate sample unit from the base. Different sampling methods can then be used to collect composite samples in each of the two sample units.

Site history is an important tool in understanding site features. For example, locations of pipes used to convey tailings, if available, can help guide distribution patterns on the landscape. Similarly, if the site went through multiple phases of ore processing, that information may be useful in interpreting different possible stratigraphy or topographical features at a site. Aerial imagery is helpful in understanding a site but is generally not sufficient to make final decisions about sampling approach. As such, site reconnaissance can be useful to more clearly identify the geography and topography of the features and to determine if a cover has been placed over the waste material.

Once sample units have been identified, select at least 30 subsampling locations per sample unit using a systematic grid or randomized location scheme that adequately covers the sample unit. Subsample locations may be planned before field work using geographic information system (GIS) tools, and any deviations from the plan can be recorded in field notes or with a global positioning system (GPS) device. For example, if a particular preselected subsample location is inaccessible, an alternative may be selected onsite as close to the original location as possible. Pin flags and flagging tape are helpful for marking out subsample locations onsite before sample collection. If subsample locations are not selected before the field work, pin flags or flagging tape can be laid out by the sampling team to visually confirm that the subsample locations adequately cover the sample unit before sampling begins, adjusting the locations and adding additional subsample locations as needed. Collect a minimum of 30 subsamples; more than 30 subsamples is acceptable. Log the subsample locations by GPS in the field if not predetermined. If a subsample location falls on a clearly unique or different feature (for example, localized material that differs in color or texture from the surrounding tailings) within the sample unit, collect and composite it as planned (also refer to the "Grab Samples" section).

If the site has an obvious soil horizon, clay cap, engineered cover (for example, a geomembrane), or other natural or engineered covering, the sampling procedure described in the "Protocols for Composite Sample Collection of Several Mine Waste Material Types" section may need to be modified to collect the waste material below the cover as long as the sampling does not compromise the integrity of an engineered cover. Site history will be helpful in determining if an engineered cover is expected at the site. Negotiating with the landowners for sampling permission, especially if a cover is expected, is the responsibility of the State agency conducting the work. Once onsite for sampling or during a reconnaissance visit, dig a test hole, if possible, (usually approximately 0.5 meters [m] deep but the depth may vary depending on site characteristics) to visually determine if samples should be collected below the surface. For example, tailings that are not acidic may have vegetative growth and a surficial layer with roots and some soil formation. This soil layer is usually apparent in the test hole, and the sample collection depth should be below this layer for all subsamples and recorded on the field sheets. Because each site has a unique setting, contact USGS Earth MRI personnel to discuss variations in sampling design as needed.

Grab Samples

Within the sample allotment and budget, additional grab samples of any unusual or notable feature may be collected. If the subsample location falls on a feature that is notable or different, collect and composite it as planned; additional grab samples of that feature may then be collected separately. For example, if a pile has a distinct orange weathered layer on the surface with continuous primary black unweathered tailings material below the weathered layer, collect the composite sample below the weathered surface layer; collecting a grab sample of the weathered orange surface layer at one or more subsampling locations is beneficial. If available, collect 1–2 kg of grab sample material to submit for analysis. However, if that mass is not available (for example, within a drill core or a smaller distinct layer in tailings), a smaller amount of material may be collected, and fewer chemical analyses may be completed on that sample at the discretion of the USGS.

Protocols for Composite Sample Collection of Several Mine Waste Material Types

These protocols are divided into four categories: (1) tailings and fine-grained material, (2) mixtures of small and large material, (3) predominantly large material, and (4) drill cores. Fine-grained material is generally less than approximately 2 millimeters (mm), and is typically found in certain types of waste, such as tailings piles. It is also possible to have mine waste that is a mixture of fine material (less than [<] 2 mm) and larger material (between 2 mm and 30 cm), all of which was derived from ore processing. Other types of waste, such as waste rock, may be mainly large cobble- to boulder-sized material (greater than [>] 30 cm) with very little fine-grained material present. Sampling drill cores is a special case for recovering material from vertical drilling.

Tailings and Fine-Grained Waste Material

In general, tailings are the waste material from ore that has been processed to recover a concentrated form of the target commodity. Processing usually includes some form of grinding or milling of the ore, and as a result, tailings are usually fine grained (<2 mm). For most tailings, material on the surface that is >2 mm may be the result of a cover or extraneous material deposition that should not be included in the composite sample. Sieving to <2 mm ensures that the sample collected is primarily the tailings material. Although unlikely in tailings piles, if most of the tailings have a grain size >2 mm, use the methodology described in the "Material Containing a Mix of Small and Large Particles" section or the "Large Material Predominantly Greater Than 30 Centimeters" section for larger material types. One composite sample will be collected for each sample unit, not including duplicate samples or additional grab samples.

Personal Protective Equipment

- Nitrile gloves—It is possible to cover work gloves with nitrile gloves, if desired, but work gloves alone cannot be used. Wear new nitrile gloves when sampling each sample unit to prevent cross-contamination.
- Additional PPE may be needed (for example, masks, eye protection, and clothing covering) depending on the site conditions and hazards.

Equipment

- Shovel.
- Trowel, small shovel, or incremental sampler (using stainless steel or plastic is necessary to prevent metal contamination).
- 5-gallon plastic bucket.
- Large (1 gallon) plastic bags or other sealable containers.
- Labels and permanent markers.
- 2 mm plastic or stainless-steel sieve.
- Flagging tape and (or) pin flags to mark subsample locations. Consider the choice of color so that the flags stand out in the sampling landscape.
- Wire brush to remove particles from the sieve as needed in between samples.
- Deionized (DI) water (or equivalent) and a squirt bottle for cleaning equipment. If DI water is not available, distilled water can be substituted. Do not use tap water because it can introduce elements that are part of the analysis package.
- Paper towels or laboratory wipes.

Rinse sampling tools, sieve, and buckets with DI water and air dry before sample collection.

Subsample Depth and Amount Collected

Collect subsamples from the upper layer of the tailings pile (0–10 cm deep), unless it is necessary to sample below a soil layer, cap, oxidized layer, hardpan, or crust. Dig a test hole before the first subsample collection to determine whether there is a soil, cap, or oxidized layer on the surface of the tailings pile. Although aerial imagery and site history are helpful for guiding sampling strategy, the best way of determining if there is a layer on top of the tailings is to dig to a depth of 0.25–1 m and observe any variation in color, particle size, plant roots (if present), moisture content, and other properties. If there is an obvious soil layer or cap on top of the tailings, then collect subsamples at the same depth below the anomalous surface layer. Record evidence of weathering (often indicated by a color change or hardpan layer) on the field sheet. The inperson guidance provided by the USGS can help identify these types of situations and inform the sampling strategy on a site-by-site basis.

Collect each subsample within a sample unit to the same depth and volume using the same equipment. Ensure the mass is at least approximately 50 grams (g) per subsample. The amount collected for each subsample can be approximated using the sampling equipment, a bag, or other container, as long as the same amount of material that is collected is consistent at each subsample location. For example, if using a trowel to collect the subsample, fill the trowel with approximately the same amount of material at each location. If using a plastic bag or container, mark the fill level with a permanent marker on the bag or container and fill to that level at each location. The method used may depend on the wetness, particle size, or other factors specific to the site material. It is not necessary to weigh each subsample. Ensure the total mass of the sieved composite sample after all the 30 or more subsamples have been combined is at least 1–2 kg dry weight.

Compositing Subsamples and Sieving

Sieve composited samples to <2 mm in the field or after being transported to a laboratory setting. If possible, sieve subsamples in the field during collection. As each subsample is collected, it can be sieved directly into a bucket; each subsample is sieved and sequentially added to the same bucket, which becomes the composite sample. It is usually possible to sieve tailings materials that are damp, unless the sample has a very high clay content. The >2 mm fraction of the material will not be submitted or analyzed and can be discarded.

