

U.S. Geological Survey Field Leach Test for Assessing Water Reactivity and Leaching Potential of Mine Wastes, Soils, and Other Geologic and Environmental Materials



Techniques and Methods 5-D3

U.S. Geological Survey Field Leach Test for Assessing Water Reactivity and Leaching Potential of Mine Wastes, Soils, and Other Geologic and Environmental Materials

By Philip L. Hageman

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Section D, Geologic Analysis
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Conversion Factors

ISI to Inch/Pound

Multiply	By	To obtain
Length		
centimeter (cm)	0.3937	inch
micrometer (μm)	0.00003937	inch
millimeter (mm)	0.03937	inch
Volume		
liter (L)	0.2642	gallon
milliliter (mL)	0.000264	gallon
Mass		
gram (g)	0.03527	ounce
kilogram (kg)	2.205	pound

Abbreviations and symbols used in report

DI	deionized water
FLT	Field Leach Test
ICP–MS	inductively coupled plasma–mass spectrometry
mg/L	milligram per liter
SPLP	Synthetic Precipitation Leaching Procedure
USEPA	U.S. Environmental Protection Agency
USGS	U.S. Geological Survey
µg/L	microgram per liter
µS/cm	microsiemens per centimeter
<	less than
~	approximately

U.S. Geological Survey Field Leach Test for Assessing Water Reactivity and Leaching Potential of Mine Wastes, Soils, and Other Geologic and Environmental Materials

By Philip L. Hageman

Abstract

The U. S. Geological Survey (USGS) has developed a fast (5-minute), effective, simple, and cost-effective leach test that can be used to simulate the reactions that occur when materials are leached by water. The USGS Field Leach Test has been used to predict, assess, and characterize the geochemical interactions between water and a broad variety of geologic and environmental matrices. Examples of some of the samples leached include metal mine wastes, various types of dusts, biosolids (processed sewage sludge), flood and wetland sediments, volcanic ash, forest-fire burned soils, and many other diverse matrices. The Field Leach Test has been an integral part of these investigations and has demonstrated its value as a geochemical characterization tool. It has enabled investigators to identify which constituents are water reactive, soluble, mobilized, and made bioaccessible because of leaching by water, and to understand potential impacts of these interactions on the surrounding environment.

Introduction

Water-induced leaching of major elements, trace metals, acids, bases, organic constituents, pharmaceutical by-products, and other constituents from geologic and anthropogenically derived materials and the effects of resulting runoff on adjacent streams, water bodies, and ecosystems are common environmental concerns. Another concern is the direct or indirect uptake of potentially toxic constituents from the leachates into the food chain.

Because leaching is constantly taking place in the environment as materials are exposed, weathered, and influenced by natural precipitation, leachate in the form of runoff is produced and released into the environment on an on-going basis. Leachates are derived from materials that include the following: naturally mineralized soil, industrial sites, mining wastes from active or abandoned mine sites, construction sites, natural and amended soils, a wide variety of dusts, volcanic or other ash, dried sludge and sediments, and a nearly endless

variety of other materials. To address this potential, it is important to have a tool that aids the geoscientist in quantifying and understanding the leachability of these materials.

Laboratory leach studies have been a useful way to assess this potential. However, the leach tests most commonly used are often expensive, complicated, time consuming, and often require specialized, expensive equipment. In light of these factors, the USGS developed the Field Leach Test (FLT), which can be performed on-site or in the laboratory to simulate quickly, effectively, and inexpensively the chemical reactions that occur when materials are leached by water. The USGS FLT is summarized in Fact Sheet 2005-3100 (Hageman, 2005).

Development of the U.S. Geological Survey Field Leach Test

The USGS Field Leach Test (FLT) is based on a leach test developed by Hageman and Briggs (2000), which was originally developed to quickly assess, predict, and compare leachate geochemistry from historical metal mine dumps. The procedure uses readily available equipment and deionized water (DI) to leach the less than (<) 2-mm fraction of a sample using a 20:1 leaching ratio (20 parts leachate to one part solid). The leachate sample is hand shaken for 5 minutes and then allowed to settle for 10 minutes. After settling, subsamples of the leachate are used to determine pH, specific conductance (SC), and other characteristics. A portion of the leachate is then filtered using a 60-cm² syringe and 0.45- μ m filter, and subsamples of the filtrate are collected and preserved for geochemical analysis. After analysis, the element concentration trends (geochemical signatures) of the leachates are used to rank the waste piles for cleanup. Since its inception, use of the USGS FLT has expanded, and the procedure is now used to predict and characterize the leaching potential of a diverse variety of materials.

As part of developing the FLT, extensive research and comparative studies were undertaken using many other types of leach tests. After the initial set of tests, USGS scientists

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focused their comparison on studies between the USGS FLT and the U.S. Environmental Protection Agency (USEPA) Method 1312, Synthetic Precipitation Leaching Procedure (SPLP) (U.S. Environmental Protection Agency, 2002). Characteristics of the USGS FLT and USEPA Method 1312 are listed in table 1.

The comparison of these two leaching procedures was emphasized because the SPLP was one of the leach tests most commonly used for leaching studies of mine wastes. Comparison of mine waste leachate geochemical data produced by the two procedures showed that element-concentration trends produced using the 5-minute USGS FLT procedure are similar to those produced by the 18-hour USEPA SPLP leach test (figs. 1 and 2). Similar trends are seen when comparing other leachate characteristics, such as mine waste leachate pH from eight mine dump composites leached using the two procedures (fig. 3). As a result of this comparison and other studies, the USGS adopted the FLT as a procedure that can be used as a surrogate for the USEPA SPLP procedure. Complete results and the methodology used in this study are reported in Hageman and Briggs (2000).

