A PHYSICAL AND CHEMICAL ASSESSMENT OF THE MAUDE MONROE MINE SITE[1]

V. S. Franciscus[2], J. F. Ranville[3], T. R. Wildeman[3], and S. Frail[4]

Abstract: The Maude Monroe Mine in Clear Creek County Colorado is an orphaned site and is one of the earliest precious metal mines in Colorado. Because of close location of the site to Interstate 70, the Clear Creek Watershed Foundation (CCWF) desires to turn it into a museum site. As part of the field session course for the environmental engineering option at the Colorado School of Mines (CSM), 16 undergraduate students spent one week performing a physical and chemical assessment of the site. The basis for the assessment was the Mine Waste Decision Tree and the physical and chemical assessment tests that have been developed by CSM and the U.S. Geological Survey. The objectives for the session were to familiarize the students with the issues involved with metals in the aquatic environment and to introduce the students to sampling and assessment procedures that can be used during a site visit. The highlight of the week was the site visit. Students were divided into four groups and tasked with performing physical assessments of important mine waste piles, and using the composite-sampling method for sampling those piles. This focused the activities of the students while at the site and provided ample material to use for analytical activities. Different groups were given assignments of: metals analysis, data management, aquatic toxicity, and human toxicity. Finally, each group made a presentation based on their assignment which provided the CCWF useful information to determine what should be done with the site in the future.

Additional Key Words: Mine wastes, metals contamination, site assessment

---

[1] Paper was presented at the 2008 National Meeting of the American Society of Mining and Reclamation, Richmond, VA, ***New Opportunities to Apply Our Science*** June 14-19, 2008. R.I. Barnhisel (Ed.) Published by ASMR, 3134 Montavesta Rd., Lexington, KY 40502

[2] V. Sofia Franciscus, Division of Environmental Science and Engineering, Colorado School of Mines, Golden, CO, 80401, e-mail: sofiafranciscus@yahoo.com [3]Thomas Wildeman and James Ranville, are Professors of Chemistry and Geochemisty, Colorado School of Mines, Golden, CO, 80401, [4]Shelby Frail, Clear Creek Watershed Foundation, Idaho Springs, CO.

Proceedings America Society of Mining and Reclamation, 2008 pp 1413-1428
DOI: 10.21000/JASMR08011413

`http://dx.doi.org/10.21000/JASMR08011413`

Introduction

Acid rock drainage (ARD) results from abandoned mine waste debris (Wildeman and Schmiermund, 2002). The natural rock weathering process is accelerated as mining operations (excavation, drilling and milling rock into fine tailings) unnaturally increase rock surface area thus increasing exposure to weathering. The acidification of the environment solubilizes heavy metals resulting in contamination of rivers and streams until remediation stabilizes the advanced erosion and leaching of the waste debris. Because of the toxicity it generates, ARD has the potential to render soils and aquatic environments uninhabitable for vegetation and other life, scarring the land with barren landscapes and discolored water bodies (Herron et al., 2001, Heflin et al., 2004). Furthermore, cementation crusts often form and are a reservoir for future terrestrial and aquatic toxicity problems.

There is a need to assess the severity of ARD toxicity to determine what method of remediation would most likely achieve site cleanup. Chosen remediation options differ depending on the intended land use, concerns being specifically addressed, and unique characteristics of the site. Site assessment prior to remediation is a daunting task, first because there are so many sites, and second, because of the myriad of factors to consider. This is a key reason why the Mine Waste Decision Tree (MWDT) was created (Wildeman, Smith, and Ranville, 2007). The MWDT and the tests and observations that accompany it were developed by research at the US Geological Survey (USGS) (Smith, et al., 200, Hageman and Briggs, 2000, Hageman et al., 2005) and the Colorado School of Mines (CSM) (Winkler et al. 1999, Herron et al, 2001, Wildeman, et al., 2003, Heflin et al., 2004) to provide a comprehensive structure for assessing whether questionable mine wastes present a physical and chemical danger to the environment. The purpose of this paper is to offer an example of how this all encompassing tool can be useful for the training of environmental engineers and others who are perhaps not specialized in the remediation of mine spoils. We hope to demonstrate that the simplicity of this approach and the tests involved allow the study and applied use of the MWDT to be easily implemented in the engineering curriculum.

Mine Waste Decision Tree

The MWDT employs simple physical and chemical tests to determine whether leachate flowing from mine-waste material poses a potential toxicity threat to the aquatic environment (Fig. 1)