If the sample cannot be sieved in the field (for example, very high clay content), the bulk material from each subsample can be directly added to the bucket to be sieved later. Remove any large particles (>1 cm) and large items (>1 cm) of manufactured material (for example, bolts, screws, or rebar) in the subsample, and record large items in field notes. If the material has a large amount of coarse (>2 mm) material, additional mass may need to be collected at every subsample location to accommodate the amount of >2 mm mass discarded during sieving. If the samples have a high clay content, sieving will be challenging in the field and in the laboratory because clay can be sticky when wet and harden into large aggregates when dry. Contact USGS Earth MRI personnel for more guidance on samples with high clay content; the USGS may be able to assist with sample preparation.

If tailings samples are wet when collected, air dry the samples before submitting them to USGS sample control. If sieving is occurring in a laboratory setting after collection, disaggregate any particle agglomerates that have formed when drying before sieving. Ensure the dry weight of the sieved composite sample is at least 1–2 kg. Once the composite sample has been sieved, the material can be transferred to a plastic bag, sealed, and labeled. Double- or triple-bagging samples before shipping will prevent loss of material during transit. If more than one bag of material has been collected, it is acceptable to submit multiple bags of the same material to the USGS for analysis to avoid having to homogenize and split the sample in the field.

Record detailed notes about soil and surface anomalies, subsample depth, volume, sieving, and other decisions for each sample unit on the field sheets, and label sample containers in the field. If the composite sample needs to be sieved in the laboratory, make a note on the field sheet and on the sample container (for example, "unsieved bulk composite sample") in addition to the label with the site name, date, and other information. Note any deviations from the described methods on the field sheet.

Material Containing a Mix of Small and Large Particles

It is not uncommon to have mine waste that is a mixture of fine material (<2 mm) and larger pieces (between 2 mm and 30 cm), such as in an ore stockpile that has had some initial bulk crushing but was never milled. In these piles, the <2 mm fraction and the >2 mm fraction are important to collect and analyze. Site history will help determine whether the >2 mm fraction is important to collect. Two composite samples will be collected at each sample unit, not including duplicates (described in the "Duplicates and Blanks for All Solid Sample Types" section) and additional grab samples. The approach consists of sieving the material to <2 mm, retaining the >2 mm fraction, and additional processing for larger pieces of material, as needed, to prevent a single large piece from biasing the composition of the >2 mm fraction. The two fractions will provide end-member compositions for the overall waste pile.

Personal Protective Equipment

- Nitrile gloves—It is possible to cover work gloves with nitrile gloves, if desired, but work gloves alone cannot be used. Wear new nitrile gloves when sampling each sample unit to prevent cross-contamination.
- Eye protection—Wear safety glasses if the material is large enough to require rock chipping.
- Additional PPE may be required (for example, masks, eye protection, and clothing covering) depending on the site conditions and hazards.

Equipment

- Shovel.
- Rock hammer, small sledge, and (or) pickaxe.
- Trowel, small shovel, or incremental sampler (stainless steel or plastic is necessary to prevent metal contamination).
- 5-gallon plastic buckets (2 per sample unit; one for the <2 mm fraction and one for the >2 mm fraction).
- Large (1 gallon) plastic bags or other spill-proof containers.
- Labels and permanent markers.
- 2 mm plastic or stainless-steel sieve.
- Flagging tape and (or) pin flags to mark subsample locations. Consider the choice of color so that the flags stand out in the sampling landscape.
- Wire brush to remove particles from the sieve as needed in between samples.
- Deionized (DI) water (or equivalent) and a squirt bottle for cleaning equipment. If DI water is not available, distilled water can be substituted. Do not use tap water.
- Paper towels or laboratory wipes.

Rinse sampling tools, sieve, and buckets with DI water and air dry before sample collection.

Subsample Depth and Amount Collected

If the material allows, a test hole will yield valuable information about material size and distribution. If some of the material is larger than 10–15 cm in diameter or length, it may be challenging to dig, and a surficial sampling approach will suffice. However, it is important to confirm, if possible, that the surficial material is not a cover on top of the mine waste.

Collect each subsample within a sample unit to the same depth and volume using the same equipment. The amount collected for each subsample can be approximated using the sampling equipment, a bag, or other container, as long as the same amount of sample is consistent at each subsample location. For this material type, a shovel or similarly sized container is usually a reasonable tool to estimate the sample amount, unless the material is mainly fine-grained, then a trowel can be used. It is not necessary to weigh each subsample. Collect enough material to yield about 50 g at each subsample location for both the <2 mm and >2 mm fractions. If the material is mainly fine grained, this protocol will be functionally similar to the tailings protocol, except that the >2 mm fraction is retained. If the material has a larger proportion of >2 mm material, ensure the subsample is large enough to collect about 50 g of <2 mm material at most locations. Ensure the total mass of each composite sample (<2 mm and >2 mm) after all 30 or more subsamples have been combined is at least 1–2 kg each, dry weight.

Compositing Subsamples, Sieving, and Processing Greater Than 2 Millimeter Fraction

Collect the <2 mm and >2 mm fractions of the sample in two separate buckets. If possible, sieve each subsample to <2 mm directly into a bucket in the field. Retain the material that does not pass through the 2 mm sieve and process as follows:

- If most of the >2 mm fraction retained on the sieve is less than approximately 4 cm in diameter and is about 50 g worth of material, it can be placed directly into the second bucket. If this fraction is substantially more than approximately 50 g of material, select an about (~) 50 g randomized subset of material, place it into the second bucket, and discard the remainder. It is important to not bias the selection toward items of a particular size or color; ensure the subset of material is representative of the bulk >2 mm material.

- If there are pieces larger than approximately 4 cm, remove them from the shovel directly and place them aside. Large pieces of material can bias the overall composition of a composite sample and need to be reduced in size to be included in the sample. Choose a random selection of large pieces and ensure care is taken to not bias the selection toward items of a particular size or color. Break these large pieces with a sledge or rock hammer and collect a single chip (less than or equal to ~4 cm in length or diameter) in the second bucket.
- If there is a mix of <4 cm and >4 cm pieces in the >2 mm fraction, then use a combination of the two processes. For example, place a random selection of <4 cm pieces in the second bucket, along with ~4 cm chipped samples from the larger pieces.

Subsamples may show different distributions of large and fine material within a single sample unit. Continue to sample at all locations using the same approach, even if it results in <50 g material for a size fraction at a single subsample location. For example, most subsample locations may have a roughly equal distribution of <2 mm and >2 mm fractions, but one subsample location only has 20 cm rocks and no fine-grained material. At this location, collect the rock chips and no fine-grained material. The composite approach normalizes across the individual subsample location variations to collect an average composition across the entire sample unit.

This procedure results in two buckets: one bucket with <2 mm sieved material and one bucket with >2 mm material, including rock chips. The material in each bucket is considered a separate sample. Ensure each composite sample (<2 mm and >2 mm) is at least 1–2 kg, dry weight. Transfer the composite sample from each bucket to different plastic bags that are sealed and labeled with unique identification. Double- or triple-bagging before shipping will prevent loss of material during transit.

Large Material Predominantly Greater Than 30 Centimeters

Some waste material, such as waste rock, may have very little fine material and only large pieces that can range from several kilograms to many hundreds of kilograms. For waste material with large pieces, a <2 mm fraction will not be collected. The following sections present a method to subsample large pieces to collect a representative composite sample.

Personal Protective Equipment

- Nitrile gloves—It is possible to cover work gloves with nitrile gloves, if desired, but work gloves alone cannot be used. Wear new nitrile gloves when sampling each sample unit to prevent cross-contamination.
- Eye protection—Wear safety glasses if the material is large enough to require rock chipping.
- Additional PPE may be required (for example, masks, eye protection, and clothing covering) depending on the site conditions and hazards.

Equipment

- Rock hammer, small sledge, and (or) pickaxe.
- 5-gallon plastic bucket.
- Large (1 gallon) plastic bags or other spill-proof containers.
- Labels and permanent markers.
- Flagging tape and (or) pin flags to mark subsample locations. Consider the choice of color so that the flags stand out in the sampling landscape.
- Deionized (DI) water (or equivalent) and a squirt bottle for cleaning equipment. If DI water is not available, distilled water can be substituted. Do not use tap water.
- Paper towels or laboratory wipes.

Rinse sampling tools and buckets with DI water and air dry before sample collection.

