Sampling Strategy for the Rapid Screening of Mine-Waste Dumps on Abandoned Mine Lands

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ABSTRACT

We developed a statistically based strategy for sampling the surficial material of mine-waste dumps for use in screening and prioritizing historic dumps on abandoned mine lands. This sampling strategy entails the collection of a representative composite sample from individual dumps and allows for regional or watershed-based assessments. 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.

INTRODUCTION

The mine-waste dump sampling strategy discussed in this paper was developed during the U.S. Geological Survey, Mineral Resources Program Solid-Phase Sampling Workshop, March 24-26, 1997, in Denver, Colorado, taught by Charles Ramsey. Workshop participants made valuable contributions to the development of this sampling strategy. Many discussions in this paper originate from the workshop notes prepared by Charles Ramsey, which are based on Pitard (1993).

There are thousands of mine-waste dumps present on abandoned mine lands. Since they are historic dumps, most are relatively small. We sought to develop a cost-effective sampling strategy that could provide the foundation for screening and prioritizing these dumps on a regional or watershed basis. In screening, one is interested in the average behavior and potential environmental effects of a waste dump. Leaching tests are one of our main screening procedures to evaluate and prioritize the dumps. Because we are concerned with average properties of mine-waste dumps, our sampling strategy entails collection of a composite sample from each dump. The U.S. Environmental Protection Agency states that "composite samples reflect a physical rather than a mathematical mechanism for averaging. Therefore, compositing should be generally avoided if population parameters other than a mean are of interest (e.g., percentiles or standard deviations)" (U.S. Environmental Protection Agency, 1996). In other words, compositing is an acceptable sampling procedure when average properties are of interest.

The sampling strategy presented herein is appropriate for screening purposes. When more detailed site characterization is required, other sampling strategies may be employed depending on the objectives of the work (e.g., MEND, 1989, 1994; Bennett et. al., 1997; Price and Errington, 1997; Price, Errington, and Koyanagi, 1997; Price and Kwong, 1997; Price, Morjn, and Hutt, 1997; Runnells et. al., 1997). However, it should be noted that the statistically based sampling strategy presented herein could be adapted to the sampling of other target populations such as an individual waste-dump lobe, pit bench, dump lift, geologic unit, or other "operational" units.

Collecting a representative sample of mine-waste material can be a difficult task due to the compositional, spatial, and size heterogeneity of the waste material. The emphasis of our sampling strategy is to try to control heterogeneity in the collection of a representative sample, where heterogeneity refers to the constituents) of interest. To do this, a careful study of the sampling errors involved in sample collection is necessary. In this paper we discuss issues relating to sampling error, our mine-waste dump sampling

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strategy, and results from a particle-size study to evaluate leaching behavior of different size fractions of mine-waste material.

DEFINING OUR TARGET POPULATION

To prevent sample bias, it is essential that all of the target population is accessible and that the sampling is performed in such a manner that all particles in the population have an equal chance of selection. We have defined our target population as the surficial material (upper 15 cm) of a mine-waste dump. We limited our target population to the surficial material because of the ease of collection and the expectation that the surficial material is the most likely material to impact runoff from snowmelt and rain storms. Drilling costs for a large number of mine-dump sites would be prohibitive, and many mine-dump sites on abandoned mine lands are relatively inaccessible and do not lend themselves to drilling or trench sampling. Although we limited our efforts to the surficial material, this sampling strategy could be adapted to apply to drilling or subsurface trench sampling.

Due to the immense size heterogeneity in many mine-waste dumps, it is also necessary to define the size fraction of the target population. Because we are collecting material from historic dumps, the mine-waste material is weathered and secondary weathering products are present We chose to collect the < 2 mm size fraction of the mine-waste material. Our hypothesis is that smaller size fractions are generally the most reactive and would control the leaching behavior of the waste material. Price and Kwong (1997) recommend a separate analysis of the < 2 mm fraction when evaluating weathering effects of waste-rock dumps. This is because all the mineral grains are physically available in the < 2 mm fraction. They note that there is a tendency for neutralization potential depletion and metal accumulation in the < 2 mm fraction when compared with whole-sample data in strongly weathered samples. Hence, collection of the < 2 mm fraction should provide a worst-case scenario for our screening and prioritization procedures to determine potential environmental effects. Because this size-fraction choice is somewhat arbitrary, we performed leaching tests on a number of size fractions from several mine-waste dumps and report the results in the final section of this paper. The user of our sampling strategy may choose to collect a larger size fraction; however, collection of a larger size fraction will result in collection of a larger total sample mass (see equation in the following section). We sought to keep the sample mass to a weight that could be easily carried in a backpack after sample collection is performed at several mine-waste dump sites.