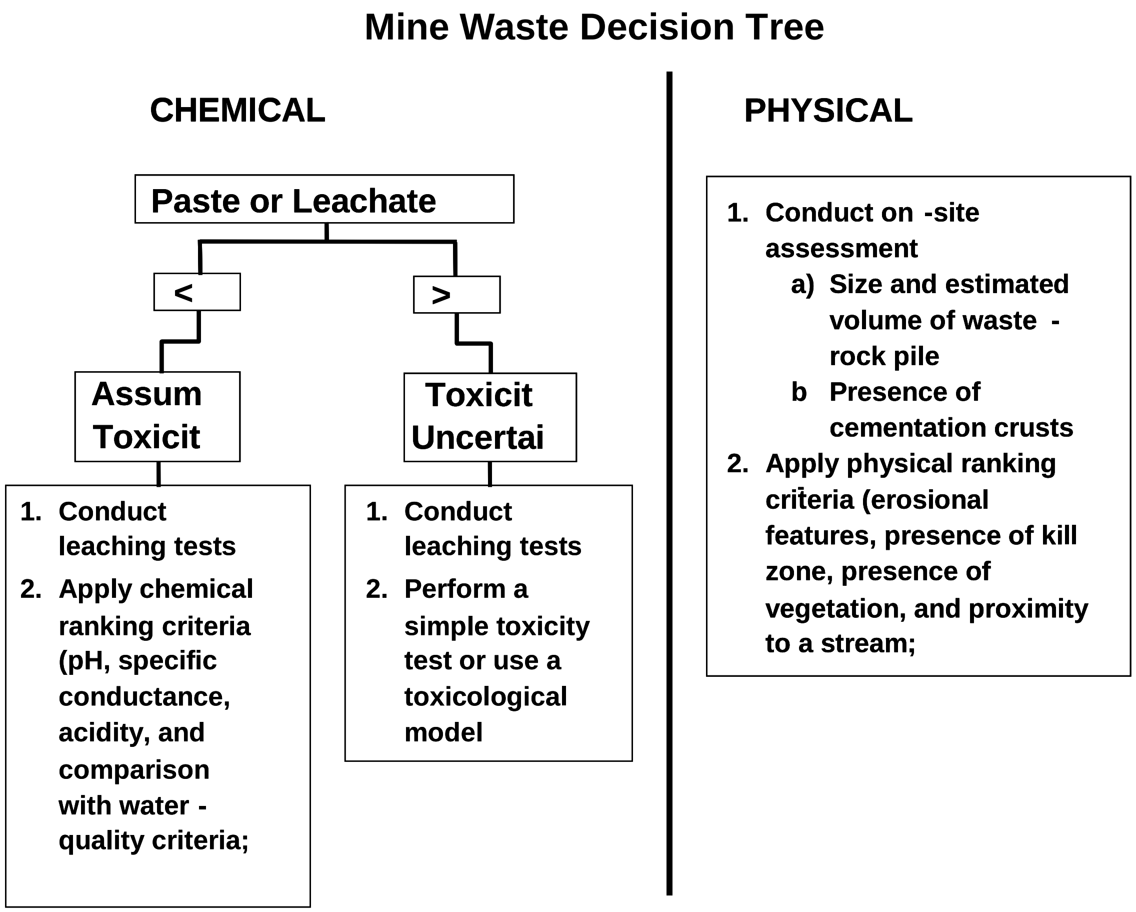

Figure 1. The Mine Waste Decision Tree.

For the chemical analysis portion of the tree, leaching tests developed by the U.S. Geological Survey (USGS) (Hageman and Briggs, 2000), the Colorado Division of Minerals and Geology (CDMG) (Herron, et al., 1999, 2001), and a modified 1311 TCLP test of the U.S. Environmental Protection Agency (EPA) (Tessier, et al., 1979, Wildeman, et al, 2003) have been extensively used as a surrogate for readily available metals that can be released into the environment from mining wastes. To assist in the assessment, element concentration pattern graphs (ECPG) are produced that compare concentrations of selected groups of elements from the three leachates and any water associated with the mining waste. Examples of how the leaching tests and the ECPG can be used in a site assessment are given in Bazin et al., 2002, Hageman et al., 2005, and Moehle et al. 2006. The MWDT makes a distinction between leachates or waters with pH less than or greater than 5. Generally, when the pH values are below 5, the ECPG of the solutions are quite similar, and potential aquatic toxicity from cationic metals, such as Pb, Cu, Zn, Cd, and Al, is assumed. Below pH 5, these metals are mostly dissolved, generally are not complexed with organic or inorganic ligands, and hence are more bioavailable. Furthermore, there is virtually no carbonate alkalinity at pH less than 5. All of these factors promote metal toxicity to aquatic organisms. On the other hand, when the pH value of the water or the leachates is above 5, the ECPGs from the solutions are variable, and inferred aquatic toxicity depends on factors in addition to the metals released from the leaching tests. Hence, leachates and waters with pH above 5 warrant further examination of their chemical composition. Physical ranking criteria provide additional information, particularly in areas where waste piles exhibit similar chemical rankings. Rankings from physical and chemical criteria generally are not correlated. In general, the chemical assessment rates the bioavailability of contaminants associated with a waste pile, and the physical assessment rates the ability of those contaminants to reach the watershed.

Field and Laboratory Methods

Field and Laboratory Work Overview

This site assessment was performed as part of a field study course, by an undergraduate class as a one week immersion topic on acid mine drainage and preliminary site assessment. The class was broken into four individual groups of five. All four groups performed chemical and physical assessments utilizing the MWDT assessment criteria which include a soil leachate test to measure pH, acidity, and specific conductance of leachate from a particular waste pile. Group 1 had the extra assignment of operating and organizing the results from all of field and laboratory tests. Groups 2 and 3 additionally performed bioassays, one using waste rock leachate with *E.coli* as the target species, while Group 3 simulated human absorption information via gastric treatment of soils. Group 4 was responsible for operating the ICP-AES and also tested hot spots that were found on the site. Curiosity-driven inquiries were encouraged to supplement the investigative framework of the MWDT. The four sample sites were chosen for field and laboratory tests based on how to manage the site in the long run and to assess water quality and risk associated with humans, with emphasis on where people would most likely interact with the site. In Fig. 2, Site 1 is the location of the main waste pile deposit on the mine site. Site 2 is on the north side of Clear Creek. Sites 3 and 4 are for proposed parking lots.

