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Mine Waste Technology Program Electrochemical Tailings Cover

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Foreword

The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, EPA's research program is providing data and technical support for solving environmental problems today and building a science knowledge base necessary to manage our ecological resources wisely, understand how pollutants affect our health, and prevent or reduce environmental risks in the future.

The National Risk Management Research Laboratory (NRMRL) is the Agency's center for investigation of technological and management approaches for preventing and reducing risks from pollution that threaten human health and the environment. The focus of the Laboratory's research program is on methods and their cost-effectiveness for prevention and control of pollution to air, land, water, and subsurface resources; protection of water quality in public water systems; remediation of contaminated sites, sediments, and groundwater; prevention and control of indoor air pollution; and restoration of ecosystems. NRMRL collaborates with both public and private sector partners to foster technologies that reduce the cost of compliance and to anticipate emerging problems. NRMRL's research provides solutions to environmental problems by developing and promoting technologies that protect and improve the environment; advancing scientific and engineering information to support regulatory and policy decisions; and providing the technical support and information transfer to ensure implementation of environmental regulations and strategies at the national, state, and community levels.

This project was conducted under the Mine Waste Technology Program. It was funded by the EPA and administered by the U.S. Department of Energy (DOE) in cooperation with various offices and laboratories of the DOE and its contractors. It is made available at www.epa.gov/minewastetechnology by EPA's Office of Research and Development to assist the user community and to link potential users with the researchers.

Sally Gutierrez, Director
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Abstract

This report summarizes the results of Mine Waste Technology Program (MWTP) Activity III, Project 40, *Electrochemical Tailings Cover*, funded by the U.S. Environmental Protection Agency (EPA) and jointly administered by EPA and the U.S. Department of Energy (DOE). MSE Technology Applications, Inc. implemented the technology demonstration for EPA and DOE. This project addressed EPA's technical issue of Mobile Toxic Constituents – Water and Acid Generation.

The objective of Project 40 was to demonstrate the effectiveness of an electrochemical enhancement of conventional soil covers to inhibit the oxidation of sulfide minerals in mine waste to control generation of acid mine drainage. ENPAR Technologies, Inc. of Guelph Ontario, Canada, was the technology provider for trademarked electrochemical cover AmdEI™, which is an alternative to conventional earthen covers for decommissioning and long-term management of deposits of mill tailings and mine waste rock containing acid-generating sulfide minerals.

This demonstration showed evidence that the electrochemical tailings cover could reduce the oxidation of sulfide minerals in sulfide-containing mine waste. The reduction in oxidation of sulfur in the tailings was best shown by the post-test ABA analysis. The electrochemically treated cells retain total sulfur and pyritic sulfur at significantly higher levels than in the control cells that had no special treatment. In fact, treatment cell T2 retained over 90% of its original sulfur content. Nearly 50% of the sulfur was retained in the other treatment cell, T1; however, there was a large degree of variation in the initial sulfur data for this treatment cell, which seemed somewhat suspect due to high total sulfur content when compared to the three other test cells. Interestingly, both cells with the electrochemical treatment retained about 4.5% total sulfur while the untreated cells contained less than 0.05% sulfur at the conclusion of the demonstration. It is apparent that the sulfur was readily oxidized and leached away from the top few inches of tailings in the untreated control cells since initial total sulfur in the cells ranged from 4.78% to 9.44%.

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Acronyms and Abbreviations

ABA	acid-base accounting
AMD	acid mine drainage
COC	chain-of-custody
DOE	U.S. Department of Energy
EPA	U.S. Environmental Protection Agency
GSM	Golden Sunlight Mine
HDPE	high-density polyethylene
MDL	method detection limits
MSE	MSE Technology Applications, Inc.
MWTP	Mine Waste Technology Program
ORP	oxidation-reduction potential
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
SC	specific conductance
SOP	standard operating procedure

Acknowledgments

This document was prepared by MSE Technology Applications, Inc. (MSE) for the U.S. Environmental Protection Agency's (EPA) Mine Waste Technology Program (MWTP) and the U.S. Department of Energy's (DOE) Environmental Management Consolidated Business Center. Ms. Diana Bless was EPA's MWTP Project Officer, while Mr. Gene Ashby was DOE's Technical Program Officer. Ms. Helen Joyce was MSE's MWTP Program Manager. Ms. Diana Bless was the EPA Project Manager, while Ms. Lauren Drees was the EPA Quality Assurance Officer, for this project.

The following people also contributed significantly to the success of this project:

Gene Shelp, ENPAR
Brian Park, MSE Technology Applications, Inc.
Gary Wyss, MSE Technology Applications, Inc.
Diane Jordan, MSE Technology Applications, Inc.
Dave Sheldon, formerly of MSE Technology Applications, Inc.
Rory Tibbals, Golden Sunlight Mine
Shannon Dunlap, Golden Sunlight Mine

Executive Summary

The Mine Waste Technology Program (MWTP), Activity III, Project 40, *Electrochemical Tailings Cover*, was funded by the U.S. Environmental Protection Agency (EPA) and jointly administered by EPA and the U.S. Department of Energy (DOE) through an interagency agreement. MSE Technology Applications, Inc. implemented the project for EPA and DOE. Project 40 addresses EPA's technical issue of Mobile Toxic Constituents – Water and Acid Generation.

The ultimate goal of the *Electrochemical Tailings Cover* project was to demonstrate the effectiveness of an electrochemical enhancement of conventional soil covers to inhibit the oxidation of sulfide minerals in sulfide-containing mine waste to control the generation of acid mine drainage (AMD). The technology is trademarked as AmdEI™ by ENPAR Technologies, Inc. of Guelph, Ontario, Canada, as an alternative to conventional earthen covers for decommissioning and long-term management of deposits of mill tailings and mine waste rock containing acid-generating sulfide minerals.