SAMPLING ERRORS

Sampling errors can be categorized into seven major groups: fundamental error, grouping and segregation error, delimitation error, extraction error, preparation error, cycles, and trends. Pitard (1993) provides a detailed discussion of sampling errors. Fundamental error results from the compositional heterogeneity of particles. Grouping and segregation error is a function of the nonrandom distribution of particles and the fact that particles are collected in groups rather than individually. Delimitation error and extraction error are both related to the choice and use of sampling tools; collectively they are termed the materialization error. Preparation errors take place after sample collection and before analysis; these errors encompass such factors as sample preservation, contamination, loss, sieving, etc. Both cycles and trends relate to changes in the concentration of a constituent of interest with respect to time or space.

The fundamental error (FE) is often the main source of sampling error (Pitard, 1993). It cannot be eliminated, but it can be estimated prior to sampling. Based on estimates of FE, steps can be taken to minimize it and thus minimize the overall sampling error. Important factors in the FE include heterogeneity, particle size, and sample mass. If the population is very heterogeneous or the particle size is large, then more sample mass is required to minimize the FE. If the mass of the population (lot), M_L, is greater than ten times the mass of the sample, the FE can be estimated by the following equation:

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

where
FE^2 is a relative variance
Ms is the sample mass (g)
c is the mineralogical factor
l is the liberation factor
f is the shape factor
g is the granulometric factor
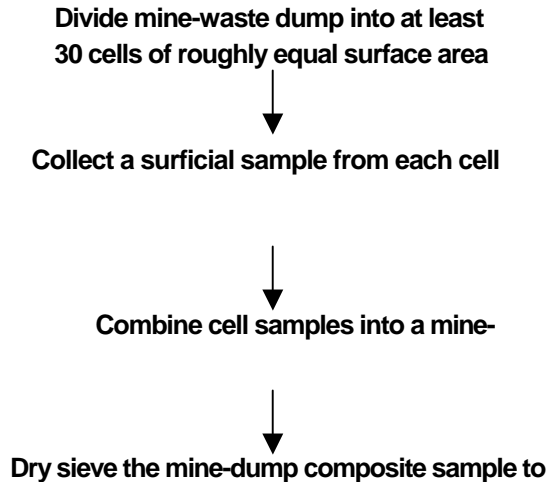d is the maximum particle size (cm)

The variables *"clfg"* are often referred to as the sampling constant, *C*. The mineralogical factor, *c,* is the maximum heterogeneity generated by the constituent of interest in the target population. It can be estimated by dividing the approximate density of the material (g/cm^3) by the average concentration of the constituent of interest (ppm), and assumes complete liberation of the constituent of interest. The liberation factor, /, is a correction factor for *c* that takes into account incomplete liberation of the constituent of interest. Values for *l* range from 0, when there is no liberation, to 1, when there is complete liberation. Values for the shape factor, *f*, vary from 0.2 for flakes to 10 for needles, with a value of 1 for a cube. A value of 0.5 is often used for *f* and represents a roughly spherical shape (Pitard, 1993). This shape corresponds with the majority of those observed in our < 2 mm size fraction. The granulometric factor, g, is a correction factor when all the particles are not the same size. Noncalibrated material has a value of 0.25, and calibrated material has a value of 0.55.