An important and advantageous feature of the USGS FLT procedure is that it was designed to use the same extraction ratio (20 parts leachate to 1 part solid) as the SPLP leach test. Use of the 20:1 ratio ensures that the readily soluble components of a sample can be taken into solution without exceeding leachate saturation limits. Use of this leaching ratio also allows geochemical results produced using the USGS FLT to be compared directly with other leaching studies that have used the SPLP procedure. Finally, this leaching ratio ensures that the FLT provides sufficient leachate to obtain all desired measurements and elemental analyses. This is important because many leach tests, such as paste pH test, do not provide enough leachate for geochemical analysis and characterization.

Finally, the USGS FLT is convenient to use. It can easily be performed either on-site or in the laboratory with equipment and reagents that are readily available and can be taken to the field in the back of a vehicle. On-site leaching requires only the return of the preserved filtrate to the laboratory for analysis. In contrast, the USEPA SPLP procedure requires specific, costly equipment, and must be performed under stringent conditions in the laboratory.

Equipment Needed to Use the U.S. Geological Survey Field Leach Test

Most of the equipment needed to use the FLT is shown in figure 4.

1. Laboratory or field balance. Field balance shown in figure 5 (Pocket Pro 150-B manufactured by ACCULAB, Newton, PA.) is capable of weighing 50 g of sample.
2. Disposable styrene weighing boats capable of holding 50 g of sample.
3. 1.0-L wide-mouth, capable plastic bottles.
4. 1,000-mL graduated cylinder.
5. Deionized water.
6. Disposable 50-mL styrene beakers.
7. pH meter and electrode.
8. Specific conductance meter.

Table 1. Leaching conditions of the U.S. Geological Survey Field Leach test and the modified Environmental Protection Agency Method 1312, Synthetic Precipitation Leaching Procedure

[USEPA, U.S. Environmental Protection Agency; SPLP, Synthetic Precipitation Leaching Procedure; USGS FLT, U.S. Geological Survey Field Leach Test; <, less than; ~, approximately]

Characteristics	Modified USEPA 1312 SPLP	USGS FLT
Test type	batch	batch
Leachate to solid ratio	20:1	20:1
Leachate composition	60/40 H ₂ SO ₄ /HNO ₃	deionized water
Leachate pH	4.2 (for mine wastes)	~ 5.7
Particle size used	< 1 centimeter	< 2 millimeters
Sample mass	100.0 grams	50.0 grams
Duration of agitation	18 hours	5 minutes
Agitation method	end-over-end rotary	hand shaken
Filtration	positive pressure	syringe
Filter type	borosilicate glass fiber	nitrocellulose
Filter pore size	0.70 micrometer	0.45 micrometer

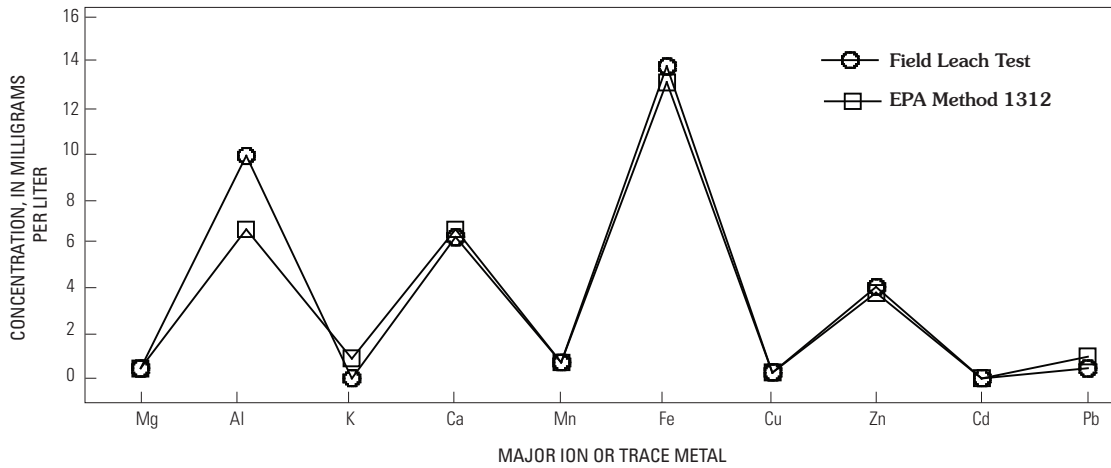


Figure 1. Leachate geochemistry comparison of the Sunday #2 mine-waste composite sample using the 18-hour U.S. Environmental Protection Agency Method 1312, Synthetic Precipitation Leaching Procedure (U.S. Environmental Protection Agency, 2002) and the 5-minute U.S. Geological Survey Field Leach Test.