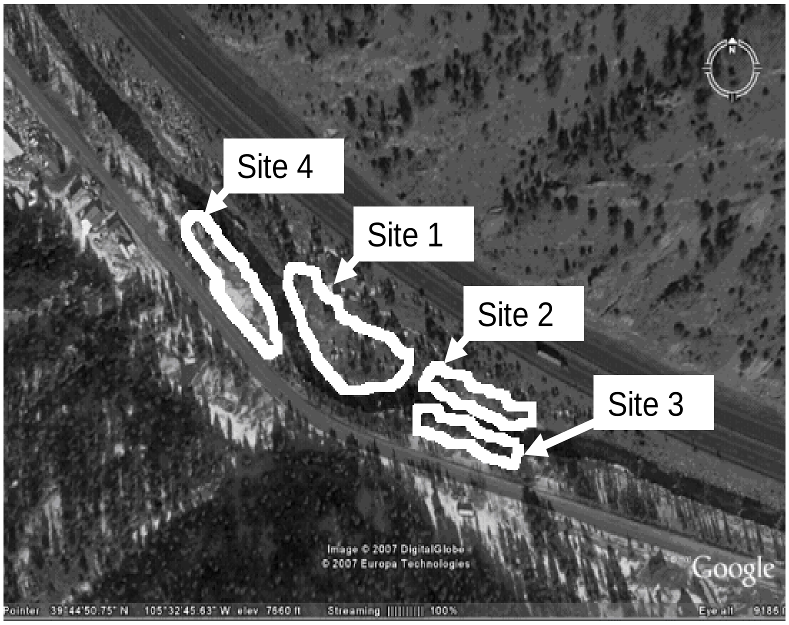

Figure 2. Satellite image of the Maude Monroe site and of the four waste piles found on the site.

Chemical and Toxilogic Analyses

Each Group individually assessed physical criteria of each site using the MWDT. Each group also collected a composite soil sample from each of the four site locations that consisted of 30, 1-cup volume, soil sub-samples. Each group combined and homogenized the sub-samples by sifting and sieving to less than 10 mesh. The CDMG leachate procedure was performed on the four homogenized samples by each group (4 samples, 4 groups resulting in 16 leachate tests altogether). Leachate procedures were performed in the field and consisted of mixing a 150 ml composite sample with 300 ml de-ionized water, stirring for 15 seconds, and covering the sample with a plastic bag for 90 minutes. At the end of the leaching period, pH and conductivity were measured using field meters, and a 10 ml volume of leachate was filtered and acidified for the ICP-AES analysis. Also, a mineral acidity titration using NaOH with a phenolphthalein indicator was performed. Each group was asked to make a sketch of the site and note what variables or characteristics might require special consideration.

Group 2 performed the METPlate ecological bio-accessibility test procedure using an 8 x 12 welled METPlate and with *E.coli* as the indicator species (Bitton, et al., 1994). In the procedure a chromophore buffer was added, followed by a 90 minute incubation period. Samples from each tube were added in duplicate to the wells. A chromogenic substrate of chlorophenol red was added to each well, and the METPlate incubated for 5, 15, 25 and 35 minutes. As per Fig. 3, the purple color developed indicates uninhibited enzyme production (CPRG hydrolization and no toxicity), while a yellow color indicates enzymes were inhibited, implying metal toxicity. The degree of color was measured with a microplate spectrometer.

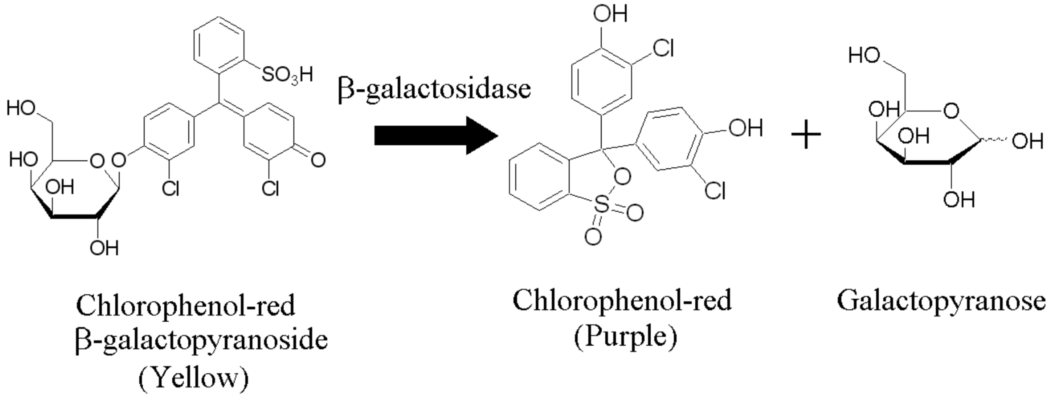

Figure 3. The chemical change occurring by the metabolism of healthy MetPlate enzymes.