The other main sampling error of concern is the grouping and segregation error (GSE). To minimize the grouping factor of the GSE, it is necessary to collect as many small increments as practically possible, assuming that sample collection and preparation are properly carried out. An increment is a group of particles collected from a population with a single operation of the sampling device. Pitard (1993, p. 187) states that a sample should be made up of at least 30 increments. Minimizing the segregation factor of the GSE is much more difficult. Complete homogenization of the target population prior to sampling is the solution, but generally is impractical. An awareness of the mechanisms that lead to segregation (e.g., differences in particle density, particle size, particle shape, mine-dump construction, etc.) while sampling may lead to better sampling practices and strategies.

MINE-WASTE DUMP SAMPLING STRATEGY
To minimize sampling errors, our sampling strategy requires that a composite sample of mine-dump material must consist of at least 30 increments (subsamples), which reduces the GSE. We used the FE equation given in the Sampling Errors section to determine the total sample mass necessary to minimize the FE to an acceptable level. Inserting the 2 mm particle size into the FE equation and assuming that $c = 200$, $l = 0.8$ (for very heterogeneous material), $f = 0.5$, and g = 0.25, the FE can be kept below 2% if 1000 g or more of sieved sample are collected.

The following flow diagram lists the steps of our sampling strategy. Particles greater than about 1 cm are excluded from the initial collection procedure. The samples are collected with a stainless steel trowel, and each increment is successively placed in a plastic bucket upon collection. Air-dried samples are dry sieved to < 2 mm, and the > 2 mm fraction is discarded. Sieved samples are stored in a plastic bag or a paper soil-sampling bag, which aids in complete drying of the sample. If samples are collected wet, they should be air dried and efforts made to break up clods prior to sieving.

**Divide mine-waste dump into at least
30 cells of roughly equal surface area**

↓

Collect a surficial sample from each cell

↓

Combine cell samples into a mine-

↓

Dry sieve the mine-dump composite sample to

In our sampling strategy, we are interested in the average properties of a given mine-waste dump. In this case, collecting more sample mass is the same as collecting a larger number of samples in terms of error, but not in terms of cost. One 30-increment dump-composite sample collected using our sampling strategy contains as much information, relative to the average value, as 30 individual grab samples at 1/30 of the analytical cost.

PARTICLE-SIZE STUDY

To test our hypothesis that the smaller size fractions of mine-waste material from our historic weathered dumps are generally the most reactive and would control the water leaching behavior of the waste material, we conducted a series of leaching tests on different particle-size fractions of several mine-waste dumps. The dumps sampled for this study originated from mining of deposits of polymetallic veins in igneous rocks and polymetallic veins and replacements in carbonate-rich host rocks. Primary sulfides of chief interest in the mine wastes studied include pyrite, sphalerite, and galena, which produce secondary iron, zinc, and lead sulfate or carbonate minerals due to weathering reactions.

Composite mine-dump samples were collected using the strategy outlined in the previous section except that they were dry sieved into size fractions of > 9.5 mm, 5 to 9.5 mm, 2 to 5 mm, 1 to 2 mm, 0.75 to 1 mm, and < 0.75 mm. Each fraction was subjected to the Synthetic Precipitation Leaching Procedure (SPLP; U.S. EPA Method 1312; U.S. Environmental Protection Agency, 1994). The SPLP solution consists of slightly acidified deionized water (60/40 H_2SO_4/HNO_3 by weight) at a pH of 4.2. The procedure extracts the solid material at a 20:1 liquid-to-solid ratio (2,000 g extractant, 100 g solid) on an end-over-end rotary agitator for 18 hours. The slurry is then filtered through a 0.7 µm borosilicate glass fiber filter using a pressure filtration unit. Leachates were then acidified with nitric acid and analyzed by inductively coupled argon plasma - mass spectroscopy (ICP-MS) and inductively coupled argon plasma - atomic emission spectroscopy (ICP-AES).

Results of SPLP leaching of particle-size fractions of the mine-waste material are given in Figure 1 and Table 1. Also shown in figure l(open symbols) and listed in table 1 are SPLP leaching results from separate <2mm mine dump composites collected using the mine-waste dump sampling strategy discussed in 1 the previous section. The pH values of leachates in the various size fractions (fig. 1) remain fairly consistent for a given mine-waste dump. The pH values for the various size fractions compare well with the pH values of the < 2 mm composite, with the composite pH tracking the lowest pH in the most variable sample.