Subsampling Approach

Subsample points likely will fall on large rocks or boulders. To subsample a large rock, use a rock hammer, sledge, or other tool to chip off 3–5 randomly selected pieces of material approximately 4 cm in diameter from the surface of each rock. Place these pieces directly into the bucket, accumulating rock chips across all the subsample locations. The composite sample will be the mixed rock chips in the bucket from at least 30 subsample locations and at least 1–2 kg dry weight. The composite sample can be transferred to a plastic bag, sealed, and labeled. Double- or triple-bag the sample before shipping to prevent loss of material during transit.

Drill Cores

Drilling is not required for site characterization, but if equipment and budget allow, it is encouraged because samples can be collected at depth in the pile. The details of the best equipment, techniques, and PPE for drilling at the site will be at the discretion of the State agency conducting the work. Tailings and other fine-grained mine waste are good candidate materials for drilling, resulting in a poorly consolidated core with some larger clasts possibly present. The methods described in the “Collecting Composite Samples” section assume the core is relatively unconsolidated and does not need a rock saw to split or collect samples. If the core does not meet this description, a discussion with USGS Earth MRI personnel may be warranted.

Equipment

- Trowel and (or) scoop (stainless steel or plastic prevent metal contamination).
- Rock hammer or other tool to split clasts as needed.
- Plastic buckets or other containers for the composited samples.
- Large (1 gallon) plastic bags or other spill-proof containers.
- Labels and permanent markers.
- Tape measure for measuring sample unit intervals.
- Deionized (DI) water (or equivalent) and a squirt bottle for cleaning equipment. If DI water is not available, distilled water can be substituted. Do not use tap water.
- Paper towels or laboratory wipes.

Rinse sampling tools and buckets with DI water and air dry before sample collection.

Determining Sample Intervals (Sample Units) Within a Core

If available, review core logs to identify where stratigraphic breaks occur in the core and how frequently they occur. Laying the core out to visually identify the stratigraphic breaks is also suggested. If the material is homogeneous, then dividing the core into equal sections along the entire core depth is an option, depending on how many samples per core are reasonable for the budget. It is more likely, however, that there are apparent heterogeneities in the core. These heterogeneities or stratigraphic breaks can define sample units within the core, such that a sample unit includes depths between the stratigraphic breaks. Samples will be composited across a sample unit defined within the core (described in the “Collecting Composite Samples” section), so that composition is not dominated by only collecting individual or interesting layers within the core. Distinct or unusual layers may be used to separate sample units; include these layers in the composite sample. Depending on the stratigraphy of the core, sample units may be of variable lengths. In sample sheets, note the depths and location in the core where sample units begin and end after laying out the entire core. Each sample unit will result in one composite sample.

For highly variable or closely spaced breaks, some judgment will need to be applied to balance the need to define sample units for compositing and capturing every heterogeneity presented in the cores. In addition to composite samples, grab samples are encouraged, particularly if there are distinct layers that are different from the surrounding material. The grab samples can be collected after the core is split and the composite sample is collected. This method ensures that the composite sample includes the distinct layers, while the composition of the layers may also be captured individually in grab samples. The balance between the number of composite samples and grab samples will depend upon the overall scope of the project, including the number of drill cores, other samples collected (for example, surface composite samples), and sample budget.

Collecting Composite Samples

Depending upon the diameter of the core, splitting the core vertically in half or quarters will likely be necessary. If there is a rock or clast in the core, split it with a rock hammer or similar tool. Collect the composite sample by taking the split material in a sample unit (within the determined stratigraphic interval) and combining it in a bucket. Ensure the composite sample is approximately 1–2 kg, dry weight. Transfer the sample material to a plastic bag, sealed and labeled. Double- or triple-bag samples before shipping to prevent loss of material during transit. Grab samples may be collected from the uncomposited split section of the core.

Duplicates and Blanks for All Solid Sample Types

Field duplicate samples give an important indicator of overall field sampling variability at a site. Collect a duplicate sample at a frequency of 1 per 10 samples. If fewer than 10 samples are collected at a site, then select one sample unit at the site for a duplicate sample collection. Ensure a duplicate sample goes through the same subsample collection, composite, and sieving process and is labeled as a separate sample with a unique identification (ID). Subsamples for the duplicate sample can be collected nearby (within ~1 m radius) and can use the same GPS location of the original subsample location, but do not collect these from the same hole as the original subsample.

For drill cores, collect a duplicate sample from a sample unit within a core at a frequency of 1 in 10 samples. Collect duplicates by compositing a separate split of the core. If collecting grab samples from the core in that section is also planned, split the core in that sample unit vertically into thirds or quarters so material is not excluded from the composite sample or duplicate sample.

A process blank is optional but can be a valuable check on equipment cleanliness. A blank consists of a known material, typically clean quartz from a commercial supplier, that is put through the sampling process using all the equipment. For example, if a trowel is used to collect the tailings subsamples, the clean trowel will be used to scoop the blank material through the sieve into a bucket and bagged as if it were a tailings sample. If you are interested in including a blank, contact USGS Earth MRI personnel, and they will supply the quartz blank material and discuss safety considerations when working with quartz.

Equipment Cleaning in Between Sample Units

Clean the trowel, shovel, sampler, sieves, and buckets before the next sample collection. Wipe them with a clean rag, paper towel, or laboratory wipe, then remove any particles stuck within the sieve mesh before washing. The wire brush may be necessary to remove particles from the sieve mesh. Lastly, rinse equipment with DI water, and completely air dry before the next sample collection. The wire brush may be used to clean the sieve in between subsample locations if the mesh is substantially occluded (for example, with clay); clean in between sample units but not necessarily in between subsamples.

Sample Labeling, Handling, and Storage for All Sample Types

Secure sieved samples in a plastic bag or other spill-proof container and label with permanent ink. Double- or triple-bag to prevent loss of sample during transport. Ensure the sample label has a unique site and sample unit ID, date of collection, and size fraction (for example, <2 mm) once sieved. Consider including a project ID that indicates the program (Earth MRI–Mine Waste Characterization) and State. Label duplicate samples with a unique ID. Tailor unique IDs for the particular site and sample type using a consistent format. For example, if the sample is from a site called "Example Tailings Site" in State "YY" and is a composite sample from sample unit 1 collected on November 1, 2022, at 15:00, the unique sample ID could be "22_ETS_SU1_composite"; details about the exact date and time can be recorded on the field sheet and on the label. A duplicate sample could be labeled "22_ETS_SU1_composite_dup" to differentiate it from the first sample. If the mine waste material is a mix of fine-grained and larger materials, resulting in two composite samples from a single sample unit, additional information needs to be added to the label to identify the sample size fraction (for example, add "<2 mm" or ">2 mm" on the sample label; fig. 1). Holding temperature (T) of samples does not have to be controlled. Air dry, inventory, and submit samples to USGS Sample Control (refer to the "Sample Submission" section). Multiple bags can be submitted for a single sample; ensure bags are labeled accordingly (for example, "1 of 2" and "2 of 2").

Extra Equipment for Sampling Solids

Even with the best planning possible, it is almost always necessary to have extra supplies. Extra supplies can include extra DI water, buckets, shovels, flags, and other supplies to the extent possible. If multiple sample teams will be sampling the site, extra sets of all necessary equipment will be needed.

22_ETS_SU1_composite
1 Nov 2022 15:00
<2 mm
EMRI-MWC-YY

Figure 1. Graphic showing an example sample label for solid composite samples. Sample labels include the site and sample unit identification, date and time of collection, sample size, and project identification. Nov, November; <, less than; mm, millimeters.

Protocols for Water Sample Collection, Preservation, Measurement of Field Parameters and Flow

Methods for water sample collection and preservation are derived from the USGS National Field Manual for the Collection of Water-Quality Data (Wilde, 2011), specifically chapters A1 (USGS, 2018), A2 (Wilde and others, 2014), A3 (Wilde, 2004), A4 (USGS, 2006), A5 (USGS, 2002), and A6 (Nordstrom and Wilde, 2005; USGS, 2019, 2020, 2021, 2023, 2024). An overview of the sample collection and preservation methods is presented in figure 2. Depending on the type of site sampled (for example, pit lake or adit drainage), additional equipment may be needed to collect the water sample (for example, boat, sampling pole, or other equipment). Generally, water will be extracted by a peristaltic pump and through a filtration apparatus or directly into a bottle, although alternate methods such as syringe filtration are acceptable. When water samples are collected, also measure field parameters such as pH, T, specific conductance (SC), and optionally dissolved oxygen (DO) and oxidation-reduction potential (ORP).