Group 3 performed a human bio-accessibility assay using simulated saliva and gastric solutions (Sips et al., 2001). This test is designed to mimic the exposure to metals via soil ingestion. Both solutions consist of inorganic salts which are combined with a third solution containing organic compounds. The saliva inorganic salt solution was made by adding 5 mL of a 1.2 M KCl solution, 5 mL of a 0.21 M potassium thiocyanate solution, 5 mL of a 0.74 M sodium hydrogen phosphate solution, 0.85 mL of a 3.0 M NaCl, and 0.9 mL of a 1.0 M NaOH solution and bringing the contents up to a volume 250 mL with deionized water. The organic portion of the saliva was made by bringing 8 mL of a 0.42 M urea solution up to a volume of 250 mL. Then the inorganic and organic solutions were added together with 0.072 g amylase, 0.007 g uric acid, and 0.052 g mucin. The gastric solution was prepared by combining 7.85 mL of a 3.0 M NaCl solution, 1.5 mL of a 0.74 M sodium hydrogen phosphate solution, 4.6 mL of a 1.2 M KCl solution, 4.6 mL of a 0.57 M NH_4Cl solution, and 4.15 mL of 37% HCl and then bringing the combination up to a volume of 250 mL. The organic portion was made by combining 5 mL of a 0.36 M glucose solution, 5 mL of a 0.010 M glucuronic acid solution, 1.7 mL of a 0.42 M urea solution, and 5 mL of a 0.15 M glucosamine hydrochloride solution and then bringing the combination up to a volume of 250 mL. Then the inorganic and organic solutions are added together with 500 mg BSA, 500 mg pepsin, and 1.50 g mucin.

The leaching procedure was performed using the final saliva and gastric solutions at 37°C. Ten (10) mL of the saliva solution were added to bottles containing 0.250 g of sediment. The bottles were then incubated at 37°C in a rotating incubator for 5 minutes. Then 15 ml of gastric solution was then added to the bottles. The bottles were placed in a rotating incubator at 37°C for 2 hours. The bottles were then centrifuged for 5 minutes at 2750 g. The leachate was then filtered with a 0.45 μm filter, acidified with nitric acid, and analyzed for soluble metals using the ICP-AES.

Results

Physical Assessment Results

All four sites were given ratings used to make a physical assessment of a waste site. The criteria used to assess the physical characteristics of the waste pile are shown in Table 1. Table 2 gives the physical assessment results for groups 1, 2, and 4 for Site 3, the down stream south site that may be a proposed parking lot. Table 3 contains the average physical ratings for all of the sites that were given a physical assessment.

Table 1. Criteria used for physical assessment of a mine waste pile.

EROSION	**DISTANCE TO CHANNEL**	**VEGETATION ON PILE**	**VEGETATIVE KILL ZONE**
1 = none	1 = > 300 yds	1 = lots	1 = no kill zone
2 = sheet wash	2 = > 100 yds	2 = yes	
3 = rills < 6” deep	3 = > 100 ft	3 = little	3 = very little kill zone
4 = rills 6” – 12” deep	4 = < 100 ft		4 = trees but not underbrush
5 = gullies > 12”	5 = < 10 ft	5 = no	5 = yes

Table 2. Physical ratings for site 3 – Downstream South.

	Site 3 – Downstream South				
	Gr 1	Gr 2	Gr 3	Gr 4	Average
Erosion	3	3	-	2	2.7
Distance to Channel	5	5	-	5	5.0
Vegetation	5	3	-	5	4.3
Kill Zone	1	1	-	4	2.0
Overall Physical Rating	3.5/5	3.0/5		4.0/5	3.5

Table 3. Average ratings for all sites assessed at the Maude Monroe Mine.

Average Physical Assessments						
Site	**Site ID No.**	**Erosion**	**Distance to Channel**	**Vegetation on Pile**	**Kill Zone**	**Overall Assessment**
Main Site	**1**	2.3	5.0	3.3	1.3	3.0
Downstream North	**2**	2.0	5.0	4.3	2.7	3.1
Downstream South	**3**	2.7	5.0	4.3	2.0	3.5
Parking Lot	**4**	2.0	5.0	3.7	2.0	3.2
Across the Road	**-**	3	4	3	1	2.75
Pump House	**-**	1	3	1	1	1.5
Site Buildings	**-**	1	3	2	2	2

Chemical Assessment Results

Table 4. Criteria used for chemical assessment by the four groups.

ACIDITY	pH	EXCEEDS CRITERIA	CONDUCTIVITY
1 = 0 - 500 mg/L $CaCO_3$	1 = 4.0 - 5.0	1 = all of Cd, Pb, Ag, As, and Se below Aquatic Life	1 = below 0.5 mS/cm
2 = 500 -1000 mg/L $CaCO_3$	2 = 3.5 - 3.9	2 = one of Cd, Pb, Ag, As, or Se above Aquatic Life	2 = 0.5 - 1 mS/cm
3 = 1000 - 2500 mg/L $CaCO_3$	3 = 3.0 - 3.4	3 = two or three of Cd, Pb, Ag, As, or Se above Aquatic Life	3 = 1 - 2 mS/cm
4 = 2500 - 7500 mg/L $CaCO_3$	4 = 2.5 - 2.9	4 = four or five of Cd, Pb, Ag, As, or Se above Aquatic Life	4 = 2 - 3 mS/cm
5 = >7500 mg/L $CaCO_3$	5 = below 2.5	5 = any element above RCRA	5 = above 3 mS/cm

Table 5. The chemical assessment results using the MWDT for the four groups.