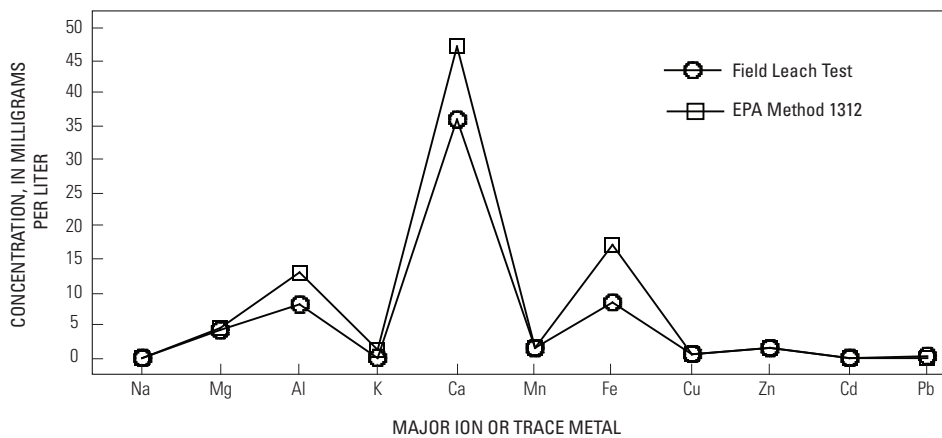


Figure 2. Leachate geochemistry of the Yukon mine-waste composite sample using the 18-hour U.S. Environmental Protection Agency Method 1312, Synthetic Precipitation Leaching Procedure and the 5-minute U.S. Geological Survey Field Leach Test.

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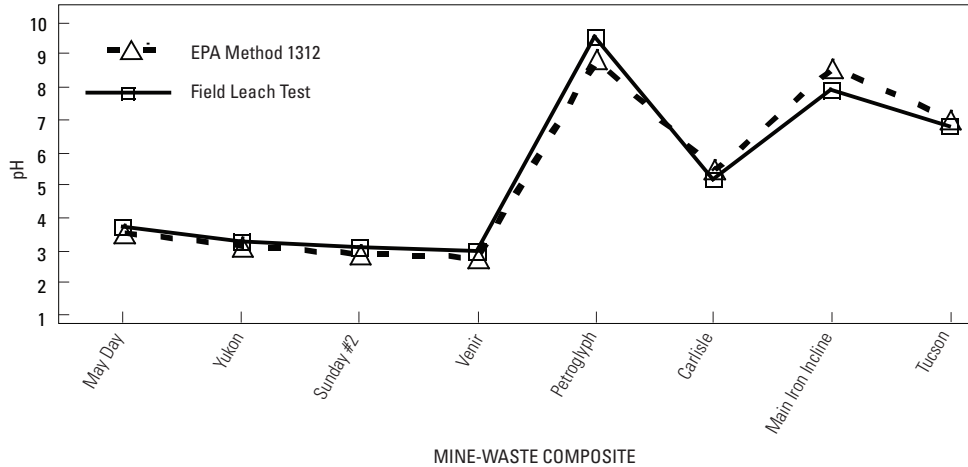


Figure 3. Leachate pH data for eight mine dump composite samples using the 18-hour U.S. Environmental Protection Agency Method 1312, Synthetic Precipitation Leaching Procedure and the 5-minute U.S. Geological Survey Field Leach Test.



Figure 4. Most of the equipment and materials needed to leach a sample using the Field Leach Test.

9. Other meters or kits as desired, such as alkalinity.
10. 60-cm² Leur Lock syringes.
11. 0.70- μ m glass fiber pre-filter, and 0.45- μ m nitrocellulose, Leur Lock capsule filters.
12. Prepared (acid washed if needed) bottles to store filtered leachate for analysis.

Note: Bottles are acid washed with 10 percent HCl and triple rinsed with deionized water.

Procedure for Using the U.S. Geological Survey Field Leach Test

1. 50.0 g of prepared sample (see following sample collecting and processing section) is weighed using a small field balance (fig. 5) or a laboratory balance, and carefully added to a 1.0-L wide-mouth, plastic bottle.
2. 1.0-L deionized water is measured in 1,000-mL clean graduated cylinder and slowly added to the bottle so that no dust is lost. (**NOTE:** Depending upon the amount of solid material available, other leaching volumes can be used as long as the 20:1 water-to-solid ratio is maintained.)
3. The bottle is tightly capped and vigorously hand shaken for 5 minutes. Alternatively, the sample can be shaken on a bench-top horizontal shaker for 5 minutes.
4. After shaking, the bottles are turned upright and the contents are allowed to settle for 10 minutes.
5. After settling, unfiltered subsamples of the leachate are dispensed into disposable plastic beakers and measured for pH, specific conductance, alkalinity, and other characteristics.
6. A portion of leachate is filtered using a 60-cm² Leur Lock syringe and a 0.45- μ m pore-size nitrocellulose filter. If filtration is difficult, a 0.70- μ m glass fiber pre-filter can be used in conjunction with the 45- μ m filter in a serial manner. Subsamples of the filtrate are collected and preserved for analysis.



Figure 5. Close-up of small field balance that can be used to weigh sample for leaching on-site using the U.S. Geological Survey Field Leach Test.

Collecting and Processing of a Composite Sample for Use in the Field Leach Test

A “representative” sample must be collected prior to using the USGS FLT. Accurate, appropriate sampling is extremely important to produce meaningful results. An important first step that must be taken prior to designing a sampling plan is to accurately define the question to be answered with the leaching study. (Is the study trying to identify surficial run-off potential or is it more concerned with the long-term leaching characteristics of a sample?) Only after the goal of the leaching study has been defined can the sampling objectives and sampling strategy be designed.

