

# A CONCISE METHOD FOR MINE SOILS ANALYSIS<sup>1</sup>

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**Abstract.** A large number of abandoned hard rock mines exist in Colorado and other mountain west states, many on public property. Public pressure and resulting policy changes have become a driving force in the reclamation of these sites. Two of the key reclamation issues for these sites is the occurrence of acid forming materials (AFMs) in mine soils, and acid mine drainage (AMD) issuing from mine adits. An AMD treatment system design project for the Forest Queen mine in Colorado's San Juan mountains raised the need for a simple, useable method for analysis of mine land soils, both for suitability as a construction material, and to determine the AFM content and potential for acid release. We have developed a simple, stepwise, go – no go test for the analysis of mine soils. Samples were collected from a variety of sites in the Silverton, CO area, and subjected to three tiers of tests including: paste pH, Eh, and 10% HCl fizz test; then total digestion in HNO<sub>3</sub>/HCl, neutralization potential, exposure to meteoric water, and toxicity content leaching procedure (TCLP). All elemental analyses were performed with an inductively-coupled plasma (ICP) spectrometer. Elimination of samples via the first two testing tiers left two remaining samples, which were subsequently subjected to column and sequential batch tests, with further elemental analysis by ICP. Based on these tests, one sample was chosen for suitability as a construction material for the Forest Queen treatment system basins. Further simplification, and testing on two pairs of independent soil samples, has resulted in a final analytical method suitable for general use.

Additional key words: mine land reclamation, soil testing, acid forming materials, acid mine drainage.

## Introduction

Since the beginning of mineral exploration and development in the Rocky Mountain region, acid mine drainage (AMD) has been a constant and serious source of pollution in the surface and ground waters. In some areas, heavy metals mobilized by waters of very low pH have rendered significant reaches of many streams effectively devoid of life. Public awareness, political pressure, and greater scientific insight have resulted in recent regulatory changes aimed at fixing the problem. However, the fact that many abandoned mine claims are located on public lands means that the brunt of the reclamation burden has fallen on only a few management agencies with limited operating budgets, and progress has been slow. In an effort to reduce the cost and labor-intensity of smaller scale efforts, attention has recently focused on the use of materials native to the

mine site region for construction of treatment system channels and impoundments. However, in some cases native materials are heavily burdened with sulfides or other acid forming materials (AFM), as well as heavy metals, and would only exacerbate the original AMD problem, making them unsuitable for use. During a recent reclamation project on the Forest Queen mine site in the San Juan Mountains of southwestern Colorado, we encountered this need to analyze local materials for suitability. A literature search, however, resulted in no easy, concise method to evaluate mine soils. By combining several analytical methods into a stepwise process, we were able to devise a clearly defined go-no go process for soil testing, which makes it possible to determine both an estimate of the AFM content of a soil, and its fitness for use as a construction material for an in situ AMD treatment system.

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## Materials and Methods

### Soils

The soils originally used in the development of the testing method were taken from the Silverton caldera region of the San Juan Mountains, and more specifically from various sites in mineralized areas of Placer Gulch, Maggie Gulch, and Cunningham Gulch. Samples from seventeen (17) separate sites were screened to minus 10 mesh in the field, then sealed into labeled resealable one-gallon plastic freezer bags. All attempts were made to make the sample representative of the waste rock pile as possible. Once in the laboratory, the samples were air dried, divided, and screened to minus 80 mesh as necessary.

Figure 1. Study location

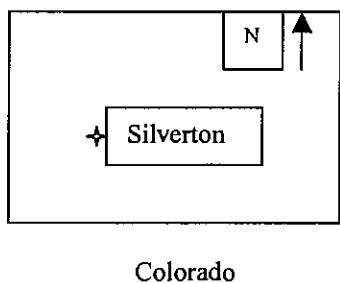


Figure 2. Silverton Area (Not to scale)