	Site 1				Site 2			
	Gr 1	Gr 2	Gr 3	Gr 4	Gr 1	Gr 2	Gr 3	Gr 4
Acidity	1	1	1	1	1	1	1	1
pH	4	4	4	4	3	3	3	3
Specific Conductance	3	3	3	3	3	3	3	3
Exceeds criteria	2	2	2	2	3	3	3	3
Overall Chemical Rating	2.5/5	2.5/5	2.5/5	2.5/5	2.5/5	2.5/5	2.5/5	2.5/5

	Site 3				Site 4			
	Gr 1	Gr 2	Gr 3	Gr 4	Gr 1	Gr 2	Gr 3	Gr 4
Acidity	1	1	1	1	1	1	1	1
pH	1	1	1	1	1	1	1	1
Specific Conductance	1	3			1	1	1	1
Exceeds criteria	1	3			1	1	1	1
Overall Chemical Rating	1	2			1	1	1	1

Bio-Assay Results

The following Figs. 4, 5, 6, and 7 give the results from the bioassay tests.

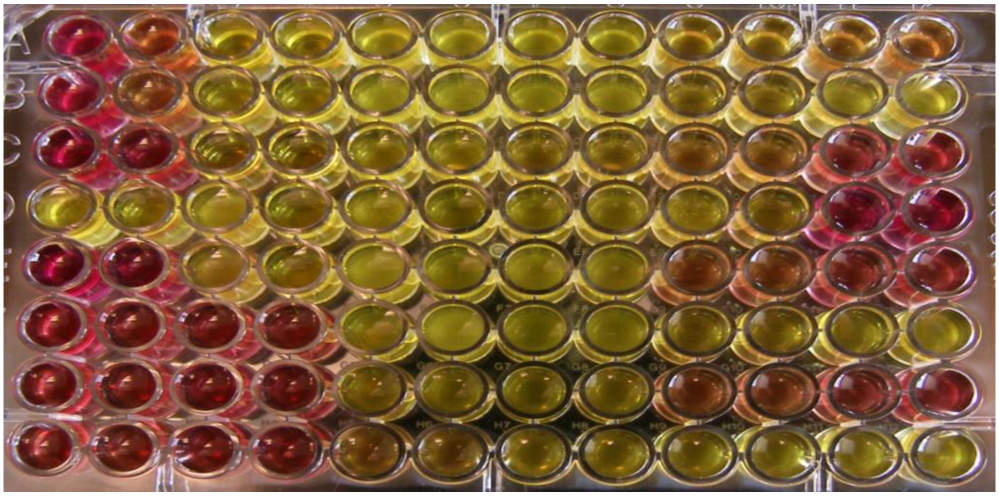

Figure 4. A photograph of the MetPlate color development after 15 minutes. Purple implies no inhibition due to metals; yellow implies inhibition and potential toxicity.

TL(-)	TL(+)	.1 10	.1 10	.1	.1	.2	.2	.3	.3	.4	.4
TL(-)	TL(+)	.2 10	.2 10	.1	.1	.2	.2	.3	.3	.4	.4
.3 10	.3 10	.4 10	.4 10	.1	.1	.2	.2	.3	.3	.4	.4
.1 10	.1 10	.2 10	.2 10	.1	.1	.2	.2	.3	.3	BLANK	BLANK
.3 10	.3 10	.4 10	.4 10	.1 50	.1 50	.2 50	.2 50	.3 50	.3 50	.4 50	.4 50
.1 10	.1 10	.2 10	.2 10	.1 50	.1 50	.2 50	.2 50	.3 50	.3 50	.4 50	.4 50
.3 10	.3 10	.4 10	.4 10	.1 50	.1 50	.2 50	.2 50	.3 50	.3 50	.4 50	.4 50
.1 10	.1 10	.2 10	.2 10	.3 10	.3 10	.1 50	.1 50	.2 50	.2 50	.3 50	.3 50

Figure 5. The configuration of samples in the MetPlate array. The first number is the Group number and the sites are in sequence down the column. Each site sample is run in duplicate. Blue = control, yellow=1:10 or 90% dilution, pink= 100% sample (no dilution), green= 1:2 or 50% dilution.