The AmdEI™ electrochemical cover is designed to prevent the influx of oxygen into the sulfide-containing tailings or other waste materials to inhibit generation of AMD. The effectiveness of the technology was assessed by monitoring changes in sulfide oxidation (as manifested by sulfate concentration and pH of leachate samples) that occurs between electrochemically enhanced cells and identical test cells with no electrochemical enhancement over a 2-year test period. Due to the short test duration, information regarding longevity of the electrochemical cover was not acquired, and an economic analysis was not performed because the scale of the demonstration was so small.

Disappointingly, the leachate water samples failed to provide any conclusive evidence about the effectiveness of the electrochemical tailings cover. No significant trends in leachate sulfate, total sulfate, or pH could be denoted. The oxidation rate of the sulfur in the tailings may be responsible for the inconclusive findings from the leachate sulfate and pH. The duration of the demonstration may have been of insufficient length for the oxidation of sulfide to dominate the leachate chemistry.

1. Introduction

This document is the final report for the Mine Waste Technology Program (MWTP), Activity III, Project 40, *Electrochemical Tailings Cover Project*. The MWTP is funded by the U.S. Environmental Protection Agency (EPA) and is jointly administered by the EPA and the U.S. Department of Energy (DOE) through an interagency agreement. MSE Technology Applications, Inc. (MSE) implements the MWTP for EPA and DOE. This report details the project history, preparation, site selection, field testing, and final results. The project demonstrated the ability of an electrochemical cover to inhibit oxidation of sulfide minerals in mine waste tailings to control the generation of acid mine drainage (AMD).

1.1 Project Description

The objective of MWTP Activity III, Project 40 was to demonstrate the effectiveness of an electrochemical enhancement of conventional soil covers. The technology, trademarked as AmdEI™ by the technology provider (ENPAR Technologies, Inc. of Guelph, Ontario, Canada), is an alternative to conventional earthen covers for decommissioning and long-term management of deposits of mill tailings and mine waste rock containing acid-generating sulfide minerals.

The AmdEI™ electrochemical cover is designed to prevent the influx of oxygen into the sulfide-containing tailings or other waste materials to inhibit AMD generation. The effectiveness of the technology was assessed by monitoring changes in sulfide oxidation as manifested by pH and the concentration of sulfate in the leachate occurring between electrochemically enhanced cells and identical test cells with no electrochemical enhancement by monitoring for 2-years. Due to a shortened test duration and small test cells, information regarding longevity of the electrochemical cover and an economic analysis for a field-scale system was not performed.

1.2 Purpose

The purpose of this demonstration was to evaluate the effectiveness of ENPAR's AmdEI™ technology as an enhancement of conventional dry covers to prevent oxidation of sulfide minerals in sulfide-containing mine wastes.

Effectiveness of the electrochemical treatment was defined by the difference between the electrochemically enhanced cells and the cells without enhancement with respect to the amount of sulfide oxidation as:

- reduced amount of sulfate in leachate from the electrochemically enhanced cells (treatment cells) compared to untreated cells (control cells);
- neutral leachate pH from the treatment cells versus acidic pH leachate from the control cells;
- reduced conductivity in leachate from the treatment cells compared to control cells; and
- retention of total sulfur in the upper tailings in the treatment cells compared to reduced sulfur (and pyritic sulfur) in the upper tailings portion of the control cells.

1.3 Project Schedule

Original plans were to conduct the demonstration at Tailings Impoundment #1 at Barrick Gold's Golden Sunlight Mine (GSM) (formerly Placer Dome, Inc.) located near Whitehall, Montana. In April 2003, the EPA Project Officer suspended the project due to concerns regarding the suitability of the intended test site. In May 2003, MSE was directed to scale the project down and perform it under more controlled conditions. In the same timeframe, GSM management chose not to host the project at their site. It was therefore decided to transport materials from GSM to MSE and conduct the demonstration at MSE's site in Butte, Montana.

In September 2003, test materials, including tailings and soil cover, were transported from GSM to the MSE facility. Set-up of the cells began in September 2003 and installation was completed in October 2003. Beginning in May 2004, water was applied regularly and leachate pumped weekly throughout the summer and fall. Operations were suspended in October 2004 due to freezing temperatures. Water application restarted in May of 2005 and continued until October 2005 when the project was terminated and the test cells dismantled.

1.4 Report Structure

The final report has been organized systematically to facilitate ease of review. Starting with the pre-demonstration activities of the project, the document continues through the demonstration site description, the technology description, experimental test design, data collection, analysis, and evaluation. Other pertinent information concerning laboratory performance, field sampling, and analysis are provided as supporting documentation in Appendices A and B.

2. Demonstration Participants and Responsibilities

2.1 Demonstration Participants

The organization and execution of the *Electrochemical Tailings Cover* project was a collaborative effort between the technology developer and support organizations listed in Table 2-1.

2.2 Responsibilities

Demonstration of the technology was set up by the developers with support and operation from MSE, under the guidelines of the quality assurance plan (QAPP). Specific responsibilities are outlined in the following paragraphs.

2.2.1 MSE Technology Applications

MSE worked in consultation with the technical lead, EPA, and was responsible for:

- developing the work plan and QAPP;
- selecting an appropriate demonstration site;
- acquiring and transporting tailings from GSM for the demonstration;
- providing backhoe, bobcat, other earth moving equipment, and personnel needed for construction of the demonstration installation;
- irrigation water for demonstration installation;
- pumping and disposal of leachate from test cells;
- documenting the experimental methodology and operation of the technology;
- training operational and sampling personnel;
- performing field analysis and sampling activities, including duplication,

- packaging, labeling, storing, and shipping of samples;
- selecting and verifying a qualified analytical laboratory for demonstration quality assurance (QA) sample analysis;
- managing, evaluating, interpreting and reporting of demonstration data;
- evaluating and reporting technology performance; and
- developing the final report for the technology demonstration.