Many studies have been conducted in the science and strategies of sampling and a broad range of techniques and approaches have been used successfully. For many studies, scientific investigators have found that collecting a composite sample of a material is an effective and representative way to sample a selected population. An important advantage of using the composite method of sampling is that it saves time and money because “one 30-increment dump-composite sample collected using this sampling strategy contains as much information, relative to average value, as 30 individual grab samples at 1/30 of the analytical cost” (Smith and others, 2000). The composite sampling method used by the USGS is statistically based and is used to characterize the average geochemical properties and/or geochemical behavior of a material (Smith and others, 2000). Using this composite sampling method, numerous increments from a site are collected and mixed together to form a “bulk” composite sample that statistically represents the area sampled. Some examples of studies that have used composite sampling

are Hageman (2004), Hageman and Briggs (2000), Piatak and others (2004), Smith and others (2006), Kathleen S. Smith, U.S. Geological Survey, oral commun. (2006), Van Gosen and others (2000, 2005).

The composite sampling procedure has worked effectively for the collection of metal-mining waste, soils, biosolids, sediments (any type), crushed and ground rock samples, many types of dusts, and volcanic ash. Following is a description of the materials needed and the procedure used to collect and process a mine-waste composite sample. Keep in mind, that the step-by-step process presented here can also be used to collect a composite sample of the other types of geologically derived material.

Equipment Needed to Collect a Composite Sample

Most of the equipment needed to collect a composite sample using this method is shown in figure 6.

1. One or more (depending on number of people collecting sample) 1.5 – 2 gallon heavy duty sample collection pails with handle.
2. 5-gallon plastic bucket/s (for mixing bulk composite sample).
3. Leather gloves.
4. Small stainless steel or heavy duty plastic garden scoops.
5. Stainless steel three-prong garden scrapers that can be used if the surface material is hard.
6. 3- by 5-foot (or larger) plastic tarp, or large, heavy duty garbage bags (for drying sample).



Figure 6. Materials needed to collect a composite sample.

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7. Global Positioning System for identifying sampling location.
8. 9.5-mm, 5.0-mm, 2.0-mm stainless steel sieves, along with a catch pan and lid (for sieving the bulk composite sample).
9. Jones splitter®, or materials needed to cone and quarter samples.
10. Large re-sealable plastic storage bags for storing splits of the < 2-mm composite sample.

Procedure for Collecting and Processing a Mine-Waste Sample

There are two methods that can be used to collect a surficial (upper 15 cm) mine-waste composite sample. In the first method, the mine waste pile is divided into a grid of at least 30 cells of roughly equal surface area. Next, increments (subsamples) are collected of the surficial material from each cell using a stainless steel or plastic shovel and plastic bucket. After collection from all cells, the increments are mixed together in a 5-gallon bucket or plastic tub.

The second method that can be used is to collect a non-grid composite sample. For this procedure, instead of dividing the mine dump pile into grided cells, the composite sample is collected by randomly walking back and forth across the entire mine waste pile or site and collecting a minimum of 30 increments. When collecting in this manner, it is important to collect the increments in a random, nonbiased manner. An example of a good way to ensure unbiased sampling is to collect increments at specific intervals, say every ten paces regardless of what the materials look like. Refrain from collecting from an area just because it looks interesting since this will introduce bias into the sample. For very large mine waste piles or sites, a nongrid composite sample could be collected randomly along transects of the pile. After collection, all increments are combined to form the bulk composite. Note that for both these collection methods, the final composite sample should weigh at least 1,000 g (1 kg) after sieving.

After collection, further processing needs to take place before the composite sample can be leached:

1. If the bulk composite sample is wet, it must be air dried at ambient temperature, either on-site or in the laboratory. Dry the sample by spreading it out on a disposable plastic garbage bag or plastic tarp on a flat surface, and then periodically mix the sample with a plastic scoop until all the material is dry. Never use heat to dry the composite, because heat could alter the geochemical characteristics of the sample.
2. After drying, a portion of the bulk composite can be split from the sample and saved for archive and further studies

(for example, other leach studies or mineralogy). After splitting, the remaining composite material is dry sieved [< 2 mm (10 mesh) for most samples] using stainless steel sieves. Note that sieves can be stacked to make sieving easier. To do this, stack three sieves (9.5 mm, 5.0 mm, and 2.0 mm), along with a catch pan and lid. Place portions of the bulk composite material into the top sieve (9.5 mm), and as the stack is agitated, the < 2 -mm fraction settles into the catch pan and is saved. Discard the material in the three sieves. Note that some samples do not need to be sieved (for example, ash or dusts) because they are already < 2 mm.

3. After sieving the entire bulk composite, place the < 2 -mm material in 1-gallon re-sealable plastic bags and store until needed.

Studies That Have Used the U.S. Geological Survey Field Leach Test

The USGS FLT has been used in diverse investigations of a broad range of sample types that include studies detailing the geochemical characterization of metal mine wastes (Diehl and others, 2005, 2006; Hageman, 2004; Hageman and Briggs, 2000; Moehle and others, 2005; Piatak and others, 2004; Kathleen S. Smith, U.S. Geological Survey, oral commun., 2006; Van Gosen and others, 2000, 2005; Wildeman and others, 2004). Other applications include environmental studies of the World Trade Center area after the September 11, 2001, attack (Clark and others, 2001; Plumlee and others, 2005); studies of the effects of surface applications of biosolids on soil, crops, ground water, and streambed sediment (Yager and others, 2004); as well as leach studies of mine-dump cores, naturally mineralized soils, forest-fire burned soils, dried chemical slurries (used to fight forest fires), various types of eolian dust, volcanic ash, and dried Hurricanes Katrina and Rita flood sediments (Plumlee and others, 2006), as well as stream and wetland sediments.