Chemical constituents, such as zinc, exhibit different leaching behavior in different size fractions (fig. 1, table 1). Table 2 lists the weight-fraction normalized leaching data for selected chemical constituents. This table shows the leachable contribution of a given particle-size fraction to the composite sample. With a
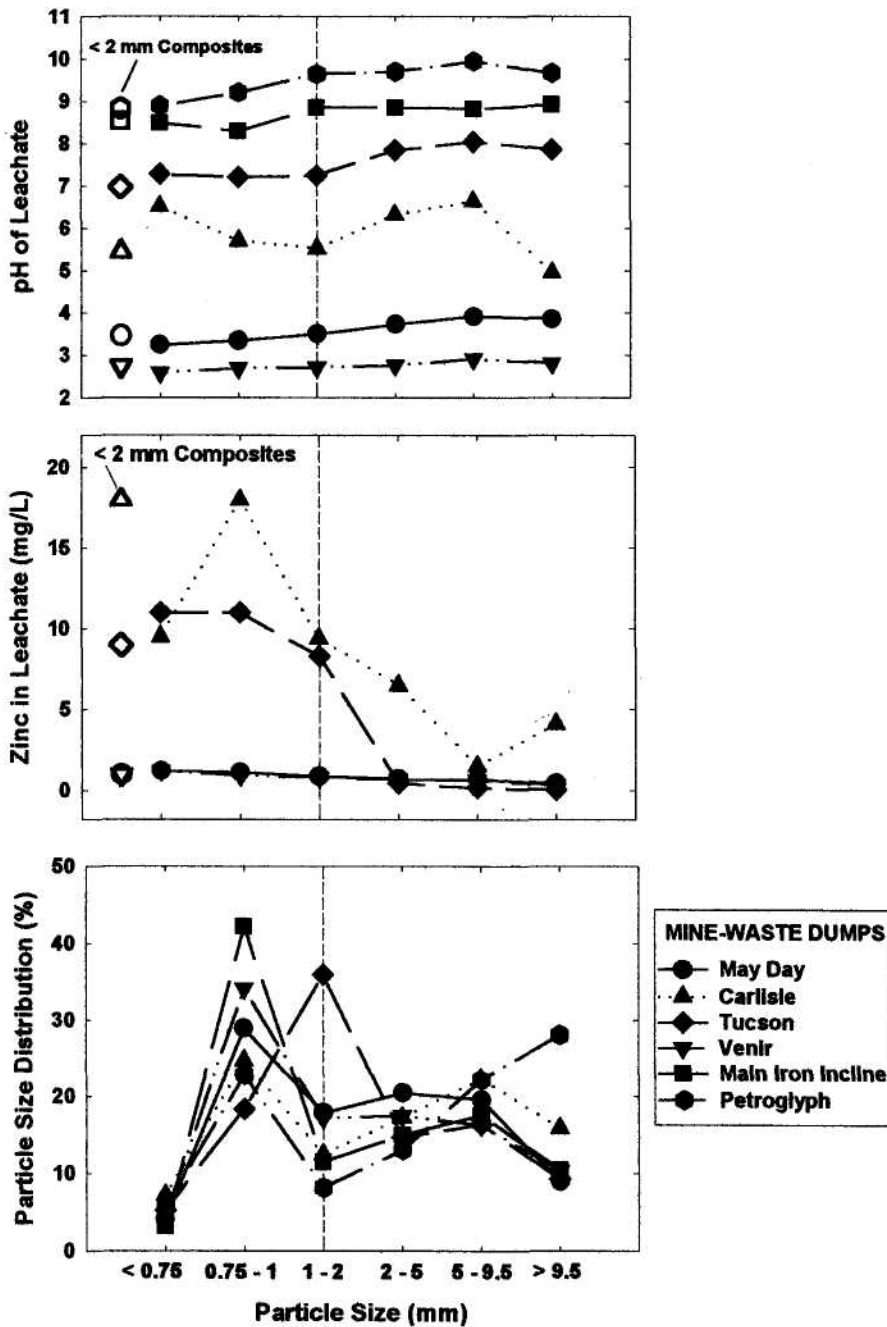
Figure 1 Particle-size distribution and results from the Synthetic Precipitation Leaching Procedure (SPLP) on different particle-size fractions of mine-waste material from several historic polymetallic dumps. Zinc values for Main Iron Incline and Petroglyph are < 1 and are not shown. The vertical dashed line denotes the < 2 mm size cutoff used in the mine-waste dump sampling strategy, and the open symbols represent pH and Zn values from SPLP tests of separate < 2 mm mine-dump composites collected using the mine-waste dump sampling strategy.