Equipment and Preparation

Personal Protective Equipment

- Wear clean nitrile or similar laboratory-grade gloves when handling equipment that comes in contact with sample water and during sample and field parameter collection.
- Sunscreen—Some sunscreens contain zinc and titanium oxides; avoid using these sunscreens if possible during water sampling. If using these sunscreens is not avoidable, be careful about minimizing any possible direct contact with equipment without clean gloves.
- Safety glasses.
- Other PPE may be necessary depending on sample collection method (for example, if collecting by boat) and site conditions (for example, flotation devices, eye protection, or clothing covering).

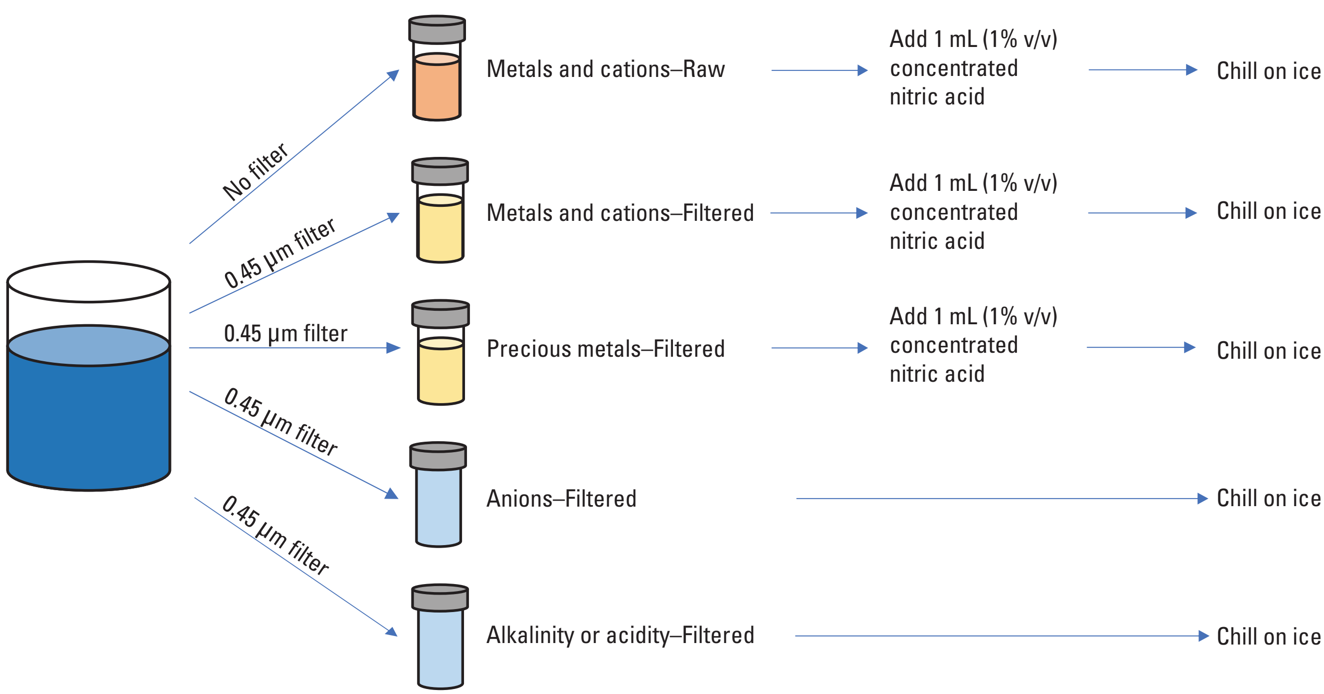

Figure 2. Conceptual flow chart of water sample collection and preservation. µm, micrometers; mL, milliliters; %, percent; v/v, volume per volume.

Bottle Sets

Each sample collected will be distributed (“split”) into a set of the following 5 bottles:

- Metals and cations—Two 125 milliliter (mL) acid-washed plastic bottles.
- Precious metals—One 125 mL acid-washed plastic bottle.
- Anions—One 125 mL deionized water-rinsed plastic bottle (not acid washed).
- Alkalinity or acidity—One 125 mL deionized water-rinsed plastic bottle (not acid washed).

Label bottles with waterproof labels or laboratory tape. If labeling bottles with tape, wrap the tape around the entire circumference of the bottle and secure by overlapping to prevent detachment. Waterproof labels can be printed or labeled with permanent ink. Uniquely label each bottle with the site name, date and time of collection, the analysis, and associated preservation. It is helpful if the label also indicates the program (Earth MRI—Mine Waste Characterization) and State. Add abbreviations for preservation to the sample name according to table 1; for example, label unfiltered metals and cation bottles with the suffix “-RA” (raw, acidified), filtered metals and cations bottles and precious metals bottles with the suffix “-FA” (filtered, acidified), filtered anions bottles with the suffix “-FU” (filtered, unacidified), and filtered alkalinity bottles with the suffix “-FALK” (filtered, alkalinity).

In the first example label in figure 3*A*, the “_001” is added if multiple samples are collected from the same site on different days (time series); the second sample could be indexed to “_002.” In the second example label in figure 3*B*, the time and date are incorporated into the unique ID; however, take care to prevent typographical errors in long strings of numbers, such as a date, when writing the labels. The abbreviation for a specific location (in this hypothetical example, Big Pit Lake [BPL]) may depend on where and how many samples are collected. If sampling multiple locations at a single large feature, “BPL01” may be used to delineate specific locations. In all examples, ensure details about the site, sample location, date, time, and so forth, are clearly described in the field sheet notes.

Acid Washing Bottles for Cation and Metal Sample Splits

Acid wash all metals, cations, and precious metals bottles before use. Some laboratory supply companies have the option to purchase precleaned bottles, but precleaned bottles are not required as long as the bottles can be sufficiently cleaned. Also acid wash a water collection container before using. If acid washing bottles, ensure the acid bath is a 10–20 percent reagent grade nitric or hydrochloric acid solution, and the bottles and caps are soaked for at least 12 hours. Rinse the bottles well and at least three times with DI water (for example, 18 megohm-centimeter or similar). Air dry the bottles in a clean environment (for example, in a laminar flow hood). Make a fresh acid bath every 6 months.

Deionized Water Rinse for Anion and Alkalinity or Acidity Sample Bottles

Soak anion and alkalinity bottles and caps overnight in DI water before use. Some laboratory supply companies have the option to purchase precleaned bottles, but precleaned bottles are not required as long as the bottles can be sufficiently cleaned. Do not acid wash bottles for anion and alkalinity sample splits. Acid washing anion and alkalinity bottles may result in erroneous chloride and alkalinity measurements.

Table 1. Overview of bottles, preparation, and preservation for water samples.

[Filtration pore size is 0.45 micrometers (µm). % percent; v/v, volume per volume; “-RA,” raw, acidified; “-FA,” filtered, acidified; °C, degrees Celsius; “-FU,” filtered, unacidified; “-FALK,” filtered, alkalinity]

Analysis	Preparation	Preservation	Label suffix
	No filtration		
Metals and cations, raw	Acid wash	1% v/v concentrated nitric acid	“-RA”
	0.45 µm filter		
Metals and cations, filtered	Acid wash	1% v/v concentrated nitric acid	“-FA”
Precious metals	Acid wash	1% v/v concentrated nitric acid	“-FA”
Anions	Deionized water rinse	Chill at 4 °C	“-FU”
Alkalinity or acidity	Deionized water rinse	Chill at 4 °C	“-FALK”

A

22_EWS_BPL_001-FA
1 Nov 2022 15:00
EMRI-MWC-YY
Metals and Cations

B

EWS_BPL01_20220111_1500-FA
1 Nov 2022 15:00
EMRI-MWC-YY
Metals and Cations

Figure 3. Example bottle labels for a hypothetical metals and cations sample collected at the “Example Water Site” from the “Big Pit Lake” in State “YY” on November 1, 2022, at 15:00 hours. *A,* An example of a label in which multiple samples could be collected from the same site on different days. *B,* A second example of a label that has the date and time included in the sample name. Nov, November.