2.2.2 Developers

ENPAR is the sole developer of the electrochemical tailings cover technology used in this demonstration. ENPAR was responsible for:

- supporting the QAPP preparation;
- design and field installation as well as test cell configuration including specifications for instrumentation and monitoring systems;
- overseeing the selection of tailings and cover material from GSM to be used in the demonstration;
- characterization of tailings and soil cover materials used in the field installation;
- supervising the installation of the experimental cells at the MSE facility including installation of culverts, filling of cells with tailings, covering cells, anode installation, and testing electrical connections;
- monitoring the installation; and
- training MSE personnel in proper sampling and measurements as required.

Table 2-1. Demonstration Support and Developer Organizations

Organization	Principal Contact	Telephone Number
U.S. Environmental Protection Agency	Diana Bless	(513) 569-7674
U.S. Department of Energy	Gene Ashby	(406) 494-7298
MSE Technology Applications, Inc.	Brian Park	(406) 494-7415
ENPAR Technologies, Inc.	Gene Shelp	(519) 836-6155

3. Predemonstration Activities

3.1 Site Selection

Original plans were to conduct the demonstration at Tailings Impoundment #1 at GSM. ENPAR conducted initial characterization of the soil cover material and tailings from Tailings Impoundment #1 in the fall and winter of 2002 to obtain information for design of the field installation. In April 2003, the EPA Project Officer suspended the project due to concerns regarding the suitability of the intended test site. Of particular concern was the dryness of the tailings, with little chance for the collection of leachate samples. In addition, the tailings were partially oxidized, making it difficult to quantify the extent of oxidation over the course of the project. In May 2003, MSE was directed to scale the project down and perform it under more controlled conditions.

As previously mentioned, during this same timeframe, GSM lost interest in conducting the project at their site. It was therefore decided to transport test materials, including tailings and soil cover, from GSM to MSE and perform the technology demonstration at MSE. Tailings Impoundment #1, the original test site, had been previously capped and permitted. Therefore, collection of tailings would have been disruptive. Consequently, tailings were obtained from Tailings Impoundment #2. These tailings were similar to those from Impoundment #1 with the exception of a slightly coarser texture. Prior to placement of the tailings on Impoundment #2, the material was passed through a hydrocyclone allowing only the coarse fraction to be deposited on the impoundment. This was advantageous, as the coarser material facilitated water flow distribution through the test cells.

3.2 Regulatory Plans and Classifications

An access agreement between GSM and MSE was finalized in August 2003. The purpose of the agreement was to allow MSE to obtain tailings and soil cover for the demonstration and then return the soil and tailings after the demonstration was complete. The agreement was modified in

September 2004, due to the extension of the project for an additional year.

3.2.1 Hazards Classification

Excavation work necessary for setup and dismantling of the installation involved several risks. Potential risks were:

- electrical lines;
- other utilities such as gas pipelines; and
- hazards associated with the use and operations of heavy equipment, such as tipping of the equipment and injuries that can result from being stricken or crushed by heavy equipment.

Installation setup and dismantling activities were monitored by health and safety personnel.

Leachate accumulated during the demonstration was analyzed for chemical hazards before disposal.

3.2.2 Quality Assurance Project Plan

A QAPP was developed for this project and submitted to the EPA Office of Research and Development for review and approval (Ref. 1). The QAPP served as the standard operating procedure (SOP) for sampling, sample preparation, analytical laboratory protocol, and data reduction.

3.2.3 Analytical Laboratory

The selection of the analytical laboratory to process the demonstration samples was based on appropriate analytical capabilities, qualifications, and overall cost. The HKM Laboratory (now the MSE Laboratory) in Butte, Montana, was selected since it has extensive experience providing the type of analysis needed for evaluation in this project. An added benefit was the proximity of the laboratory to the MSE facility, where the demonstration was conducted. Samples were transported to the laboratory within one day of collection, prior to the expiration of any holding times for the requested analysis.

4. Demonstration Installation Description and Design

The test cells for the AmdEI™ technology demonstration were setup at the MSE Testing Facility in September 2003. Four in-ground test cells (two electrochemically enhanced treatment cells, T1 and T2, and two untreated cells, C1 and C2) were arranged in a square for ease of construction and to facilitate irrigation (Figure 4-1). Each test cell consisted of a 5-foot-diameter in-ground hole 10 feet deep. The test cells were contained within an upright corrugated steel culvert lined with an acid-resistant, water-impervious liner to contain the leachate. The top of each cell liner was located approximately 6 inches below the final grade. The total volume of each test cell was 7.3 cubic yards. A cross section view is shown in Figure 4-2.

The bottoms of the test cells were lined with 1.5 feet of quartz sand to facilitate collection of leachate. The use of high-grade quartz sand minimized the possibility of chemical reactions between the leachate and sand that could confound the results. Approximately 7.5 feet of tailings were placed in each cell on top of the quartz sand, with approximately 1.5 feet of soil cover obtained from GSM placed on top of the tailings. The 1.5-foot soil thickness was selected as a compromise to a more typical 3-foot thickness, as an effort to promote oxidation in the control cells during the relatively short 2-year test period. The soil cover was not compacted. The test cells that used electrochemical enhancement had the cathode mesh placed on top of the tailings and below the soil cover, and were wired to sacrificial anodes placed within the soil cover. The cathode mesh is shown on top of treatment cell T1 in Figure 4-3. This cathode mesh was harnessed to the magnesium anode shown in Figure 4-4.