Table1 Leaching results from the Synthetic Precipitation Leaching Procedure (SPLP) on different particle-size fractions of mine-waste material from several historic polymetallic dumps, Bolded values are SPLP results from separate < 2 mm mine-dump composites collected using the mine-waste dump sampling strategy. Italics denote samples analyzed by ICP-MS and non-italics denote samples analyzed by ICP-AES.

Particle-Size Fractions	Cu (µg/L)	Pb (µg/L)	Ni (µg/L)	Cd (µg/L)	Zn (mg/L)	Fe (mg/L)	Al (mg/L)	Ca (mg/L)
Venir								
< 0.75 mm	4,800	*1,200*	38	*16*	1.2	**49**	17.8	7.8
0.75 -1 mm	4,000	*690*	28	*12*	**0.9**	33	13.2	5.1
1 - 2 mm	3,200	*940*	*22*	*10*	0.8	**25**	11.3	3.7
2 - 5 mm	2,700	*1,600*	*18*	*9.3*	0.67	**23**	9.71	3.2
5 - 9.5 mm	3,000	*1,400*	*16*	**8.9**	0.63	17	9.28	3.5
> 9.5 mm	1,000	*34*	*6.4*	*4.8*	0.31	**9.9**	**4.79**	0.58
< 2 mm Comp.	**3,900**	**759**	**43.8**	**13.3**	**0.97**	**27**	**14**	**6.2**
May Day								
< 0.75 mm	*202*	*6,900*	*16.0*	*7.8*	1.2	*1.4*	0.66	**2.6**
0.75 - 1 mm	*162*	*3,280*	*5.4*	*6.4*	1.1	*1.0*	0.47	**2.2**
1 - 2 mm	*112*	*2,840*	*3.8*	*5.3*	0.85	*0.87*	0.37	**1.6**
2 - 5 mm	*73.0*	*1,880*	*3.3*	*4.4*	0.7	*1.3*	0.21	**1.4**
5 - 9.5 mm	*39.4*	*4,820*	*2.2*	*3.7*	0.68	*2.3*	0.27	1.8
> 9.5 mm	*42.6*	*3,060*	*1.7*	*2.3*	**0.44**	*1.9*	0.22	**1.6**
< 2 mm Comp.	**133**	**2,750**	**5.6**	**6.1**	**0.98**	**0.72**	**0.43**	2.3
Carlisle								
< 0.75 mm	*54.3*	*86.2*	*22.4*	*489*	**9.5**	*<0.05*	*0.006*	170
0.75 -1 mm	*250*	*428*	*22.1*	*443*	18	*<0.05*	*0.15*	72
1 -2 mm	*312*	*969*	*10.8*	*195*	**9.4**	*<0.05*	*0.25*	17
2 - 5 mm	*32.7*	*8.2*	*9.7*	*325*	**6.5**	*<0.05*	*0.05*	18