Cleaning Pumps and Tubing

Peristaltic pumps are generally the best option for collecting water samples, as long as the height of the pump above the waterbody does not exceed the hydraulic head rating of the pump. If the water needs to be pumped to a height that exceeds the pump rating, then an alternate pump will need to be considered. Acquire tubing that is compatible with the pump and pump head used, rinse the tubing with DI water, and air dry completely before sampling. If feasible, have tubing dedicated for each site to prevent carryover across sites. However, if the tubing needs to be reused, rinse the tubing with DI water in the field, if possible, or thoroughly rinse with sample water before collecting the sample. Do not rinse with sample water if sampling water with low concentrations immediately following water with high concentrations, or if the relative concentrations cannot be determined in the field based on geochemical context.

Filters

Preferred filters are clean, unused 0.45 micrometers (µm) pore size, high-capacity capsule filters, although other 0.45 µm filtration systems are acceptable. Examples of other filters include plate filters, syringe and syringe filters. Prepare the filters according to the manufacturer’s instructions, if applicable. Record the filtration type and membrane material on the field sheet.

Water Collection Containers

Depending on the site, extra containers to collect raw samples for filtration or splitting into bottles may be required. Containers may be needed if water cannot be directly pumped from the source. The container(s) may be a carboy, a large bottle, or other clean (acid-washed) container that can hold enough sample to rinse equipment, bottles (as needed), and collect sample splits. The volume needed for sample collection will depend upon the type of equipment being used; for example, if a capsule filter is being used, use a minimum of a 2.5–3-liter (L) water collection container so that the filter can be rinsed with at least 1 L of sample before sample splits are collected per the manufacturer’s instructions. Rinse the water collection container(s) three times with the sample before filling. Rinse and fill a separate container for field parameters at the same time, if in situ (electrodes placed directly into the source water) measurements are not possible.

Additionally, access to water may require a collection mechanism such as a dipper sampler (for example, a “bottle on a stick”) which allows for sampling water from a distance as much as 2–3 m. This device is useful at adits, ponds, and pit lakes where there may not be direct, easy access (for example, a water tap). There are commercially available dipper samplers, but homemade samplers are also acceptable. Clean (acid wash) and change the dipper container between sites. Reconnaissance and (or) discussion with personnel familiar with the site may be helpful to determine if a dipper or other sampling devices are necessary.

Preservation Reagents and Distribution Equipment

Water sample splits have specific preservation requirements, illustrated in figure 2 and described in detail in this section.

Preservation for samples with the suffix “-FU” and “-FALK”.—Store the samples in a cooler with ice in the field and during shipping to USGS Sample Control. The samples may be stored on ice or refrigerated in the laboratory after collection and before shipping.

Field preservation for samples with the suffix “-FA” or “-RA”.—Samples with the “-FA” or “-RA” suffix need acidification in the field to preserve the sample for analysis. Use concentrated (16 molar) high-purity nitric acid (HNO_3) for preservation. The acid can be added to the sample after collection, or prewashed bottles with nitric acid preservative added by the bottle manufacturer can be used. If adding the nitric acid after the sample is collected, use a distribution system that includes either (1) a pipette, clean pipette tips, and a bottle for acid (Teflon preferred), or (2) a dripper bottle calibrated in the laboratory for how many drops approximately equals 1 mL of acid.

Water for Blanks

Bring DI water into the field for a field blank. The volume of water will depend on the length of tubing and the filtration system used; ensure the volume is sufficient to replicate the entire water collection process with DI water, including rinses and filling a complete bottle set.

Extra Equipment for Water Sampling

Even with the best planning possible, it is almost always necessary to have extra supplies. Consider packing at least 10 percent extra bottle sets, filters, preservation materials (if not using prepreserved bottles), DI water, water collection containers, and other supplies. If the water has a lot of particulates, additional filters may be needed. For example, if using 33-mm diameter syringe filters, having 5–10 filters per sample may be needed. One high-capacity capsule filter is usually sufficient per site, even with high particulate loads.

Equipment Reuse

Some equipment used for sampling cannot be reused. Bottles used in bottle sets are single use and will not be returned. Similarly, all filters are single use and not for use at multiple sites, across duplicate samples, or on multiple dates. If using a pipette to distribute acid into bottles requiring preservation, a single tip can be used to distribute acid into multiple bottles at a single site as long as the tip does not touch the sample water or a dirty surface. Avoid reusing pipette tips across sites.

Some equipment may be reused if cleaned between sites. Items include pump tubing and collection containers. If possible, tubing and collection containers dedicated to a site are preferable, but thorough DI water and sample rinses between sites may be used if necessary. Bring extra DI water into the field if rinsing equipment between sites.

Field Parameters

If possible, it is best to measure field parameters in situ. If in situ measurements are not feasible, a flow-through cell, bottle, or flask may be used. If measuring in situ, place the probes downstream from where the water sample is being collected or collect water samples immediately before or after measuring field parameters to prevent contamination. A meter or meters will be needed to measure the output from each electrode and perform the calibrations. If carefully selected, the same meter may be used for field parameters such as pH, T, SC, ORP, and DO (if a probe is used). Alternatively, a sonde with the ability to read the output and perform calibrations may be used. Consider testing electrodes and meters in the office or laboratory before conducting field work to ensure performance and familiarity with the equipment. It is not uncommon for mine waters to degrade electrode performance; therefore, consider bringing spare electrodes to the field. Consider measuring ORP and DO (optional parameters) if the equipment is available. The following are specific equipment and supplies needed to conduct the various measurements:

- pH
  - pH electrode with appropriate outer filling solution, if required. A triode (with T) or the ability to measure T simultaneously with pH on the same meter (for example, using a thermistor or specific conductance probe) allows for appropriate T compensation. An electrode with guards around the glass membrane is encouraged but not required.
  - Meter to display results.
  - Calibration buffers (a set of at least three, bracketing the pH of the water to be measured. Buffers can be purchased with pH values of 1.68, 4.01, 7.00, and 10.01).
  - A quality assurance and quality control (QA/QC) check sample (a separate aliquot of a buffer or other solution of known pH).
  - Flask or flow-through cell.
  - DI water.
  - Laboratory tissues.
- Temperature
  - Thermistor or liquid-in-glass thermometer (nonmercury); specific conductance and pH probes may have a thermistor included and may be used to measure T.
  - Meter to display results.
  - Flask or flow-through cell.
  - DI water.
  - Laboratory tissues.
- Specific Conductance
  - Conductivity probe (typically a 1-cm cell width).
  - Meter to display results.
  - Calibration standards (typically 500 microSiemens per centimeter [µS/cm], 1,413 µS/cm, or 12,900 µS/cm, whichever is closest to, and higher than, the water being measured; 1,413 µS/cm is a typical calibration standard).
  - A QA/QC check sample (a solution of known SC, similar to the expected concentration of water to be measured; may be a separate aliquot of a calibration standard).

    - Flask or flow-through cell.
    - DI water.
    - Laboratory tissues.
- Optional—ORP
    - Platinum electrode with associated filling solution.
    - Meter to display results.
    - ORP check standard (for example, Zobell’s solution or the manufacturer’s standard).
    - Flask or flow-through cell.
    - DI water.
    - Laboratory tissues.
- Optional—DO
    - DO kit that uses colorimetric reagents (for example, Rhodazine D or Indigo Carmine). Alternatively, a DO meter and probe may be used.
    - Waste disposal container.
    - DI water.
    - Laboratory tissues.

Water Collection Protocol

The following sections describe methodology for collecting, filtering, preserving, and storing water samples.

Rinsing with Sample

Rinse the tubing with sample by pumping water through the tubing without collecting a sample; the amount of water may depend on the length of the tubing used but should be approximately 1 L. Similarly, rinse the filter accord to the manufacturer’s instructions (for example, at least 1 L for capsule filters). Read the manufacturer’s guidance for specific filter requirements. The tubing and filter can be rinsed simultaneously. Rinse the bottles three times with sample water before collecting a sample, unless the bottles are prefilled with preservative. If a bottle is prefilled with preservative, fill it with the sample directly without rinsing.