Each test cell was equipped with a central 2-inch diameter well for the collection of leachate and to control the depth of an artificial water table. The well was curved at the bottom to form a water trap and prevent the influx of oxygen into the bottom of the tailings through the well. “Beads” of bentonite grout were placed at identical locations

in all cells (i.e., around the outer edge of the test cell, at the top of the cell and at the soil/tailings interface, as well as the same locations around the well) to prevent oxygen transport along those paths. Figure 4-5 shows the bentonite beads around the outer edge of the test cell and the 2-inch well pipe.

An irrigation system was installed to apply water to the test cells during the warmer months. The additional water was intended to further challenge the electrochemical cover and accelerate oxidation in the test cells during the relatively brief 2-year test. Meteorological data for the Butte area indicated an average annual precipitation of 12.3 inches, primarily received as rainfall in the summer months. The irrigation system supplemented the total annual precipitation, yielding a total annual precipitation to the test cells of approximately 30 inches. The increased amount of precipitation to the test cells was expected to ensure adequate volume for leachate sample collection and promote sulfide oxidation in the tailings.

After placement of the culvert sections in the ground, silica sand was placed in each cell to provide a small cone shape in the floor so that leachate was directed toward the center of the cell. Pre-fabricated 60-mil high-density polyethylene (HDPE) liners were placed in each cell. The central well was located in each cell and 1.5 feet of silica sand was placed in the bottom of each cell. Tailings were then loaded into the cells. The cells were loaded using a small “Bobcat” front-end loader, placing one bucket-load in each cell in a clockwise fashion [i.e., bucket-loads were placed sequentially in cells T1 (Treatment #1), T2 (Treatment #2), C2 (Control #2), and C1 (Control #1), referring to Figure 4-1]. After the addition of tailings, approximately one vertical foot each, the tailings were distributed by hand within each cell to ensure there were no gaps or air pockets. There was a slight deviation in the loading for test cell C1, which was filled to about 1.5 meters from the top of the culvert before the previously described

sequence was followed. Samples were taken at approximately 1-foot intervals and submitted to the laboratory for acid-base accounting (ABA) analysis. Simultaneous loading of the test cells ensured reasonable homogeneity and an equal starting point for all treatments. In addition, the tailings used for testing were obtained from a single location on the Impoundment #2 dam; these tailings had been greatly homogenized by passing through GSM's mill (i.e., crushing, grinding, vat leaching) and then deposited at approximately the same time. 1.5 feet of soil cover from GSM was placed in each cell, with the final grade being 6 inches above the top of each cell resulting in cells that were representative of being in the ground. The cells' ground cover was extended above grade to prevent the possibility of snowdrifts accumulating in an uncontrolled manner and channeling water and/or oxygen down the sides of the cells. The final test cell configuration is shown in Figure 4-6.

A central irrigation system was installed, with one sprinkler head in the center of the cell arrangement

that delivered water to all four cells. The irrigation system was calibrated so that the amount of water was known and uniform.

Upon completion of test cell installation in October 2003, irrigation water was applied regularly (and leachate pumped out weekly) until cold weather prohibited further irrigation. The purpose of this was to initiate water flow through the system and to allow the bacteria responsible for facilitating sulfide oxidation (e.g., *Thiobacillus ferrooxidans*) to become acclimated to their new environment; this could be thought of as "priming" the system. Little activity was expected during the winter months due to low temperatures and no additional supplemental water. Beginning in the following spring, May 2004, water was regularly applied and leachate pumped out weekly throughout the summer and fall, until operations were suspended due to freezing temperatures in October 2004. Water application began again the following spring in May and continued until October 2005, when the project was terminated and the cells dismantled.