5 - 9.5 mm	*6,1*	*2.6*	*4.7*	*69.9*	1.5	*<0.05*	*0.008*	17
> 9.5 mm	*283*	*374*	*4.4*	*54.7*	**4.1**	*0.10*	*0.12*	7.3
< 2 mm Comp.	**242**	**500**	**22.2**	**455**	**18**	*<0.05*	**0.16**	**75**
Tucson								
< 0.75 mm	*3.0*	*47.1*	*5.6*	*284*	11	*<0.05*	*0.002*	140
0.75 **-1 mm**	**2.6**	*58.0*	*7.7*	*350*	11	*<0.05*	*0.001*	160
1 -2 mm	**2.2**	*45.1*	*5.8*	**275**	8.3	*<0.05*	*0.003*	120
2-5 mm	*1.1*	*0.57*	*2.2*	*22.1*	0.41	*<0.05*	*0.003*	**62**
5-9.5 mm	*1.3*	*0.2*	*22*	*8.34*	0.15	*<0.05*	*0.002*	71
> 9.5 mm	*0.79*	*0.05*	*2.6*	*0.47*	0.01	*<0.05*	*0.002*	85
< 2 mm Comp.	**2.5**	**43.1**	**5.6**	**308**	**9**	*<0.05*	**0.003**	**133**
Main Iron Incl.								
< 0.75 mm	*0.65*	**15.6**	*0.9*	*1.17*	0.06	*<0.05*	*0.04*	**29**
0.75 -1 mm	*0.50*	*5.3*	*0.9*	*1.50*	0.04	*<0.05*	*0,03*	33
1 - 2 mm	*<0.5*	*1.8*	*0.4*	*0.06*	0.02	*<0.05*	*0.01*	17
2 - 5 mm	*<0.5*	*0.73*	*0.5*	*0.07*	0.007	*<0.05*	*0.009*	21
5 - 9.5 mm	*<0.5*	*0.2*	*0.8*	*0.09*	0.01	*<0.05*	*0.007*	32
> 9.5 mm	*<0.5*	*0.2*	*1.0*	*0.04*	0.008	*<0.05*	*0.006*	**34**
< 2 mm Comp.	**2.0**	**4.0**	**0.8**	**1.25**	**0.05**	**0.05**	**0.03**	31
Petroglyph								
< 0.75 mm	*7.2*	*107*	*0.7*	*0.61*	0.08	*0.11*	*0.17*	15
0.75 -1 mm	*5.8*	*88.1*	*0.7*	*0.38*	0.08	*0.09*	*0.17*	**14**
1 -2 mm	*2.9*	*3.3*	*0.4*	*0.16*	0.004	*<0.05*	*0.009*	13
2 -5 mm	*1.2*	*0.4*	*0.3*	*0.15*	0.002	*<0.05*	*0.008*	11
5 - 9.5 mm	*0.61*	*0.1*	*0.2*	*0.03*	0.005	*<0.05*	*0.003*	7.7
> 9.5 mm	*0.56*	*0.3*	*0.3*	*<0.02*	0.002	*<0.05*	*0.005*	**9.1**
< 2 mm Comp.	**3.7**	**1.7**	**0.5**	**0.11**	**0.004**	**<0.05**	**0.005**	12