Filtration

For the raw metals and cations sample, do not filter when collecting the sample. Ensure all other samples go through a 0.45 µm filter before collection. Fill the bottles to the shoulder of the bottle (~100 mL), except alkalinity samples—fill those bottles to the top and leave minimal headspace in the bottle.

Preservation of Water Samples

For raw and filtered metals and precious metals samples, acidify the sample in the field to 1 percent volume per volume nitric acid. For example, if a 125 mL bottle is filled to the shoulder and contains approximately 100 mL of sample, add 1 mL of concentrated nitric acid to the bottle. Wear safety goggles and gloves while acidifying, and shake the bottle after capping to mix. For anions and alkalinity samples, store filled bottles in a cooler on ice in the field with no additional preservation. Keep ice and samples in separate sealable plastic bags to prevent melted ice from contaminating the samples.

Quality Assurance and Quality Control—Duplicates and Blanks

For each sampling event (date), collect at least one DI water field blank to test the cleanliness of the sampling process. Follow the entire sample collection protocol with DI water, including rinses and a complete set of sample bottles. Collect a duplicate sample set at least once per sampling trip or 1 per every 20 sample sites, if more than 20 sample sites are collected in a single sampling event.

Sample Handling, Storage, and Shipping

Clearly label each bottle as described in the “Equipment and Preparation” section. Group samples of a single analytical type (for example, alkalinity or precious metals) into a sealable bag; bags of samples with mixed analytical types are not acceptable. Multiple samples may be stored in a single plastic bag as long as they are of a single analytical type, but keep the bag sealed and consider double bagging the samples. Store samples with caps tightly closed to prevent leakage and in sealed plastic bags to prevent melted ice or dirt from contaminating the samples.

Keep anion and alkalinity samples chilled in the field, during shipment, and when stored in the laboratory. Metals and precious metals samples may be chilled, but it is not required. Do not freeze samples at any time. Inventory samples before shipping them and submit them to USGS Sample Control (refer to the “Sample Submission and Geochemical Analysis” section).

Flow and Discharge Measurements

If sampling water that is flowing (for example, from an adit), measure or estimate the flow or discharge. If the site is managed, the site manager may be able to provide flow data (for example, a gage, valve, or permanent weir). If so, that flow data may be used. If no flow data are available, various measurement techniques may be used depending on the volume of flow and site characteristics (Science Applications International Corporation, 2001). For example, a weir (permanent or temporary) or flume may be used for lower flow systems, whereas direct flow velocity measurements (for example, with an anemometer, propeller, or Doppler flow meter) may be needed for larger flows. Tracer techniques with salts or dyes can also be used. Volumetric flow measurements are also acceptable. The choice of method will depend on expected flow, site conditions, channel geometry, availability of equipment, and access. If no direct flow data can be obtained, make a visual estimate. Regardless of the technique used, record details of the methodology used on the field sheet.

Load Calculations

Elemental load is a useful expression of how much of a particular element is flowing past a point in space as a function of time; load values incorporate concentration and discharge information for a flowing water source. Instantaneous load is calculated for a location by multiplying discharge by constituent concentration. Units are typically expressed in grams (or kilograms) per day for each element reported. Calculate the loads and include them in the final report.

Field Parameters

The following sections describe methods for measuring pH, SC, T, ORP, and DO in water samples.

pH

Measure pH in the field, either in situ or in a flask or beaker filled with a sample as close to ambient conditions as possible. Because pH is T dependent, it is important to measure T simultaneously with pH and to calibrate at ambient T for automatic T compensation. The following steps are for calibrating and measuring pH in samples:

1. Attach the electrode to the meter (if not already in a sonde) and prepare the electrode according to the manufacturer's instructions.

2. Rinse the pH electrode with DI water and blot dry with a laboratory tissue when moving the electrode between solutions, including buffers and samples, to prevent cross-contamination.

3. Calibrate on 2 or 3 buffers that bracket the pH of the water being sampled. Ensure the calibration buffers are as close to the same T as the sample as possible. Record calibration slope, T, and buffers on the field sheet. Ensure the slope is between 92 percent and 102 percent. If it is not, repeat the calibration or use a different electrode. Record the lot numbers and expiration dates of the buffers on the field sheet.

4. Check the pH of a QA/QC check sample (for example, a different aliquot of a buffer or a solution with known pH). Ensure it is within 0.05 units of its accepted value. Record this result on the field sheet. If the QA/QC check fails, then repeat the calibration.

5. Measure the pH of the sample and record the value on the field sheet.

6. As many as 10 samples may be measured before remeasuring the QA/QC check sample, fewer than 2 hours have passed, and the pH meter and electrode have not been moved to a new location. If any of those conditions are not met, then repeat steps 4 and 5. It is acceptable to measure the QA/QC check sample after every sample if desired.

7. At the end of the sampling day, analyze a final QA/QC check sample according to step 4 and record it on the field sheet.

8. Store the electrode according to the manufacturer's instructions.

If the pH of the water falls outside of the buffers used for calibration, note this condition on the field sheets and in the final data report.

It is preferable to measure pH in the field. However, if it is not possible, an additional sample split may be collected in a clean bottle by filling the sample to the top of the bottle and chilling the bottle during transport and storage at the laboratory. Ensure measurements are made within 48 hours and at room temperature to minimize changes in water chemistry. Note if there are visible precipitates or color changes in the sample split. Also note this deviation from field measurement on the field sheet.

Specific Conductance

Because SC is highly T dependent, it is important to measure T simultaneously with SC for automatic T compensation. The following steps are for calibrating and measuring SC in water samples:

1. Connect the SC probe to the meter according to the manufacturer's instructions.

2. Rinse the SC probe with DI water and blot dry with laboratory tissue in between each solution, including calibration standards, QA/QC check samples, and samples.

3. Many meters and sondes use one-point calibration for SC, although some meters allow for multipoint calibrations. Record the calibration standard, T, and cell constant (one-point calibration) or slope and intercept (multipoint calibration) on the field sheet. Calibrations may be performed in the laboratory before field measurements; if performed in the laboratory, then

also record the date of the calibration on the field sheet. Also record the lot number and expiration date of the calibration standard and QA/QC check sample on the field sheet.

4. Analyze a QA/QC check sample. Ensure the value is within 10 percent of the expected value. If it is not, recalibrate according to step 3.
5. Measure the SC of the sample by allowing the reading to stabilize and record on the field sheet. Be particularly careful to rinse and avoid contamination if the sample has a low SC value.
6. As many as 10 samples may be measured before remeasuring the QA/QC check sample, fewer than 2 hours have passed, and the pH meter and electrode have not been moved to a new location. If any of those conditions are not met, then repeat steps 4 and 5. It is acceptable to measure the QA/QC check sample after every sample if desired.
7. At the end of the sampling day, analyze a final QA check sample according to step 4 and record it on the last field sheet.
8. Store the electrode according to the manufacturer's instructions.

It is preferable to measure SC in the field. However, if it is not possible, an additional sample split may be collected in a clean bottle by filling the sample to the top of the bottle and chilling the bottle during transport and storage at the laboratory. Ensure measurements are made within 48 hours and at room temperature to minimize changes in water chemistry. Note if there are visible precipitates or color changes in the sample split. Also note this deviation from the field measurement on the field sheet.

Temperature

Temperature can be measured using a separate thermistor or thermometer, or more commonly, using the thermistor incorporated into the SC probe or a pH triode. If measured with an SC probe or pH triode, record which probe or electrode's T value is being used on the field sheet and use that same source consistently through the sampling program. The following steps are for calibrating and measuring T in samples:

1. Ideally, verify the thermistor's T readout in the laboratory with a certified thermometer or other method of verification (for example, the ice-water method). Thermistors are generally stable and may only need to be checked as recommended by the manufacturer.
2. If using a separate thermistor or thermometer, rinse the probe with DI water and blot dry with laboratory tissue in between each solution measured.
3. Measure the T of the sample when stable and record on the field sheet. If using a flask, observe whether the T changes during the pH and SC measurements, and consider refreshing the sample in the flask or setting the flask in the outflow to prevent warming or cooling. Alternatively, use a flow-through cell or measure field parameters in situ to get more accurate measurements.