Leachates produced using these procedures have been analyzed for a wide variety of constituents. In addition to characterization of inorganic constituents, FLT leachate has been analyzed for organic and pharmaceutical compounds (Yager and others, 2004). Field Leach Test leachates have also been used for bioassessibility testing.

These studies have shown that the USGS FLT is a useful tool for geochemical characterization of the reactive, readily soluble, and potentially bioavailable constituents that are mobilized when these materials are leached by water.

Results

Some examples of geochemical results produced using the FLT procedures are presented here. These data show leachate

geochemical signatures for selected inorganic constituents in a wide variety of materials. In these examples, data are presented for leachate pH and specific conductance, as well as element concentration data that were generated using inductively coupled plasma–mass spectrometry (ICP–MS) (Lamothe and others, 2002). Comparison of these data show that unique geochemical profiles for trace metals (fig. 7a), and pH and specific conductance (fig. 7b) are produced, identified, and compared for a variety of sample matrices.

In other examples, FLT leachate geochemical data from one mine waste site can be plotted against other sites to compare their geochemical signatures. Examples of these types of comparisons are shown in figures 8a, 8b, and 8c. Field Leach Test leachates of other materials, such as volcanic ash, are compared in a similar manner (figs. 9a, 9b, and 9c), along with World Trade Center dust (figs. 10a, 10b, and 10c).

All of these comparisons show the unique leachate geochemical fingerprints that are produced using the USGS FLT,

and how useful leachate geochemical profiles can be whether assessing one site or comparing a number of different sites.

Summary

The U.S. Geological Survey Field Leach Test has proven to be a valuable tool for a diverse group of investigators including Federal, State, and other governmental agencies, environmental professionals, and academia. The procedure has been used extensively on-site and in the laboratory and has proven effective for the characterization of geologic and environmental materials for a variety of geochemical, environmental, and toxicological investigations. The FLT is very useful in revealing the readily soluble, water reactive geochemical components and characteristics of a sample, and has been a critical and integral part of these studies.

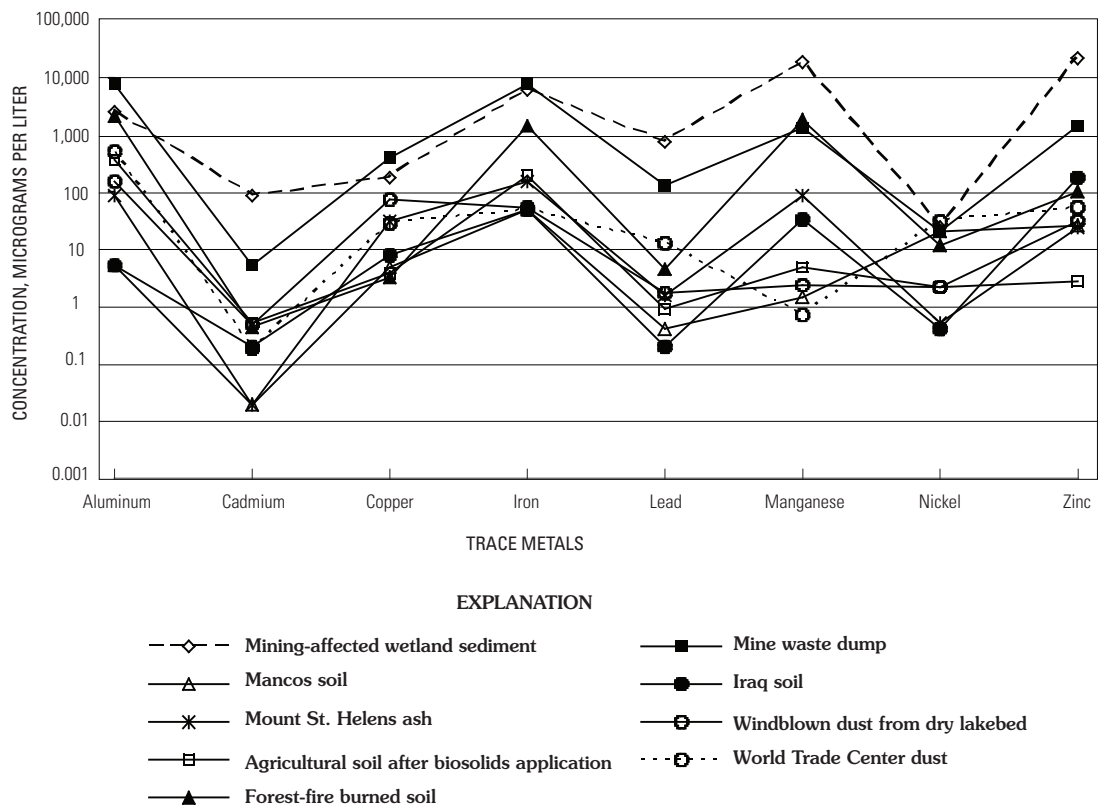


Figure 7a. Selected leachate trace metal concentration signatures for a variety of matrices leached using the U.S. Geological Survey Field Leach Test.