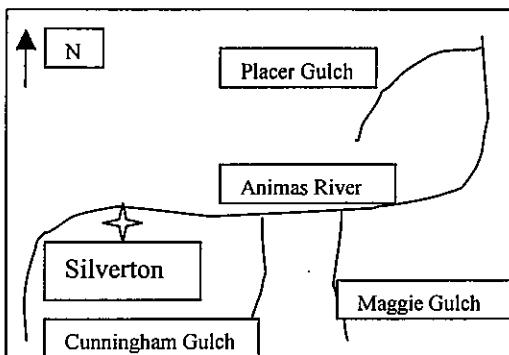


Table 1. Soil Samples by Site Location

Sample No.	Location
1	SE side of Placer Gulch (high)
2	SE side of Placer Gulch (middle)
3	SE side of Placer Gulch (low)
4	Sunbank Mine tailings, NW PG
5	Talus 1 mi above Eureka Bridge
6	Talus E of Eureka Bridge
7	Stony Pass tunnel tailings (high)
8	Talus at beginning of Stony Pass Rd.
9	Old 100 Mine tailings pile
10	Talus NE of Maggie Gulch Rd.
11	Maggie Mine tailings
12	Talus 3.3 mi up Animas from Silverton
13	Talus N of Animas 1 mi above CG fork
14	Pride of the West Mine tailings
15	Highland Mary Mine coarse tailings
16	Highland Mary Mine fine tailings
17	Green Mountain Mine tailings

### Test Method

The test method was designed with ease of use in mind, while incorporating all the crucial aspects of analysis. In the interest of being able to eliminate grossly unsuitable materials early on in the process, without having to perform the entire test battery on them, a go-no go process consisting of three test tiers was used. Soils which failed to pass the standards of the first or second tier were eliminated from further consideration.

**Test Tier 1.** The first tier of tests consisted of two simple tests: paste pH, and 10% HCl fizz (EPA Field and Lab Methods 3.2.3).

To accomplish the paste pH test, 25 g of minus 10-mesh soil sample were placed in a clean beaker, and saturated with 50 ml of nanopure water. The soil/water mixture was agitated, and measurements were taken at 0.5 hr and again at 24 hrs. Half-hour pH measurements for the seventeen samples ranged from 3.49 to 7.32. Twenty-four-hour measurements were between 3.63 and 7.10. Eh readings were taken concurrently with the 24 hr pH readings, in order to obtain some idea of the oxidizing or reducing characteristics of the soil/water mixture.

The 10% HCl fizz tests were accomplished using approximately 0.5 g of minus 10-mesh soil sample, placed in a watch glass, and then saturated with several drops of a 10% HCl solution. The solution was then

watched for the appearance of gas bubbles (indicating reaction between the hydrochloric acid and carbonate materials in the soil resulting in the evolution of CO<sub>2</sub> gas). The presence of bubbles was graded as a positive result, while an absence of bubbles was graded as a negative result.

Based on the combination of pH results and fizz test scoring, five samples were selected from the original seventeen to move into the next phase of testing. All five showed positive fizz test results, and had pH readings over 6.00. Samples showing either a negative fizz result, or a pH less than 6.00 were eliminated from consideration due to AFM content or lack of alkalinity, or both.

Test Tier 2. The second phase of testing was a series of four additional analyses, including: an HNO<sub>3</sub>/HCl aggressive digestion, a neutralization potential test (EPA Field and Lab Methods 3.2.3), a meteoric water exposure test (Nevada Department of Environmental Protection), and a modified 20:1 toxicity content leaching procedure (TCLP) test (EPA method 1311).

To perform the aggressive digestion, 0.2 g of minus 80-mesh soil were measured into a Teflon beaker. Five ml of concentrated HNO<sub>3</sub> were added, and then 5 ml of concentrated HCl. The beaker was then heated to 100° C on a hotplate in an appropriate fume hood for three hours, or until all liquid was evaporated from the beaker. Next, five more ml of HNO<sub>3</sub> were added and the residue redissolved. The beaker was then covered with a Teflon watchglass and returned to the hotplate at 40° C for 10-12 hours. Finally, the hotplate temperature was increased to 90° C for four hours. The resulting solution was then diluted for ICP elemental analysis.