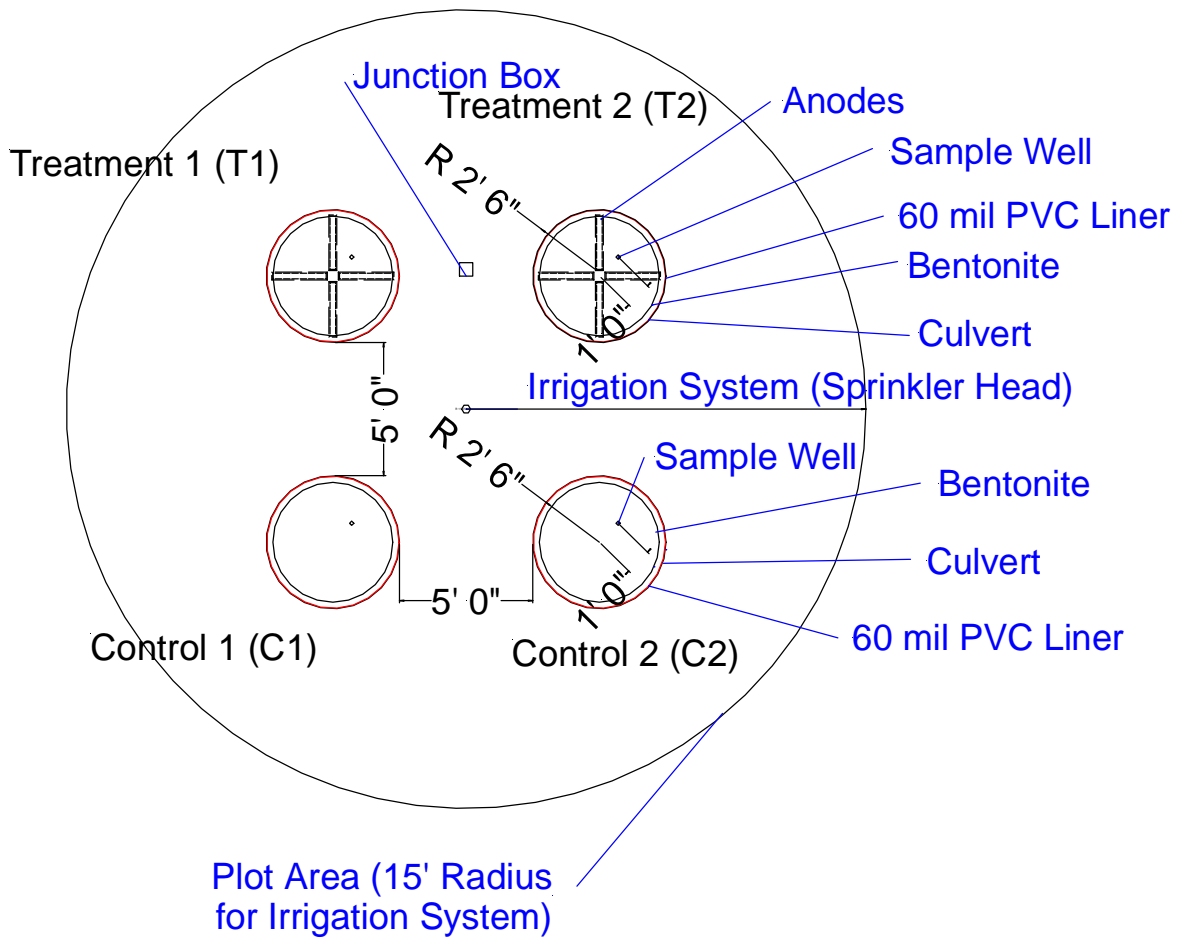


Figure 4-1. Top view of demonstration installation.

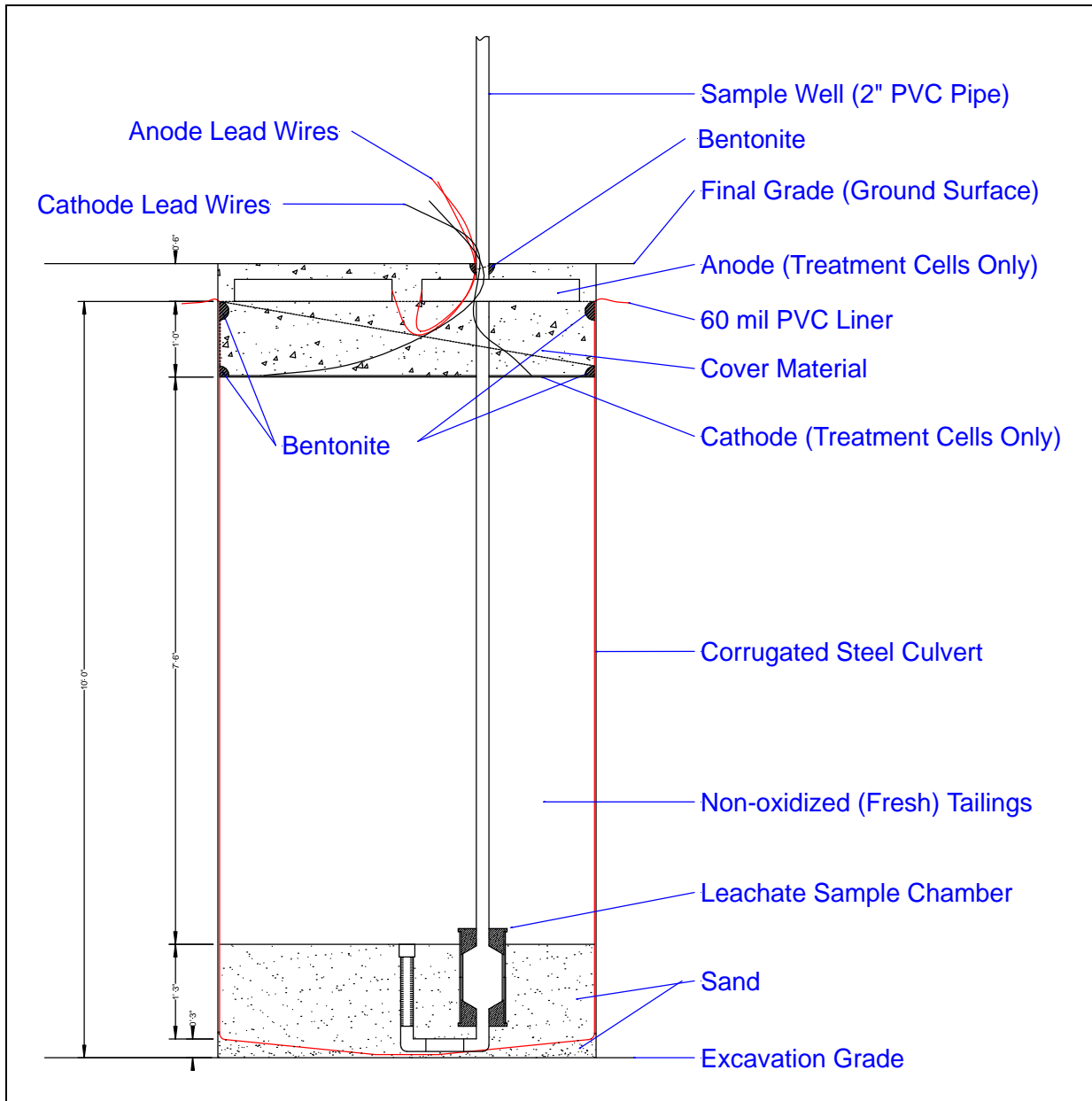


Figure 4-2. Cross-sectional view of cell installation.