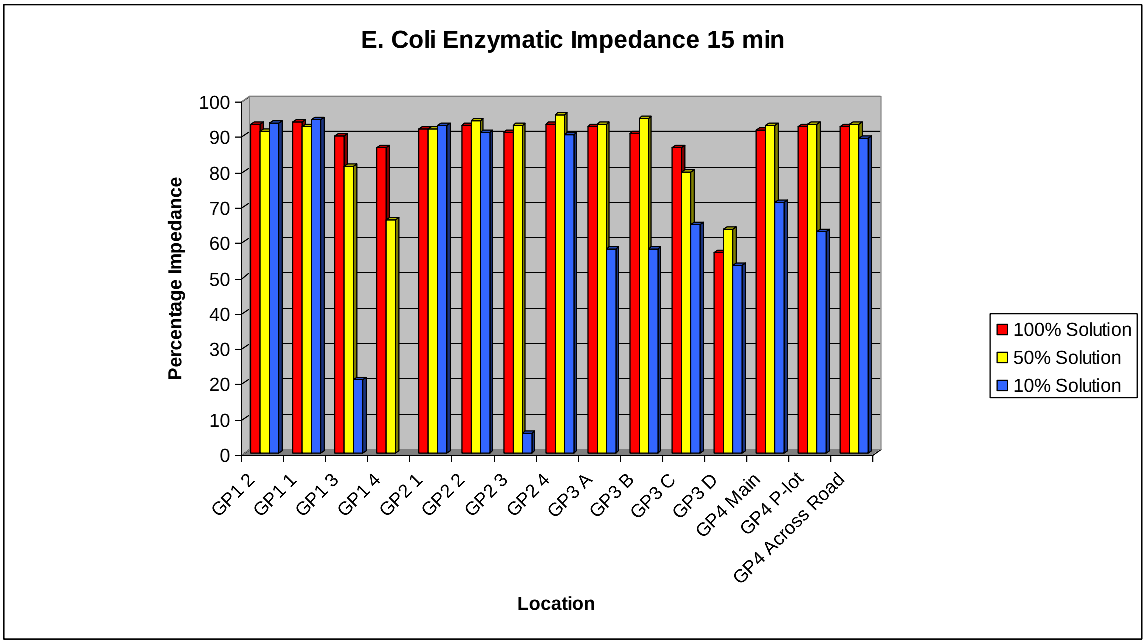

Figure 6. The percent impedance (implied toxicity) for the MetPlate test after 15 minutes.

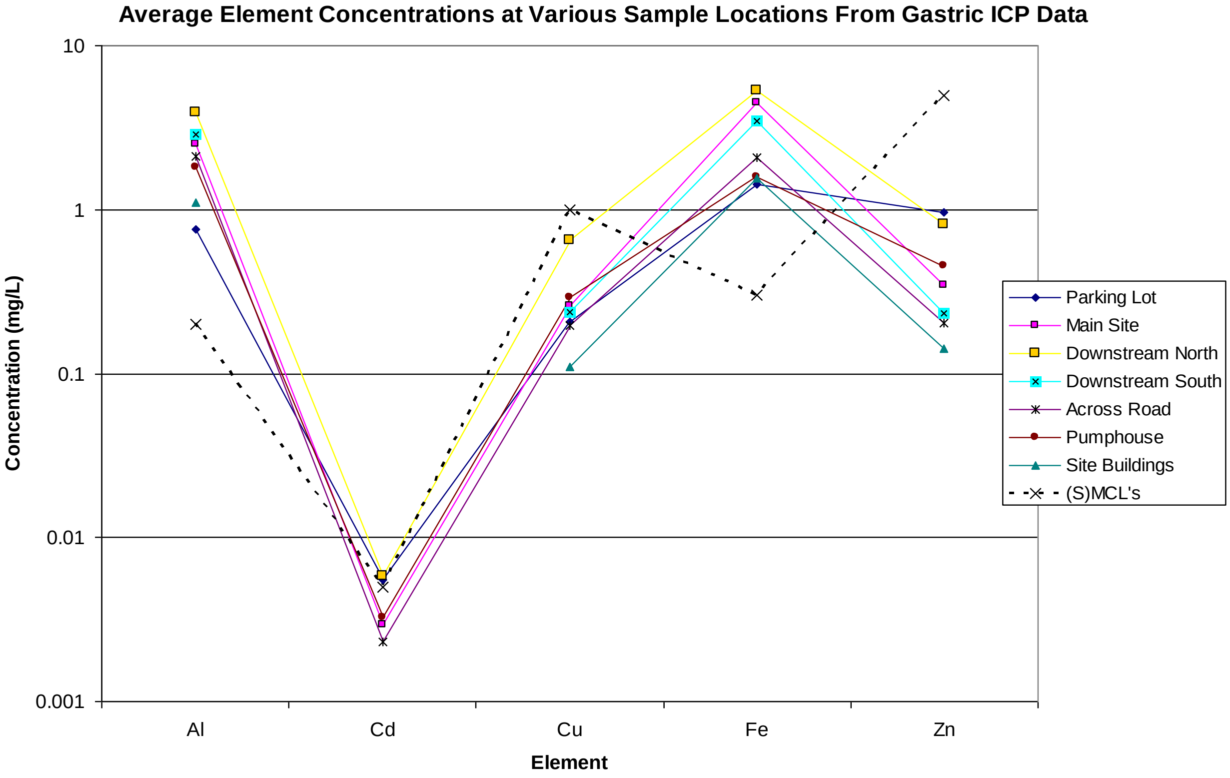

Figure 7. Gastric bio-assay results; the (S)MCL points are the primary or secondary concentration levels for drinking water.

Group 4 examined the mine site for "hot spots" or high concentrations of contaminants in small areas. Figure 8 is a graph of element concentrations in the leachate for an area near the mine shaft. Hot spots were visually identified via thick salt deposits, cementation crusts, and/or intense coloration of the soil surface.