A neutralization potential test was also performed during this series of analyses. To accomplish this, the soil was sieved to minus 80 mesh, and then 2 g were weighed into a beaker. Forty ml of 0.1M HCl solution were then added. The mixture was allowed to settle, and then was back – titrated to pH 7.00 using a 0.2M NaOH solution, to determine the carbonate content of the soil.

Meteoric water exposure testing was done to determine the potential for metals release from the soils when exposed to natural waters. To accomplish this, 6 g of minus 10 mesh soil were weighed into a beaker, and then mixed with 20 ml of nanopure water which had been brought to pH 5.00 by HNO<sub>3</sub>. The mixture was allowed

to sit for 24 hours, and then a water sample was drawn and filtered for elemental analysis by ICP.

The last analysis of the second tier was the modified TCLP test. The procedure consisted of weighing 2 g of minus 10 mesh soil sample into a beaker, and then mixing it with 20 ml of a 1M HOAc solution made pH 4.50 by NaOH. As with the meteoric water test, the mixture was allowed to sit for 24 hours, and then a water sample was drawn for ICP elemental analysis for Ag, Al, As, Cd, Cu, Fe, Pb, Se, and Zn.

Test Tier 3. Based on the results of the second battery of tests, two soils were advanced to the third and last tier. This consisted of two further analyses: a column test, and a sequential batch test.

The column test was performed using either a 250 ml or 500 ml acid-washed burette. A glass wool plug was first inserted into the burette. Next, a layer of approximately 50 ml of pure silica sand was added. Using a hand vacuum pump, nanopure water was then drawn up into the glass wool and sand, a volume just sufficient to completely saturate these two layers. Next, a 100 ml volume of uncompacted minus 10 mesh soil was added to the top of the column. Nanopure water was then used to completely saturate the soil, and the volume required was recorded as the pore volume of the soil sample. Testing then commenced using a local source of AMD (the McClelland Tunnel, near Dumont, CO), which had already been analyzed by ICP for elemental content. Measured pore volumes were added one at a time to the top of the column, and were allowed to drain either to be collected as samples, or as waste water when taking samples incrementally. The samples were then acidified and analyzed by ICP for elemental content. Sixty pore volumes of AMD were passed through the original two test samples. To complete the final testing a 20 mg/L Zn / 200 mg/L S solution was passed through each column to test for ZnCO<sub>3</sub> precipitation. Pore volume one of this solution was collected for ICP analysis, and then pore volumes 3 through 5 were collected and analyzed for alkalinity and pH. Pore volumes 6 through 8 were adjusted to pH 4.00 before being run into the column, and then were collected and analyzed for alkalinity and pH.

The sequential batch testing was carried out using 500 ml polyethylene bottles. To begin the procedure, 250 g of minus 10 mesh soil sample and 250 ml of AMD water were

combined in a polyethylene bottle, sealed, and agitated using electric motor-driven rollers for 24 hours. Once the mixing was complete, the water was separated from the soil sample using a 0.45 µm filter. At this point, a water sample was taken, and then the remaining water, and all of the soil, were split into two tracks.

On the "water track", 125 ml of water from the original bottle were combined with 125 g of fresh (dry) soil sample, in a polyethylene bottle. The bottle was sealed and agitated for 24 hours, and then the water was separated using a 0.45µm filter. A water sample was taken, and the soil discarded. Finally, 62.5 ml of this second fraction of water were combined with 62.5 g of fresh (dry) soil, sealed in a polyethylene bottle, and agitated for 24 hours. The water was then filtered to 0.45 µm, and a

sample was taken. The remaining water and soil were then discarded.

On the "soil track", all the original soil was returned to the polyethylene bottle, and was combined with a second 250 ml aliquot of AMD. The mixture was then agitated for 24 hours, the water was filtered to 0.45 µm, a sample was taken, and then the remaining water was discarded. Once again, all of the soil was returned to the bottle, and a third 250 ml aliquot of AMD was added. After a 24 hour period of agitation, the water was once again filtered to 0.45 µm, a final sample was taken, and the remaining water and soil were discarded.

All water samples from the sequential batch procedure were subsequently analyzed for elemental content by ICP.