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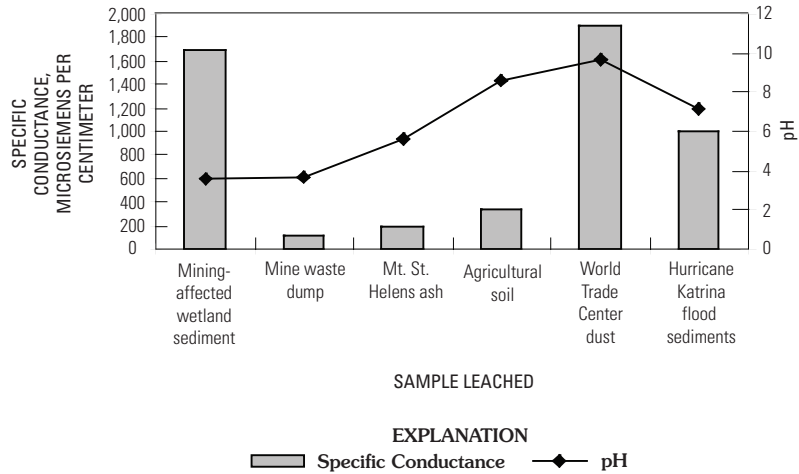


Figure 7b. Field Leach Test leachate pH and specific conductance signatures for some of the samples shown in figure 7a.

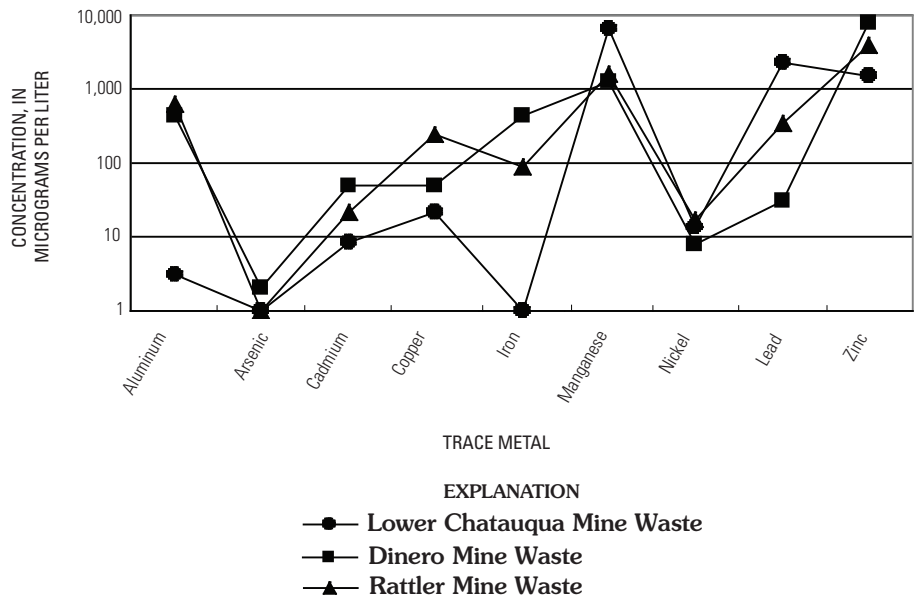


Figure 8a. Field Leach Test leachate geochemical trends for selected trace metals from three different surficial mine-waste composite samples collected from mine-waste sites in Colorado.

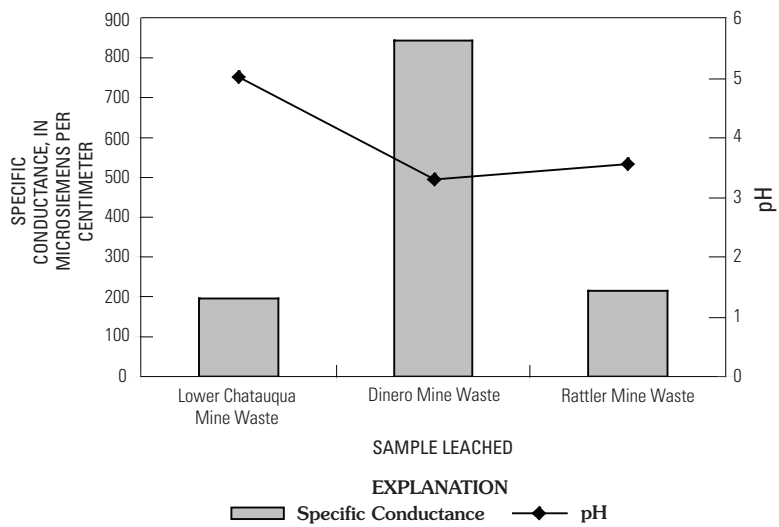


Figure 8b. Field Leach Test leachate geochemical trends for pH and specific conductance for three mine-waste composite samples.