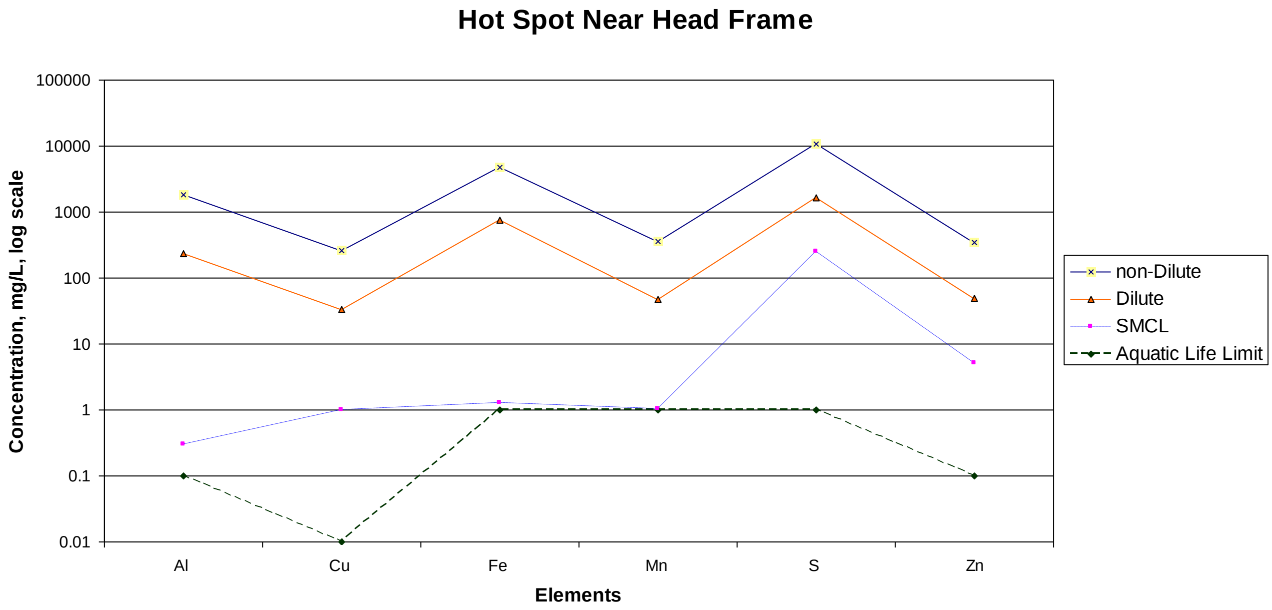

Figure 8. Contaminant concentrations in the "hot spot" leachates compared with aquatic toxicity concentrations and drinking water standards. The dilute points are the leachate diluted by a factor of 10.

Discussion

Physical and Chemical Assessments

As seen in Table 2, the results from the physical assessment of sites showed variation among the groups especially in the assessment of vegetation on the pile and the presence of the kill zone. On the other hand, the results from the chemical assessments were quite uniform. This is somewhat surprising because each group collected their own 30 sub-sample composite to make each chemical assessment. This definitely shows the efficacy of collecting a composite sample even though a waste pile can appear to be considerably heterogeneous. For both assessments, a value of 1 is good and 5 is bad. It is interesting to note that the physical assessment is worse than the chemical assessment at all four sites. In particular site 4, a possible site for a parking lot, physically appears to be bad but is relatively benign from a chemical point of view. This reinforces an observation made during analysis of other site and watershed assessments that there is no obvious relation between the physical and chemical assessment (Bazin et al., 2003, Heflin et al., 2004).

Although taking a composite sample is the first priority, this does not preclude the need to evaluate the site for unusual situations. The discovery of "hot spots" and exhibiting their possible toxicity is a good example of the need for this type of activity. The severity of the results from the leachate at the hot spot demonstrates the need to remediate this area before the public is allowed access to the site. As Fig. 8 shows, all elements that have primary and secondary concentration levels for drinking water and aquatic life limits established by the EPA, these levels were exceeded within in the hot spot leachate. Group 4 concluded the site seemed safe and adequate for use as a tourist attraction, with the exception of "hot spots". They suggested locating and remediating these areas, and adding signs and explanatory posters to alert the public of the risks associated with abandoned mine sites.

Bioassay tests

The bioassay tests are surrogates for toxicity monitoring using water fleas and fat head minnows. These conventional tests are difficult and take over a week to perform. On the other hand, MetPlate tests take only a few hours to set up and perform, and all the leachate waters can be tested in duplicate in one array plate. The results provide an indication of toxicity and identify the leachate samples that will need to be studied in greater detail. Also, the effect of diluting the leachates can be readily seen. Figure 5 shows that the undiluted leachates demonstrated significant metal toxicity for all four groups at all four sites.

It can be said that with respect to metal contaminants, aquatic organisms act like the canary in the coal mine. They are much more susceptible to metals toxicity than are humans. This can readily be seen in Fig. 7 where all leachate concentrations are below the drinking water standards, but Cd, Cu, and Zn, often exceed the aquatic concentration limits of 0.005 mg/L, 0.010 mg/L, and 0.10 mg/L, respectively (US EPA, 2004). Figure 8, depicts the aquatic limits, the drinking water standards, and the hot spot leachate concentrations. These data demonstrate that the aquatic limits for Cu and Zn are more than an order of magnitude lower than the drinking water standards.

Educational Observations

The four groups presented their findings in a class presentation. The groups individually arrived at similar conclusions. Specifically, they concluded that Al, Fe, Cu, and Zn concentrations exceed the Secondary Maximum Concentration Levels (SMCLs) for drinking water, as well as the Maximum Concentration Level (MCL) for Cd at Sites 1 and 2. Hence, the site's runoff and impact on water quality should be monitored, and if possible, remediated. The groups all concluded that the mine site could be successfully used as a museum, provided the public access to the site remains controlled such that access to hotspots and danger zones are restricted or the areas of inappropriate risk are stabilized or remediated. This joint conclusion was made based on the concept of "dose" – that all things have a potential risk, but if dose and exposure are controlled, the risk is acceptable. Groups 2 and 3 recommended debris removal and a safety inspection, particularly near the old mine shaft and for the equipment scattered around the site. Group 4 raised the issue of "error analysis" given the vast area covered. Collecting to represent the whole is a challenge that is exacerbated by the few samples that were taken. Group 4 also posed the question; how should hot spot areas be treated relative to the rest of the highly variable surface?