Figure 3. Sequential Batch Test Flowchart

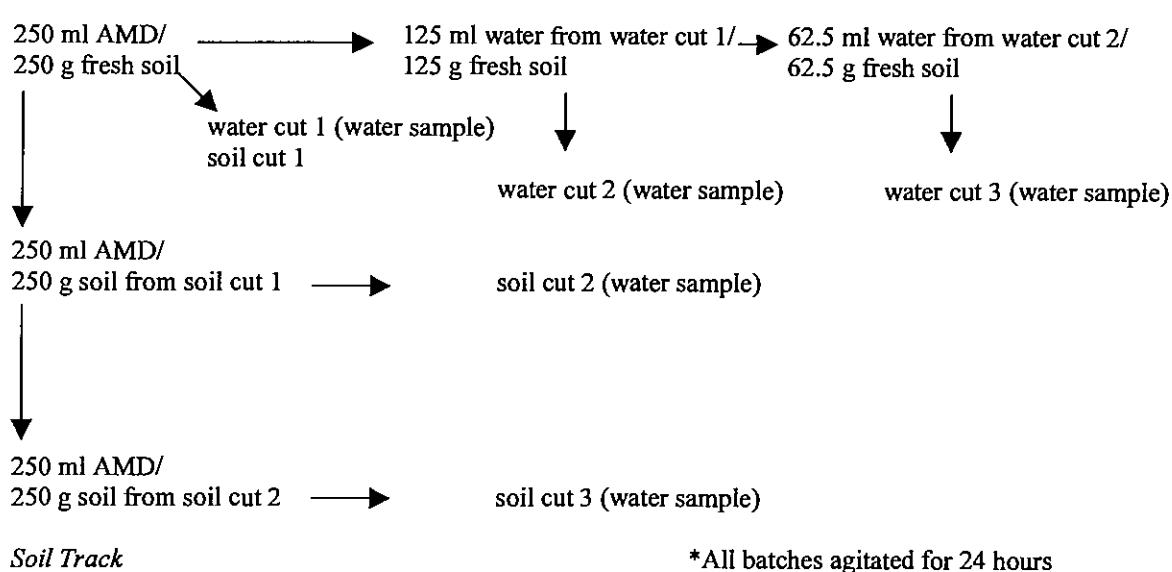
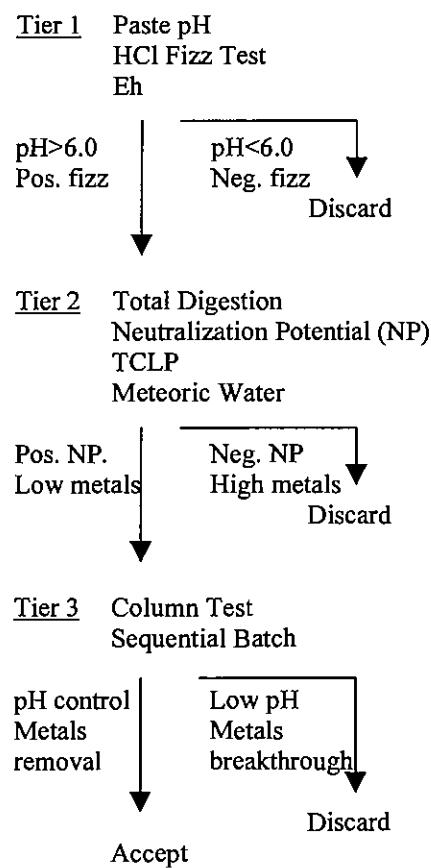


Figure 4. Overall Test Method Flowchart



### Verification

In order to verify the usefulness of the method, the procedure was used to analyze two pairs of independent samples, also collected in the Silverton, CO, area, but collected by second parties.

### Results and Discussion

Results of the soil testing were varied, due to differences in the materials. However, useful patterns of data did emerge from the testing process.

### Test Tier 1

The first battery of tests was conceived as a quick, inexpensive, and easy way to eliminate some materials from further consideration. Soils with high levels of AFMs will show up quickly by giving low paste pH numbers. Materials which have no ability to

buffer acidic waters will give a negative fizz test result. By eliminating these poor soils early in the process, further cost and the logistical problems associated with handling large numbers of samples may be avoided. As previously discussed, paste pH testing of the original group of 17 samples yielded readings between 6.49 and 7.32 over 0.5 hrs, and between 3.63 and 7.10 over 24 hrs. As the soils are likely to be exposed for very long periods of time to AMD in a treatment system, more weight was given to the 24-hour results.