Table 2 Normalized leaching results from the Synthetic Precipitation Leaching Procedure (SPLP) on different particle-size fractions of mine-waste material from several historic potymetallic dumps. Data have been normalized by multiplying their Ieachate values in Table 1 by their weight fraction in the dump-composite sample. Note the contribution of each particle-size fraction to the leachable concentration of a given chemical constituent

Particle-Size Fractions	Cu (µg/L)	Pb (µg/L)	Ni (µg/L)	Cd (µg/L)	Zn (mg/L)	Fe (mg/L)	Al (mg/L)	Ca (mg/L)
Venir								
< 0.75 mm	216	54	1.7	0.7	0.05	2.2	0.8	0.3
0.75 - 1 mm	1,364	235	9.5	4.1	0.31	11.3	4.5	1.7
1 -2 mm	550	162	3.8	1.7	0.14	4.3	1.9	0.6
2 -5 mm	472	280	3.1	1.6	0.12	4.0	1.7	0.6
5 - 9.5 mm	489	228	2.6	1.4	0.10	2.8	1.5	0.6
> 9.5 mm	104	3	0.7	0.5	0.03	1.0	0.5	0.1
May Day								
< 0.75 mm	8	283	0.7	0.3	0.05	0.06	0.03	0.11
0.75 -1 mm	47	948	1.6	1.8	0.32	0.29	0.14	0.64
1 -2 mm	20	508	0.7	0.9	0.15	0.16	0.07	0.29
2 -5 mm	15	385	0.7	0.9	0.14	0.27	0.04	0.29
5 -9.5 mm	8	945	0.4	0.7	0.13	0.45	0.05	0.35
> 9.5 mm	4	275	0.1	0.2	0.04	0.17	0.02	0.14
Carlisle								
< 0.75 mm	4	6	1.6	35	0.68	<0.05	0.0004	12.2
0.75 - 1 mm	62	106	5.5	109	4.45	<0.05	0.037	17.8
1 -2 mm	39	121	1.3	24	1.17	<0.05	0.031	2.1
2 -5 mm	6	1.4	1.7	56	1.12	<0.05	0.009	3.1
5 - 9.5 mm	1	0.6	1.0	16	0.33	<0.05	0.002	3.8
> 9.5 mm	45	59	0.7	9	0.65	0.02	0.019	1.2
Tucson								
< 0.75 mm	0.16	2.5	0.30	15	0.58	<0.05	0.0001	7
0.75 -1 mm	0.48	10.7	1.42	64	2.02	<0.05	0.0002	29
1 -2 mm	0.79	16.2	2.08	99	2.98	<0.05	0.0011	43
2 -5 mm	0.16	0.08	0.32	3.2	0.06	<0.05	0.0004	9
5 -9.5 mm	0.21	0.03	0.36	1.4	0.02	<0.05	0.0003	12
> 9.5 mm	0.07	0.005	0.24	0.04	0,0009	<0.05	0.0002	8
Main Iron Incl.								
< 0.75 mm	0.02	0.50	0.03	0.04	0.002	<0.05	0.001	0.9
0.75 -1 mm	0.21	2.24	0.38	0.63	0.017	<0.05	0.013	13.9
1 -2 mm	<0.5	0.21	0.05	0.01	0.002	<0.05	0.001	1.9
2 -5 mm	<0.5	0.11	0.07	0.01	0.001	<0.05	0.001	3.1
5 - 9.5 mm	<0.5	0.03	0.14	0.02	0.002	<0.05	0.001	5.6
> 9.5 mm	<0.5	0.02	0.10	0.004	0.001	<0.05	0.001	3.6
Petroglyph								
< 0.75 mm	0.4	6	0.04	0.03	0.005	0.006	0.010	0.9
0.75 -1 mm	1.3	20	0.16	0.09	0.018	0.020	0.039	3.2
1 -2 mm	0.2	0.3	0.03	0.01	0.0003	<0.05	0.0007	1.1
2 -5 mm	0.2	0.05	0.04	0.02	0.0003	<0.05	0.001	1.4
5 - 9.5 mm	0.1	0.02	0.04	0.01	0.001	<0.05	0.0007	1.7
> 9.5 mm	0.2	0.08	0.08	<0.02	0.0006	<0.05	0.001	2.6

few exceptions, particle-size fractions < 2 mm contribute the highest concentrations of leachable chemical constituents. When the normalized teachable concentrations of the < 2 mm particle-size fractions are summed, there are only three cases in which the < 2 mm fractions contribute less than 50% of the total normalized leachable concentrations. The three cases include lead at the Venir mine-waste dump, iron at the May Day mine-waste dump, and calcium at the Petroglyph mine-waste dump.

In general, higher concentrations of chemical constituents are leached from the smaller size fractions. This finding supports our hypothesis that smaller size fractions are generally more chemically reactive than larger size fractions in our dumps. Our choice of the < 2 mm size fraction may tend to slightly overestimate the teachability of the mine-waste material as a whole, but mis size-fraction cutoff does not appear to "miss" any leachable phases. For screening and prioritizing mine-waste dumps on abandoned mine lands, use of this size fraction generally should provide a worst-case scenario for the dumps sampled. For our application, the < 2 mm size fraction appears to be a good choice to reduce the sampling error and make the sample mass a reasonable size for field collection.

SUMMARY

We sought to develop a method to collect material from historic weathered mine-waste dumps to aid in screening and prioritization of a large number of dumps on abandoned mine lands. We present a cost-effective, statistically based sampling strategy for collecting composite surficial samples from mine-waste dumps to obtain average properties of the dumps. This strategy minimizes sampling error while providing a sample of the average properties of a given mine-waste dump.

ACKNOWLEDGMENTS

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