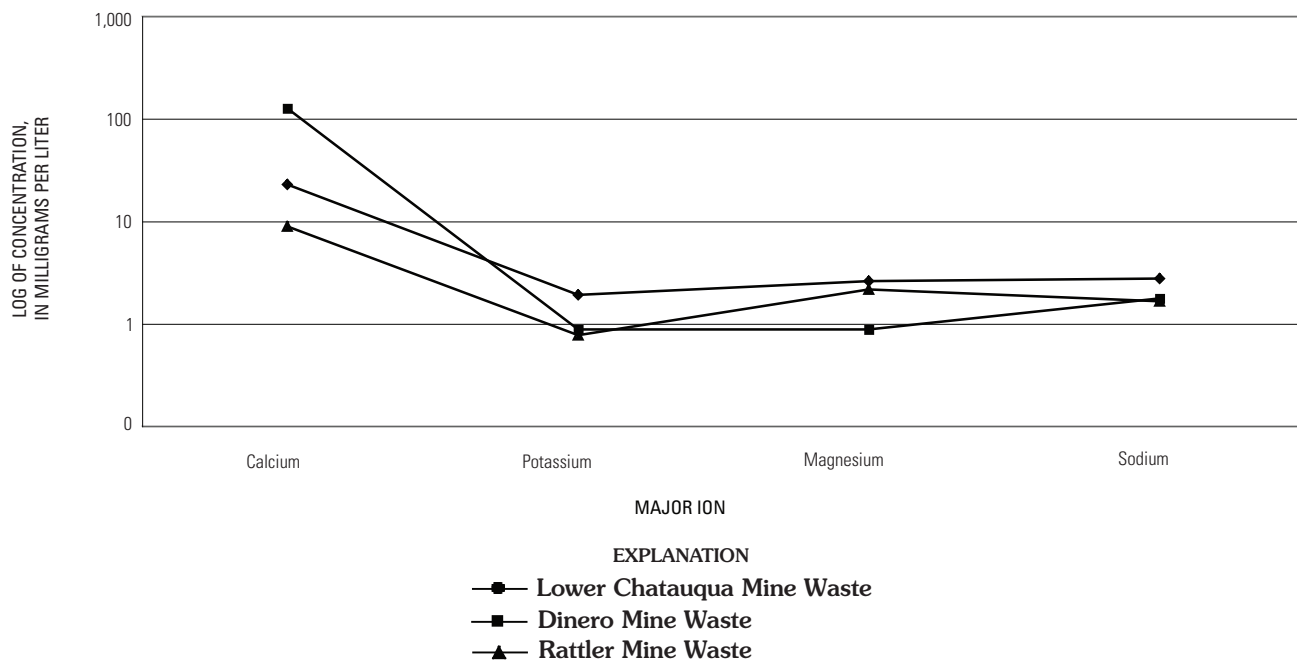


Figure 8c. Field Leach Test leachate geochemical trends for selected major ions for three mine-waste composite samples.

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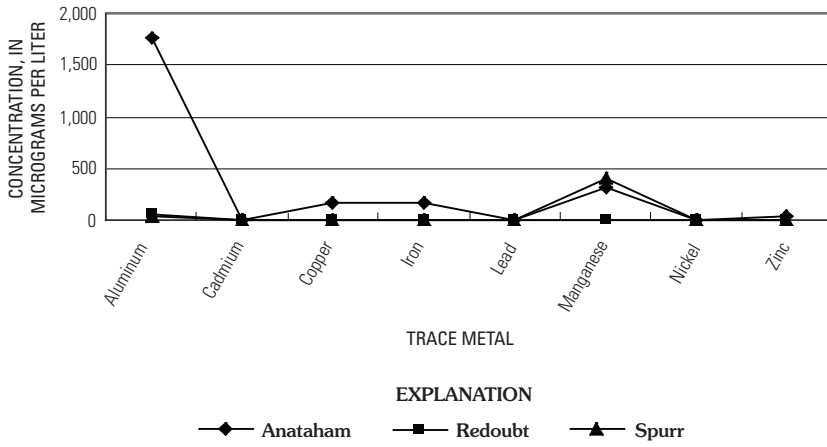


Figure 9a. Field Leach Test leachate geochemical trends for selected trace metals from three different volcanic ash composite samples.

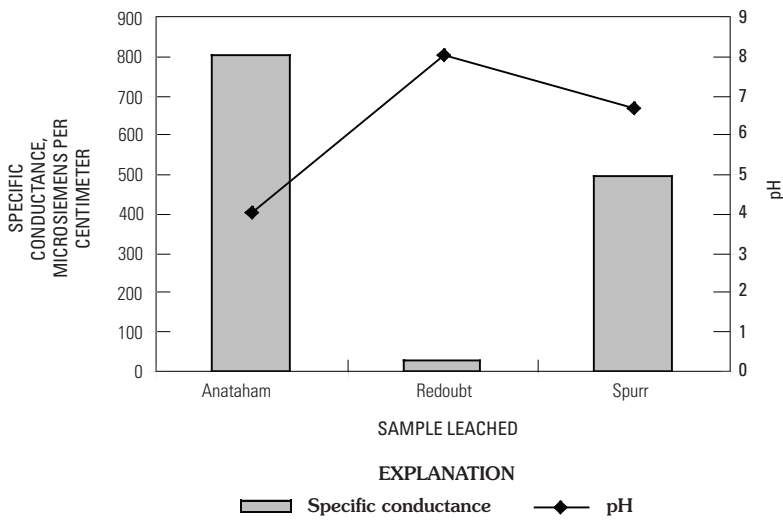


Figure 9b. Field Leach Test leachate geochemical trends for pH and specific conductance for three different volcanic ash composite samples.

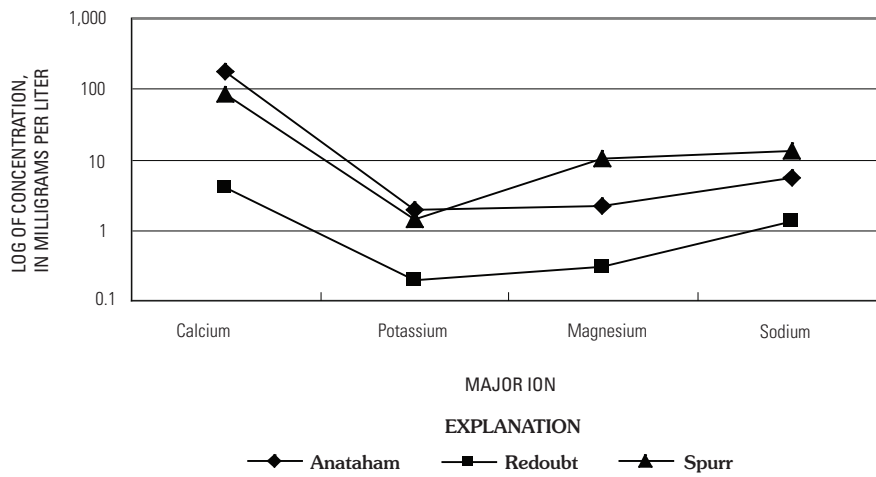


Figure 9c. Field Leach Test leachate geochemical trends for selected major ions from three different volcanic ash composite samples.