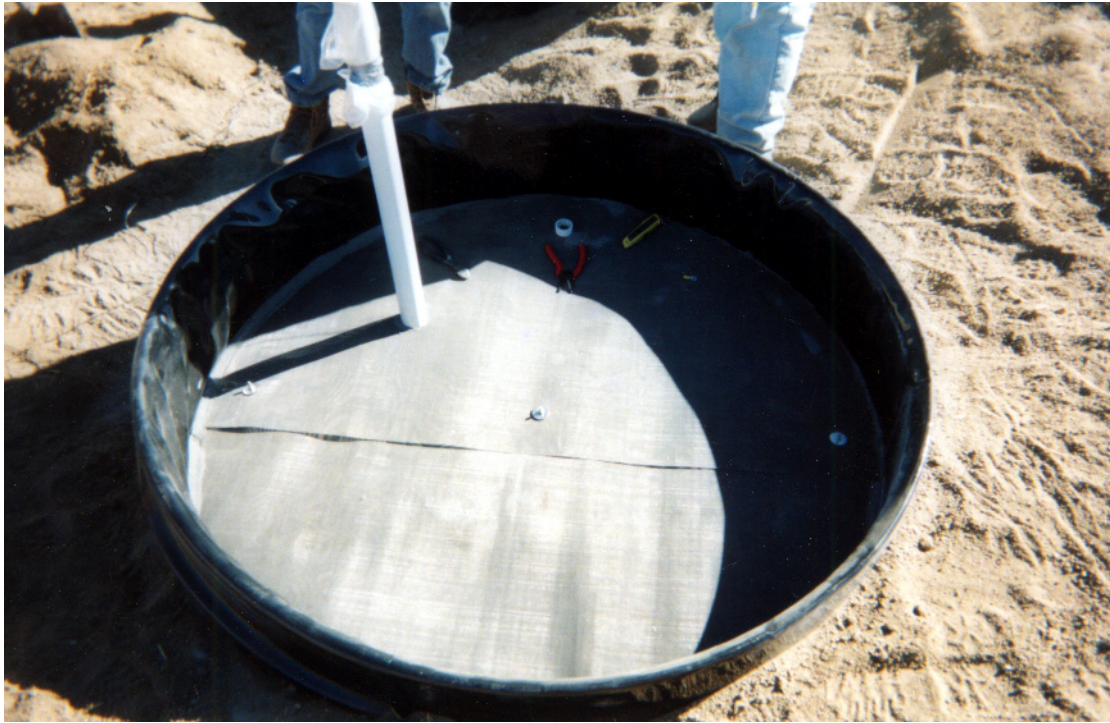


Figure 4-3. Cathode mesh in treatment cell T1.



Figure 4-4. Magnesium anodes.



Figure 4-5. Bentonite beads around test cell outer edge and well pipe.



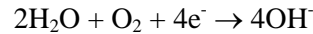
Figure 4-6. Test cell final configuration September 30, 2003.

5. Demonstration Technology Description

This demonstration implemented ENPAR's electrochemical cover technology. One of the conventional approaches currently used to control the generation of AMD from mining waste is to install engineered dry earthen or soil covers over the waste material. In these types of installations, earthen materials trucked from local sources are commonly used to construct dry soil covers. The AmdEI™ technology is designed to reduce the amount of earthen material/soil cover needed for a typical repository. The cost of earthen covers is dependent upon the thickness and complexity of the cover and the distance the earthen materials must be transported to the site.

The ENPAR technology is designed to limit the infiltration of water and oxygen into sulfide-containing mill tailings and mine waste rock; thus, eliminating oxidation of the sulfide minerals, and consequently inhibiting the generation of AMD. This electrochemical cover technology is an enhancement of conventional dry covers and consists of a cathode (i.e., steel mesh), an electrolyte (i.e., the soil cover mixture), and an anode (i.e., a sacrificial material such as magnesium). The concept of the electrochemical cover is to prevent oxidation of sulfide minerals by inhibiting the infiltration/diffusion of oxygen into the sulfide wastes. The fundamental electrochemical process is the transfer of electrons from the anode to the cathode through an external circuit due to the presence of the galvanic couple set up between the cathode and anode. This transfer of electrons produces a chemical reaction

at the cathode/electrolyte interface that reduces the concentration of oxygen and produces alkalinity according to the following reaction:



The technology is covered by U.S. patent 5,630,934 (May 20, 1997). There are both Canadian (209581) and international (9927716.2) patents pending.

There is also some evidence that the cathode can generate an electro osmotic effect by attracting dissolved cations, creating a localized zone with a higher concentration of cations compared to the surrounding soil matrix. The result is a difference in osmotic potential between the cathode surface and the soil matrix producing a localized gradient where water is drawn to the cathode, increasing the moisture content around the cathode. This effect would also inhibit the transport of oxygen to the underlying sulfide waste material, further enhancing the ability of the electrochemical cover to prevent AMD.

The AmdEI™ electrochemical cover is independent of the mineralogy and the associated electrical properties of the underlying waste material. The system provides a uniform sink for oxygen established over the entire surface of the tailings/waste rock deposit. Given the nature of the electrochemical system, the cathodic material is prevented from breakdown, similar to cathodic protection of underground steel pipes and tanks.

6. Experimental Design

6.1 Technology Demonstration Objectives

The objective of the project was to demonstrate the effectiveness of electrochemical enhancement of conventional soil covers to inhibit the oxidation of sulfide minerals in sulfide-containing mine waste, and consequently control AMD generation.

6.2 Factors Considered

Several factors considered in the development of the experimental design were:

- type of material to be used for tailings and cover;
- selection of the materials to be tested;
- selection of analytical parameters to measure extent of oxidation;
- design of the test cell;
- amount of precipitation needed to maintain the appropriate artificial “water table” and promote weathering; and
- sampling frequency of the test cell leachate.