Eh measurements performed alongside the 24-hr pH measurements gave readings ranging from 403.1 mV to 510.0 mV, referenced to H<sub>2</sub>. The five samples chosen to advance to tier two demonstrated Eh readings averaging less than 450 mV, indicating less oxidizing conditions in agreement with their generally higher pH results.

The 10% HCl fizz test results were also varied, with only seven of the original 17 samples showing positive results.

All five of the samples picked to advance to the second round of tests demonstrated a positive fizz test result, and gave paste pH readings of greater than 6.00, indicating minimal amounts of AFMs and the presence of at least some alkalinity.

### Test Tier 2

Test tier two was developed for the purpose of determining the content of the soil materials, and how those materials would react to weathering and exposure to acidic waters. Obviously, materials which contain AFMs and/or high levels of metals would be undesirable, while those which possessed low metals concentration and carbonates or other forms of alkalinity would be very useful as treatment system construction materials. All results are shown in Table 2.

One of the more enlightening aspects of this testing regime was the HNO<sub>3</sub>/HCl aggressive digestion of the samples. This procedure allowed a detailed analysis of exactly what elements were present in each of the soils.

The neutralization potential test was also very useful, as it provided some idea of just how much alkalinity was present in each soil. Of the five "second tier" soils, neutralization ranged from  $1.10 \times 10^{-3}$  mol acid neutralized/g soil, to  $9.84 \times 10^{-3}$  mol acid neutralized/g soil, with one sample showing acid-forming characteristics rather than neutralization potential.

Table 2. Test Tier 2 Analyses Results (in mg/L)

Meteoric Water:

<u>Element</u>	<u>3</u>	<u>7</u>	<u>14</u>	<u>15</u>	<u>16</u>	<u>DL</u>	<u>Animas R. Stds.</u>	<u>HazWaste Stds.</u>
Ag	BDL	BDL	BDL	BDL	BDL	0.005	0.000065	-
Al	0.100	0.148	0.039	1.045	6.281	0.015	0.087	-
As	BDL	BDL	BDL	BDL	BDL	0.02	0.100	5.0
Cd	0.001	BDL	0.095	0.003	0.005	0.001	0.002	0.5
Cu	0.016	BDL	0.007	0.016	0.032	0.001	0.011	-
Fe	0.088	0.051	0.023	0.473	1.660	0.005	0.132	-
Pb	BDL	BDL	0.289	0.080	0.066	0.005	0.003	5.0
Se	BDL	BDL	BDL	BDL	BDL	0.03	0.017	5.0
Zn	0.004	0.002	4.188	0.033	0.071	0.001	0.540	-

TCLP:

<u>Element</u>	<u>3</u>	<u>7</u>	<u>14</u>	<u>15</u>	<u>16</u>	<u>DL</u>	<u>Animas R. Stds.</u>	<u>HazWaste Stds.</u>
Ag	0.022	0.009	0.016	0.051	0.014	0.005	0.000065	-
Al	3.899	4.503	11.073	5.366	3.191	0.015	0.087	-
As	BDL	BDL	BDL	BDL	BDL	0.02	0.100	5.0
Cd	0.010	0.012	0.677	0.240	0.148	0.001	0.002	0.5
Cu	1.313	0.724	15.303	13.527	9.983	0.001	0.011	-
Fe	2.009	18.690	10.781	26.691	8.407	0.005	0.132	-
Pb	0.115	0.151	1054.8	268.07	25.164	0.005	0.003	5.0
Se	0.052	BDL	0.049	BDL	0.051	0.03	0.017	5.0
Zn	0.371	1.095	159.73	23.628	16.429	0.001	0.540	-

HCl/HNO<sub>3</sub> Digestion:

<u>Element</u>	<u>3</u>	<u>7</u>	<u>14</u>	<u>15</u>	<u>16</u>	<u>DL</u>	<u>Animas R. Stds.</u>	<u>HazWaste Stds.</u>
Ag	BDL	2.835	6.249	2.740	15.853	0.005	0.000065	-
Al	6782.2	9095.3	7034.8	10252	2674.9	0.015	0.087	-
As	BDL	33.418	31.137	18.054	43.782	0.02	0.100	5.0
Cd	1.838	4.236	38.073	8.404	3.977	0.001	0.002	0.5
Cu	210.18	110.04	1969.8	707.03	267.51	0.001	0.011	-
Fe	19849	49534	44363	32022	12228	0.005	0.132	-
Pb	BDL	BDL	BDL	BDL	BDL	0.005	0.003	5.0
Se	BDL	BDL	BDL	BDL	BDL	0.03	0.017	5.0
Zn	88.166	189.01	7664.0	809.84	430.32	0.001	0.540	-

Neutralization Potential:

<u>Sample</u>	<u>Acid Neutralized (mol acid/g soil)</u>
3	5.37x10 <sup>-3</sup>
7	1.69x10 <sup>-3</sup>
14	1.07x10 <sup>-3</sup>
15	1.97x10 <sup>-3</sup>
16	2.19x10 <sup>-4</sup>

Finally, the meteoric water and TCLP tests provided some insight into the likely behavior of the mine soils when exposed to weathering and acidic waters. All samples showed elevated levels of Ca, K, and Si.

Two samples were picked to advance to the third battery of tests based on their elemental content, by their acid neutralizing characteristics (and lack of acid forming tendencies), and the fact that they do not release significant levels of metals into meteoric or acidic waters.

#### Test Tier 3

The purpose of the third tier tests was to directly evaluate the reactions of soils to AMD exposure, and to determine their true ability to handle many pore volumes of acidic, metal-bearing waters.

In the course of the column test, sixty pore volumes of AMD were run through each of the two final soil samples. The elemental analysis of the AMD was determined prior to running the columns, and particular attention was paid to levels of Cu, Fe, Mn, and Zn in the column effluent. As can be seen in Tables 3 and 4, levels of all the metals were drastically reduced by passing through the columns, and the ability of the soils to remove metals from the water was apparently not diminished, even after passage of sixty pore volumes. Alkalinity and pH readings were also taken at the close of AMD testing, and resulted in readings of 64 and 48 mg/L CaCO<sub>3</sub>, and 8.63 and 8.81 pH for the two columns, in contrast to the 4.2 mg/L CaCO<sub>3</sub> of alkalinity and pH 4.72 of the AMD before treatment. Clearly, sixty pore volumes of AMD did not diminish the soils' ability to "treat" the water by adding alkalinity and removing metal.

To verify that the results of the column testing were not erroneous, and that the soils actually were removing metals, further analysis was done on both the AMD and the soils. Samples of the AMD were acidified before and after filtration and analyzed by ICP to verify that metals levels were the same using either method. Also, a standard solution of 20 mg/L Zn and 200 mg/L S were run through the columns, and showed that the soils were in fact removing Zn, probably by precipitation as ZnCO<sub>3</sub>.

The sequential batch test run on the soils provided a second method for evaluating the ability of the soil samples to handle exposure to, and possibly treat, the AMD. Results were similar to those of the column tests, and showed that the soils were more than capable of raising

the pH of the AMD waters, and precipitating metals.

Table 3. Column Test, Stony Pass Soil (7)

	Pore Volumes			
	AMD	15	35	60
pH	4.72	-	-	8.63
Cu (mg/L)	1.845	BDL	BDL	BDL
Fe (mg/L)	1.781	BDL	BDL	BDL
Mn (mg/L)	26.114	0.761	3.798	1.376
Zn (mg/L)	10.519	BDL	0.037	0.003

Table 4. Column Test, Highland Mary Soil (15)

	Pore Volumes			
	AMD	15	35	60
pH	4.72	-	-	8.81
Cu (mg/L)	1.845	BDL	BDL	0.155
Fe (mg/L)	1.781	BDL	0.008	BDL
Mn (mg/L)	26.114	0.071	0.657	BDL
Zn (mg/L)	10.519	0.014	0.033	0.003

#### Verification of Method

To evaluate the usefulness of this testing method, two pair of independently collected samples were obtained from abandoned mine sites in the Silverton area: two from the Kitty Mack mine, and two from the Little Maude mine.