Oxidation-Reduction Potential

An optional measurement is ORP, which measures the potential in a water sample across a platinum electrode, relative to the standard hydrogen electrode. ORP measurements are susceptible to several limitations, including disequilibrium conditions in the water and slow or low electrochemical response to the electrode surface for some redox couples. However, in waters with high iron concentrations, such as some mine-affected waters, ORP can be a valuable qualitative measurement.

Measure ORP in the field, either in situ or in a flask or beaker filled with sample as close to ambient conditions as possible. The following steps are for calibrating and measuring ORP in samples:

1. Attach the electrode to the meter (if not already in a sonde) and prepare the electrode according to the manufacturer's instructions (for example, adding filling solution and wetting the junction). The meter output will typically be in millivolts; if using the same meter for multiple measurements, the output setting may need to be adjusted for ORP. Record the electrode and filling solution details on the field sheet.
2. Suspend the electrode in the solution, and if using a container, do not let the electrode surface touch the bottom of the container.
3. When moving the electrode between solutions, rinse the electrode with DI water and gently blot dry with a laboratory tissue to prevent cross-contamination, taking care to not scratch the electrode surface.
4. Check electrode performance using a standardized ORP solution (for example, Zobell's solution or the manufacturer's ORP solution). The ORP solution may contain harmful compounds, so dispose of the waste properly. The expected value of the check solution is dependent upon the electrode-filling solution and is T dependent. It is useful to bring a table of expected values (in volts or millivolts) of the check solution with the specified electrode and filling solution combination as a function of T. These tables are usually available in the electrode manual or from the manufacturer. Ensure the value is ±10 millivolts of the expected value and record it on the field sheet. The check solution only needs to be measured before the first sample of the day, or if the electrode surface shows signs of any visible change (scratch, tarnish, and so forth), to verify the electrode's performance.

5. Place the electrode in the sample, allow the reading to stabilize, and record the ORP of the sample on the field sheet. ORP measurements may take as much as 15 minutes to stabilize, and ensure measurements are done at a constant T. If the reading does not stabilize in 15 minutes, record this information on the field sheet.
6. Clean and store the electrode according to the manufacturer's instructions.

It is preferable to measure ORP in the field. However, if it is not possible, an additional sample split may be collected, stored in a clean bottle, filled to the top of the bottle with sample water, and chilled during transport and storage at the laboratory. Ensure measurements are made within 48 hours to minimize changes to sample chemistry, and do not proceed if there are visible precipitates or color changes in the sample split. Record this deviation in the field measurement on the field sheet.

Dissolved Oxygen

Another optional measurement is DO, which can be measured by a colorimetric method (Rhodazine D or Indigo Carmine) in the field with a commercially available kit or a DO probe and meter. The kits rely on reagent ampules that react with sample water to produce a color. The kits may have a calibration color chart or use a portable spectrophotometer to convert color saturation in the reacted ampule to a concentration of DO. It is important that the manufacturer's instructions are followed carefully, particularly regarding collection requirements and wait times for analysis because inclusion of atmospheric oxygen can compromise the analysis. There are several caveats with this technique that may be encountered at mine or mill sites:

- Copper, chromium, and iron can affect the results, causing a significant analytical bias; refer to the kit manufacturer's instructions for more details. Although it may not be possible to know in the field whether these elements are present at concentrations that affect the DO results, when the cation results are returned, the DO data may need to be qualified or discarded if they exceed the method limits for DO.
- Low pH waters (pH of 2 or less) can compromise the analysis. Do not report the DO if the pH is low enough to bias the results.

Dissolved oxygen may also be measured by a meter and calibrated DO probe, if available. There are several types of probes that each have unique maintenance requirements. A meter and probe setup is more expensive than the colorimetric method and requires calibration by one of several methods. In addition, membrane probes may become clogged by ferric iron precipitates in mine-affected waters and produce incorrect results.

Regardless of which method and manufacturer is chosen for DO measurements, record the details on the field sheets. It is important to measure DO in situ or as close to the source water as possible because DO will ingas or degas throughout time and as the water T changes. Also, relative saturation depends upon T and elevation, and it is important that these parameters are recorded on the field sheet.

Field Sheet, Notes, and Observations

Assign a unique location ID to each site and sample unit (for solid samples). When sampling each location, assign a different field sheet for each sampling event. The field sheet may be a digital or hard-copy format. Example field sheets are given for reference in appendix 1, but any format may be used as long as it includes the following information:

- Site ID and sample unit (if applicable), creating a unique location ID.
- Description of the sample type (for example, tailing composite sample or pit lake water sample).
- Geospatial data (refer to the "Collecting Geospatial Data" section).
- Date and time of sample collection.
- Sampling staff names.
- Weather conditions at the time of sample collection, and recent weather affecting collection conditions, if applicable.
- Other notes and observations.
- Photograph of the sample site(s). For composite samples, an overview photograph of the site is sufficient.

For water samples, additional information includes the following:

- Checklist of splits of samples collected for water samples.
- Field parameters (pH, T, SC, ORP, and DO) and associated calibration, check standard information, measurement units (as applicable).
- Filtration system used.
- Flow measurement technique and source of data.

Additional Suggested Observations

There are many additional pieces of useful information that can be documented in the field and subsequent laboratory notes. Additional information can include observations regarding the following:

- Munsell field soil color and notes on textures for solid samples.
- Depth of waste piles and method used, if measured in the field.
- Description of location, type, and extent of efflorescent salts.
- Moisture content of solids.
- Vegetation site cover.
- Presence of wetlands or pools of water not sampled.
- Erosional features.
- Hardpans.
- Seeps and description of biofilms, if present.
- Paste pH.
- Handheld X-ray fluorescence, if used.
- Additional photographic documentation.

To prevent data loss, digitally back up field sheets by scanning hard-copy sheets or use a separate backup for digital sheets after returning from the field. Digital versions of all field sheets are part of the final data delivery requirement to the USGS.

Collecting Geospatial Data

Geospatial data include sample unit and subsample locations, water sample locations, and site and feature boundaries.

Tailings—Composite Sample Sites

Subsample locations can be selected before field work using aerial photographs, satellite imagery, or other current maps. If any deviations are made from the planned locations, record the actual subsample locations onsite with a GPS, along with the datum used. If subsample locations are determined in the field, record all subsample locations onsite. Record notes on how subsample locations are determined and any deviations on the field sheets. Document all actual subsample locations in the final report.

Water—Sampling Locations

Record each water sample location onsite with a GPS, along with the datum used.

Site and Feature Boundaries—Polygons

Site boundaries may be determined by the perimeter of all the mine features, land ownership, or natural features such as topography. Within a site, there may be one or more mine features, including tailings piles, mine workings, mill workings, pit lakes, adits, waste rock piles, and overburden piles. The perimeter of the mine features that were sampled is important to measure or estimate. If it is safe, collecting waypoints along the perimeter of the mine feature that was sampled can provide the necessary GIS polygon. Alternatively, current satellite imagery, airborne photography, or other current data can be used to estimate the mine feature perimeter if the method used is documented in the final report. If multiple mine features will be sampled at a site, ensure each feature has a perimeter defined. For flowing adits, a single GPS point of emergence will suffice for the feature location.

Volume Estimates of Tailings and Other Mine Waste Piles

Approaches for estimating volumes and masses of mine waste piles may vary, particularly with regard to the pile depth. Waste pile boundary polygons can be combined with a depth estimate to calculate volume. Additional assumptions about density can be used to estimate mass. Clearly describe the methodology used, including assumptions for estimating volumes of solid piles in the final report.

Volumes of Flowing Adits and Pit Lakes

Use the discharge value at the time of sampling to calculate the metal loads. If the flow is highly seasonal, any data on seasonal flow variation are useful in refining the load calculations. Discuss this information and methodology in the final report. For pit lakes, estimate or measure the volume of water. The data may come from a site manager, direct measurement by geophysical or remote sensing data, or estimates based on geospatial data. Discuss this information and methodology in the final report.