6.3 Installation Tailings Characterization

Initial samples of tailings taken during cell loading were analyzed for ABA, total metals, size distribution, and paste pH. The laboratory data is presented in Appendix A.

6.3.1 Acid-Base Accounting

The ABA results were used to establish the acid-generating variability between cells as well as within cells. The ABA values provided the initial sulfur concentrations for the test. Some samples were obtained from the quartz sand and soil cover for ABA for completeness, even though these materials were known to be free of acid-generating sulfur. This is verified by the ABA results as shown in Appendix A.

6.4 Sampling Design

Quality control (QC) sampling format was derived from the QAPP. The sampling frequencies were somewhat relaxed when the decision was made to extend the demonstration an additional year to

October 2005. Sampling was performed in three separate phases: 1) field installation, 2) monitoring, and 3) post-test or completion. The type of samples, parameters, classification (i.e., primary/non-primary), matrix, and proposed frequency were defined in the QAPP. The original sampling criteria is found in Appendix B.

6.5 Monitoring Phase Measurements

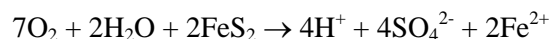
Measurements obtained after the installation and prior to dismantling of the test cells were considered monitoring phase samples. Samples from this phase comprise the majority of data obtained during the demonstration. This data reflected the impact that the electrochemical cover technology had on the treatment cells during the demonstration.

6.5.1 Electrochemical Measurements

The electrochemically enhanced treatment cells had the cathode mesh placed on top of the tailings and below the soil cover, and were wired to sacrificial anodes placed within the soil cover. During the monitoring phase, voltage and current measurements were made intermittently, but at least monthly, to ensure the galvanic couple was present. The galvanic couple provides the fundamental electromotive force (EMF) that maintains the desired reducing conditions in the treatment cell tailings.

6.5.2 Sulfate and pH Measurements

Leachate sulfate and pH measurements were taken to assess oxidation of the tailings during the monitoring phase. Sulfate and acidity are produced during the oxidation of tailings containing sulfide minerals. Typically, pyrite is the sulfide mineral present that provides the sulfur source in AMD generation. Pyrite undergoes oxidation to generate AMD by the following equation:



Fresh tailings contain little sulfate; however, as water percolates through the tailings as they are being oxidized, the sulfate will increase while the pH decreases. By this mechanism of measuring the leachate sulfate and pH over time, the progress of sulfide oxidation can be monitored. In the demonstration, leachate sulfate concentration was used as the primary parameter to indicate the presence of oxygen in the tailings; thus, showing the amount of sulfide oxidation that has occurred. Data was compared between treatment and test cells to indicate the effectiveness the electrochemical cover had on oxidation of the sulfide minerals and AMD.

During the period when water was applied, intermittent leachate samples were collected and analyzed for sulfate and pH as primary measurements. Total leachate volume was recorded and used to calculate total mass of sulfur removed. Specific conductance (SC) measurements were taken in the field from the combined leachate. As AMD is generated, increases in total dissolved solids should be seen, resulting in increasing SC values.

6.5.3 Dissolved Metals

Leachate samples analyzed for dissolved metals were gathered periodically throughout the project. As a secondary measurement, dissolved metals results provided additional information to assist in the interpretation of the chemistry in the test cells.

6.6 Completion Phase Measurements

At the completion of the project, in the fall of 2005, the test installation was dismantled and the tailings and soil cover were returned to GSM. Prior to dismantlement, three tailings cores were taken by the geoprobe in random locations in each test cell for visual evaluation of oxidation and laboratory analysis. Samples were taken from the top three inches of the cores and analyzed for ABA and 1:1 pH.

6.6.1 Acid-Base Accounting

The ABA samples taken at the completion of the demonstration were to determine changes in the sulfur content of the upper-most portion of the

cells. Comparison of the tailings total sulfur and pyritic sulfur content was used to evaluate oxidation in the test cells, as well as determine the effectiveness of the electrochemical cover when compared with the treatment and control cells.

6.6.2 1:1 pH

Since each core sample was limited, 1:1 pH analysis was used at test completion instead of the saturated paste pH. The 1:1 pH is a measure of the hydrogen ion activity in a soil slurry. This measurement indicates the presence or absence of free acids in the soil, much like a saturated paste pH. Differences between the two methods are equilibration time and the solid to liquid ratio. Equilibration time for the paste pH is at least four hours, but typically overnight, while a 1:1 pH is approximately half an hour. This means that there is less time for slightly soluble salts to dissolve and moderate the pH in the 1:1 pH determination. The solid to liquid ratio is greater for the saturated paste pH than for the 1:1 pH, but the dilution effect alone does not greatly alter the resultant pH. The pH result is not significantly biased between the two methods because de-ionized water is used in the slurry and paste, and has no buffering capacity to contribute to the mixture.

6.7 Statistical Analysis

The original experimental design was to be a completely randomized design using a one-way treatment structure with repeated measures. Due to changes in the frequency and number of samples taken when the project duration was extended, the application of the original design for statistical analysis was not possible. In planning the demonstration, the factors considered critical to indicate the success of the demonstration were:

- sulfate leachate production;
- leachate pH; and
- total mass of sulfate leach from the test cells.