Preparation of the soils was identical to the original seventeen samples – upon receiving them in the lab they were air dried, divided and sieved to minus 10 mesh (minus 80 as appropriate), and stored in labeled recloseable freezer bags.

**Kitty Mack Soils.** The Kitty Mack soils were received and tested in late summer 1998, and failed to pass the first tier of tests. Paste pH testing performed on the samples (labeled KM05 and KM06) gave low readings (circa 3.50), both after 0.5 hr, and 24 hrs. Eh readings were all above 700 mV referenced to H<sub>2</sub>, verifying the fact that the soil/water mixtures had strongly oxidizing properties. Fizz tests on both materials were negative, indicating the absence of alkalinity in the soils.

The quick and inexpensive manner in which these soils could be eliminated from further consideration (and identified as a water quality threat) demonstrates the efficiency of the "tiered" testing method. No further analysis was

necessary to reveal the presence of AFMs, or to discover the soils' unsuitability as treatment system construction materials.

**Little Maude Soils.** Soils from the Little Maude mine site were received in late fall 1998, and analyzed early in 1999. Two samples were evaluated, labeled Little Maude North (LMN), and Little Maude South (LMS).

First tier testing results were quite promising. LMN gave readings of 7.66 for 0.5 hr pH, 6.92 for 24-hr pH and 652.4 mV for 24 hr Eh. LMS showed readings of 7.78, 5.22, and 663.2, respectively for the same analyses. HCl fizz test results were positive for LMN, and very strongly positive for LMS, completing the qualifications of both samples to advance to the second battery of tests.

Test tier two results are shown in Table 5, and are compared with the Hazardous Waste

Standards, and with USEPA Standards for metals in the upper Animas River. Neutralization potential tests showed  $4.5 \times 10^{-3}$  mol acid neutralized/g soil for LMN, and  $8.7 \times 10^{-3}$  mol acid neutralized/g soil for LMS.

The third tier tests for the Little Maude samples were conducted with a synthetic AMD solution consisting of 10 mg/L each of Cu, Mn, and Zn in nanopure water, buffered to pH 5.0 using NaOH. This method was used to eliminate variables in native AMD such as microbial effects and precipitation. Column testing on the soils was terminated at 25 pore volumes due to time constraints, but no breakthrough of Cu, Mn, or Zn was seen to that point. Sequential batch testing on the samples using the same synthetic AMD solution also showed no breakthrough of any of the three metals.

Table 5. Little Maude ICP Analyses (mg/L)

Element	LMN Digest.	LMS Digest.	LMN TCLP	LMS TCLP	HazWaste Stds.
Ag	6.223	4.290	0.012	0.011	-
Al	3409	2278	0.162	0.017	-
As	95.08	63.79	BDL	BDL	5.0
Cd	44.11	36.35	0.024	0.006	0.5
Cu	241.3	136.3	0.157	0.089	-
Fe	12311	9127	0.051	0.050	-
Pb	307.12	172.7	BDL	BDL	5.0
Se	55.00	50.32	BDL	BDL	5.0
Zn	375.8	291.1	1.273	0.208	-

Element	LMN MW	LMS MW	Animas Stds.
Ag	BDL	BDL	0.00065
Al	0.023	BDL	0.087
As	BDL	BDL	0.100
Cd	0.003	BDL	0.002
Cu	0.091	0.197	0.011
Fe	0.042	0.113	0.132
Pb	BDL	BDL	0.003
Se	BDL	BDL	0.017
Zn	0.105	0.046	0.540

### Conclusions

The criteria of this study were established to investigate native material which could possibly be used in the construction of passive AMD systems. With some revision to the criteria of the first two tiers, the test could easily be used to discover materials that are the worst contributors to water quality degradation, thereby eliminating (or identifying) the worst offenders from further consideration (or singling them out for remediation efforts).

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