Sample Submission and Geochemical Analyses

Geochemical analyses are performed at the USGS and USGS contract laboratories. All samples are handled and analyzed using the same sample methods to provide an internally consistent and comparable dataset. Solid samples are ground and homogenized before analysis. Water samples for anions and alkalinity samples have a 30-day holding time from collection to analysis, so timely shipping of these samples is essential for quality analysis.

Sample Submission

As of 2025, samples will be submitted to the USGS Geology, Geophysics, and Geochemistry Science Center's Sample Control group (https://www.usgs.gov/centers/gggsc/science/analytical-chemistry) using the following process:

1. Email Sample Control at MTSampleControl@usgs.gov specifying "Earth MRI Mine Waste Characterization" to obtain the sample submittal spreadsheet and fill out the spreadsheet. Most spreadsheet fields are completed with drop-down boxes. For those fields without drop-down boxes, such as sample comments, additional information less than or equal to 255 characters can be added. Samples are grouped into jobs that are assigned a unique identifier by Sample Control. Take note of the following when submitting samples:

   A. Only one sample media type per job.

   B. Maximum of 45 samples per job.

   C. All samples in a job are analyzed by the same set of analytical protocols.

   D. Assign each sample a unique field identifier.

   E. Include a project identifier with each job.

2. Email the spreadsheet(s) to MTSampleControl@usgs.gov. Also, print a copy of the submittal spreadsheet for each job and include it in the shipment of samples (every box or cooler). If water samples are being shipped, place the printed sheet in a sealable bag to prevent water damage.

3. Confirm that the paperwork matches the physical samples, including that the sample names in the spreadsheet match the sample labels. If errors are found, contact Sample Control by email.

4. Prevent the spread of invasive species. Make sure that the samples are free of roots, seeds, and insects. Some samples from certain areas are subject to U.S. Department of Agriculture (USDA) Federal regulations. Failure to follow the required shipping procedures will result in the immediate destruction or disposal of samples upon receipt by USGS Sample Control in accordance with USDA guidelines.

5. Package the samples for shipping and make sure each sample bag or container is sealed to prevent spillage, leakage, and cross-contamination:

   A. Consider using hard-sided shipping containers rather than cardboard boxes. Fill the empty spaces in the shipping container to prevent sample damage during shipping.

   B. Water samples requiring chilled conditions (anions and alkalinity samples) need to be shipped on ice or with cooling packs in a cooler. Place ice in leak-proof bags (or double bag) because standard commercial bags for ice are not sufficient to prevent melted water from leaking and contaminating samples.

   C. Use tape to secure all sides of the shipping container. Carefully seal the shipping container openings because rough handling of heavy containers often causes sealed edges of containers to split open.

   D. Do not exceed a total weight of 23 kilograms for hard-side containers and 15 kilograms for boxes.

   E. Include a return label if you would like your coolers or other shipping containers returned to you.

6. Contact USGS Sample Control and confirm the shipping address before shipping samples. As of 2025, the following address is where samples are shipped:

   Sample Control

   U.S.Geological Survey

   Denver Federal Center, Building 20

   Box 25046, MS 973

   Denver, CO 80225

Solid Phase Analyses

Solid samples will be crushed, ground, and homogenized by the USGS before submitting samples to a contract laboratory for analysis. The methods used by the analytical laboratories are briefly described in this section.

Metals and Other Elemental Analyses

Cations and metals are measured by preparing the sample using a sodium peroxide fusion method in which samples are fused at 750 degrees Celsius (°C) with sodium peroxide and the fusion cake is redissolved in dilute nitric acid.

Sixty-one elements are analyzed in the dissolved fusion cake by inductively coupled plasma mass spectrometry (ICP–MS) and ICP optical emission spectroscopy (ICP–OES). Elemental analytes are Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ge, Hf, Ho, In, K, La, Li, Lu, Mg, Mn, Mo, Nb, Nd, Ni, P, Pb, Pr, Rb, Re, S, Sb, Sc, Se, Si, Sm, Sn, Sr, Ta, Tb, Te, Th, Ti, Tl, Tm, U, V, W, Y, Yb, Zn, and Zr. Major elements are determined by dissolving the sample into a lithium metaborate and tetraborate fusion disk and measuring major element oxides by wavelength dispersive X-ray fluorescence spectrometry. Analytes are Al_2O_3, BaO, CaO, Cr_2O_3, Fe_2O_3, K_2O, MgO, MnO, Na_2O, P_2O_5, SiO_2, TiO_2, SrO, V_2O_5. Loss on ignition is also measured. Gold, palladium, and platinum are determined by a lead fusion fire assay in which the precious metals are separated from the rest of the sample in a flux, followed by lead and other base metals removal. The precious metals are dissolved in aqua regia and analyzed by ICP–MS and ICP–OES. Inorganic carbon is determined by treating the sample with perchloric acid ($HClO_4$) and measuring the resulting carbon dioxide with an infrared detector. Total sulfur and total carbon are determined by sample combustion and infrared detection of sulfur dioxide and carbon dioxide gas. Mercury is determined by cold vapor atomic absorption spectroscopy. Fluorine is determined by digestion and measurement with an ion-selective electrode.

Mineralogy

Mineralogy is determined by powder X-ray diffractometry using the spiked Rietveld method (Albinati and Willis, 2006) by USGS laboratories.

Acid-Base Accounting

The method used for acid-base accounting is based on the method developed by the U.S. Environmental Protection Agency (Sobek and others, 1978). A sample aliquot is subjected to a preliminary fizz test to determine the volume and concentration of acid needed for the analysis. Another aliquot is reacted with water to measure the paste pH. Based on this information, the sample is dosed with acid and backtitrated with a base. From this information, the neutralization potential (NP), the acid generating potential (AP), and the net neutralization potential (NNP) can be calculated.

Quality Assurance and Quality Control

The USGS QA/QC practices include the submission of analytical duplicates and standard reference materials as unknowns to the analytical laboratories for all methods. Twenty percent of the submitted samples are analyzed as analytical duplicates. In addition, reference materials similar to the submitted matrix are included as blind samples to the laboratory. The performance of the duplicates and reference material analysis is evaluated and reported to the submitter.

Aqueous Analyses

Acidified samples are analyzed by ICP–MS and ICP–OES for Ag, Al, As, Au, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ge, Hf, Hg, Ho, In, Ir, K, La, Li, Lu, Mg, Mn, Mo, Na, Nb, Nd, Ni, P, Pb, Pd, Pt, Rb, Re, Rh, Ru, S, Sb, Sc, Se, Si, Sn, Sr, Ta, Tb, Te, Th, Tm, Ti, Tl, U, V, Y, Yb, Zn, and Zr. Anions are analyzed in filtered, unacidified samples by ion chromatography. Whether an alkalinity or acidity titration is performed is determined by the field pH of the sample. The maximum time that samples can be held before analysis (holding time) is 1 year for cations and 30 days for anions and alkalinity or acidity. The data are qualified when the data are released, if samples are analyzed after their holding time has passed.

Quality Assurance and Quality Control

In addition to field blanks and duplicates, the USGS will submit additional reference water samples as blind samples to the laboratory. The performance of the blanks, duplicates, and reference material analysis is evaluated and reported to the submitter.

Sample Archive

Solid samples are archived at the USGS Geology, Geophysics, and Geochemistry Science Center in Denver, Colorado. Bulk materials (before crushing and grinding; 300–500 g) as well as ground and prepared materials (30–50 g) are archived for future analyses. Water samples are not archived.

Data Reporting

All geochemical data are reviewed and publicly available through USGS data releases on ScienceBase (https://www.sciencebase.gov/catalog/). The data releases are revised and released multiple times per year, including all the data collected for mine waste characterization.

Summary

The U.S. Geological Survey Earth Mapping Resources Initiative program created these protocols to standardize field sampling of mine waste and mine waters carried out by State geological surveys and cooperators. Methods may be applied to other applications as appropriate to evaluate critical minerals in mine waste. The data provide important information that can be used in estimating critical mineral endowment nationwide.

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Appendix 1. Example Field Sheets

Optional example U.S. Geological Survey Earth Mapping Resources Initiative mine waste characterization field sheets for solids and water sampling are available for download at https://doi.org/10.3133/sir20255068.

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