The total project was completed in one week and thus the groups did not have sufficient time to compare their results. Thus, they were not able to generate some of the ideas that are given in the above discussion where the results of all groups were compared. If this project was undertaken during a semester, it would be good to conduct a comparison of data and information gathered by the groups. Nevertheless, the students found this project to be the highlight of their four week field course. They were quite impressed that meaningful results could be generated using rather simple tests and analyses.

Conclusions

Exposing university students to the use of the mining waste decision tree (MWDT) through a field exercise proved successful in this abandoned mine site assessment project. Results were fairly consistent among the four groups, showing uniformity in observations and conclusions. This attests to the robustness of the decision tree and the chemical tests used in the assessment. Hence, this supports the use of the MWDT as a tool that can be used in similar field assessments by educational as well as professional groups. In addition, the use of the decision tree need not be limited to abandoned mine sites, but expanded to any site assessment requiring preliminary environmental study.

Literature Cited

Bazin, A., T. R., Wildeman, and N. Heflin. 2003. Aquatic toxicity assessment of two abandoned mine sites in the Lake City Mineral District, Colorado. Tailings and Mine Waste '03, A.A. Balkema Publishers, Lisse, Netherlands, 29-36.

Bitton, G., K. Jung, B. Koopman, 1994. Evaluation of a Microplate Assay Specific for Heavy Metal Toxicity. Arch. Environ. Contam. Toxicol. 27, pp. 25-28. http://dx.doi.org/10.1007/BF00203883.

Hageman, P. L., and P. L. Briggs, 2000. A simple field leach test for rapid screening and qualitative characterization of mine waste dump material on abandoned mine lands. Proceedings from the 5th International Conference on Acid Rock Drainage, Society For Mining, Metallurgy, and Exploration, Littleton, CO, pp.1463-1476, 2000.

Hageman, P. L., K. S. Smith, T. R. Wildman, and J. F. Ranville, 2005, Comparison of mine waste assessment methods at the Rattler Mine Site, Virginia Canyon, Colorado Proceedings America Society of Mining and Reclamation, 2005 pp 470-486.

https://doi.org/10.21000/JASMR05010470

Heflin, N., T. R. Wildeman, and R. Abel, 2004. Characterization and Contamination Assessment of Mine Waste Piles and Sediment Materials in Gilpin County, Colorado. Society for Mining, Metallurgy and Exploration, Preprints for Annual Meeting, No. 04-052.

Herron, J., Stover, B., and P. Krabacher, 1999, Cement Creek Reclamation Feasibility Report, Upper Animas Basin. Colorado Division of Minerals and Geology, Denver, CO.

Herron, J, Jordet, J. A., and T. R. Wildeman, 2001, Reclamation Feasibility Report: Virginia Canyon. Colorado Div. Of Minerals and Geology, Denver, CO, 60 pp.

Moehle, J., J. F. Ranville, L. Choate, T. R. Wildeman, and P. Ross. 2006, Leachate and enzyme bioassay toxicity assessment tests at the Tip Top Mine, a marginally impacted site, 7th International Conference on Acid Rock Drainage, 2006 pp 1314-1333. http://dx.doi.org/10.21000/jasmr06021314.

Sips, A. J. A. M., M. A. Bruil, C. J. G Dobbe, E. van de Kamp, A. G. Oomen, D. P. K. H., Pereboom, C. J. M. Rompelberg, and M. J. Zeilmaker. 2001, “Bioaccessability ofcontaminants from ingested soil in humans”. National Institute of Public Health and the Environment. RIVM Report

Smith, K. S., Ramsey, C., A., and P. L. Hageman, 2000, Sampling strategy for the rapid screening of mine-waste dumps on abandoned mine lands. Proceedings from the 5th International Conference on Acid Rock Drainage, Society For Mining, Metallurgy, and Exploration, Littleton, CO, pp.1453-1462.

Tessier, A., Campbell, P. C. G., and M. Bisson, 1979, Sequential extraction procedure for the speciation of particulate trace metals. Anal. Chem., Vol. 51, pp 844-851. http://dx.doi.org/10.1021/ac50043a017.

US Environmental Protection Agency, 2002, Test Methods for Evaluating Solid Wastes, Physical and Chemical Methods, http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm.

U.S. EPA (U. S. Environmental Protection Agency). 2004. National Recommended Water Quality Criteria. United States Environmental Protection Agency. Office of Water. (4304T) Available at http://www.epa.gov/waterscience/criteria/wqcriteria.html.

Wildeman, T. R., J. F. Ranville, J. Herron, and R. H. Robinson, 2003, Development of a simple scheme to determine the chemical toxicity of mine wastes, *In* Proceedings of the 9th Billings Conference and 20th National Conference of the. American Society for Surface Mining and Reclamation, pp 1501-1516.
https://doi.org/10.21000/JASMR03011501

Winkler, S., T. R. Wildeman, R. Robinson, and J. Herron. 1999. A concise method for mine soil analysis. *In*: Proceedings of 16th Annual Meeting of American Society for Surface Mining and Reclamation, pp. 236-244.
https://doi.org/10.21000/JASMR99010236