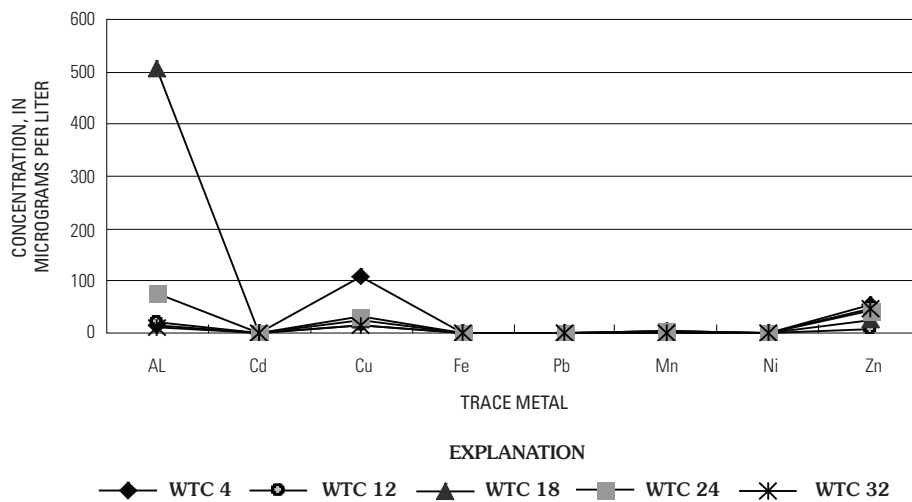


Figure 10a. Field Leach Test leachate geochemical trends for selected trace metals from five World Trade Center (WTC) dust composite samples.

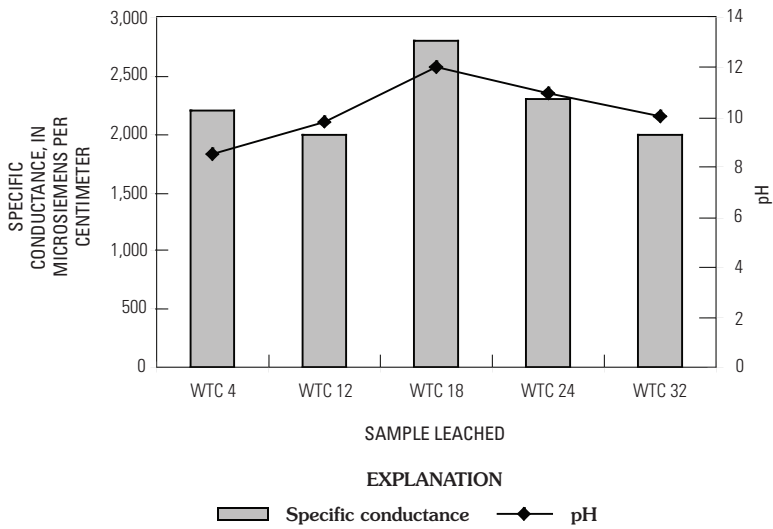


Figure 10b. Field Leach Test leachate geochemical trends for pH and specific conductance for five World Trade Center (WTC) dust composite samples.

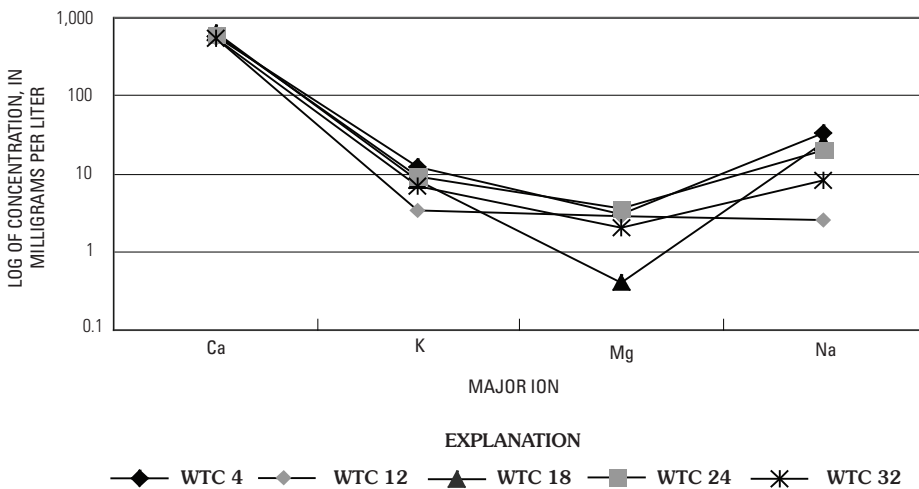


Figure 10c. Field Leach Test leachate geochemical trends for selected major ions for five World Trade Center (WTC) dust composite samples.

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