The percent reduction in the oxidation of sulfide is quantified by measuring the difference between the mass of sulfate leached from the treatment and control cells as shown by the equation:

If the treatment is effective, the percent reduction of oxidized sulfide would increase with time. Figure 6-1 presents the results from the demonstration. Inspection of the plot reveals no specific trends, and the percent reduction appears to be nearly random with respect to time. It must be concluded that either reduction of oxidized sulfide was an inappropriate measure of the electrochemical cover effectiveness, or the cover was ineffective.

$$\% \text{ reduction} = \frac{(\text{SO}_4^{2-}{}_{\text{control}} - \text{SO}_4^{2-}{}_{\text{treated}})}{\text{SO}_4^{2-}{}_{\text{control}}} 100$$

After summarization and review of the analytical data, it appears that the total sulfur and pyritic sulfur content, from the ABA analysis, display marked differences between the start and end of the demonstration. For statistical analysis, tailings pre-test (n=8) and post-test sulfur (n=3)

measurements were assumed to follow a normal distribution. Total and pyritic sulfur concentrations, means, and standard deviations are summarized in Tables 6-1 and 6-2. Significant change in sulfur concentration was indicated when the initial and post tests decreased by more than 2 times the standard deviation (2σ) of the original concentration. The control cells showed a significant loss of total sulfur over the duration of the demonstration. Total and pyritic sulfur are retained to a much greater degree in both treatment cells. The initial and final sulfur ranges overlap for cell T2 suggesting that there was not a significant loss of sulfur within that cell. Treatment cell T1 retained approximately the same amount of total and pyritic sulfur as cell T2. Treatment cell T1 initial sulfur content was outside the range of the final sulfur content for that cell. Even though there was a large fraction of sulfur remaining in cell T1, it showed a significant loss of sulfur during the course of the demonstration.

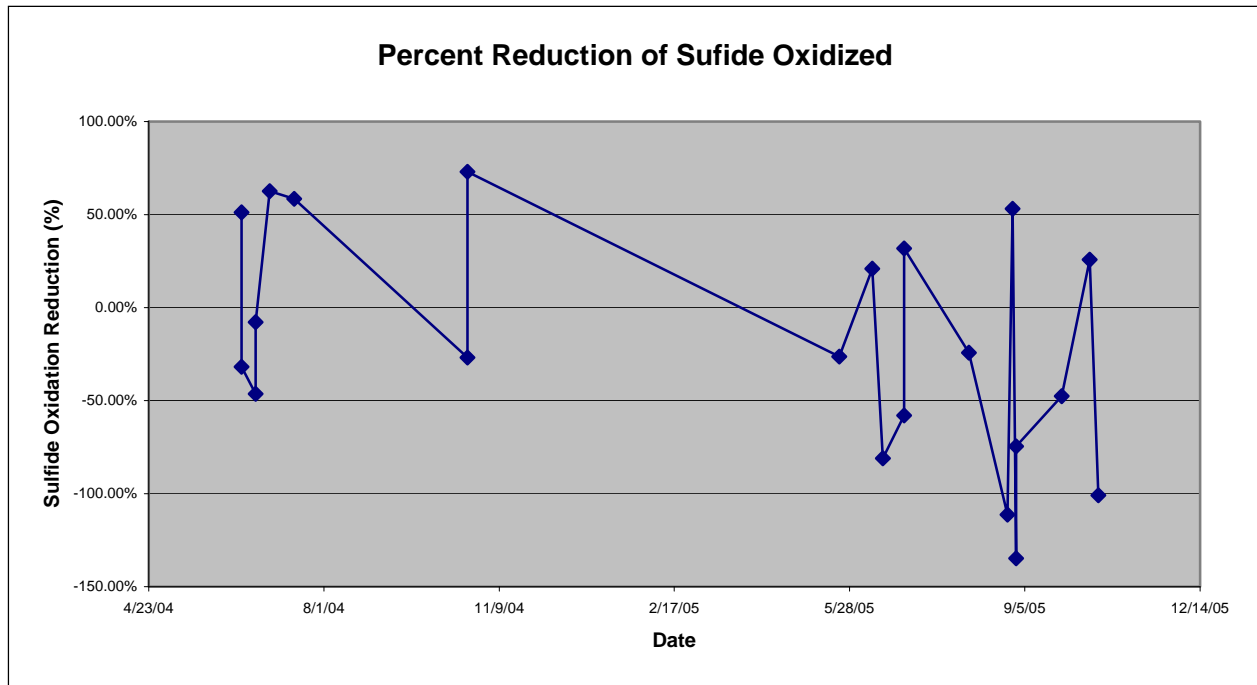


Figure 6-1. Percent reduction in sulfide oxidation.

Table 6-1. Pre-Test and Post-Test ABA Total Sulfur Comparison

Test Cell	Project Phase	Total Sulfur Average (%)	Standard Deviation (%)	2 σ (%)	Range	
					Average + 2 σ (%)	Average - 2 σ (%)
C1	Initial	5.40	0.64	1.29	6.69	4.11
	Final	0.05	0.010	0.02	0.07	0.03
C2	Initial	6.07	0.66	1.33	7.40	4.74
	Final	0.04	0.000	0.00	0.04	0.04
T1	Initial	9.44	1.40	2.80	12.24	6.64
	Final	4.51	0.18	0.35	4.86	4.16
T2	Initial	4.78	0.23	0.45	5.24	4.33
	Final	4.44	0.14	0.27	4.72	4.17

Table 6-2. Pre-Test and Post-Test ABA Pyritic Sulfur Comparison

Test Cell	Project Phase	Pyritic Sulfur Average (%)	Standard Deviation (%)	2 σ (%)	Range	
					Average + 2 σ (%)	Average - 2 σ (%)
C1	Initial	4.45	0.61	1.23	5.68	3.22
	Final	0.03	0.010	0.02	0.05	0.01
C2	Initial	4.88	0.65	1.30	6.18	3.58
	Final	0.02	0.010	0.02	0.04	0.00
T1	Initial	7.83	1.52	3.05	10.88	4.78
	Final	3.98	0.30	0.60	4.58	3.38
T2	Initial	3.67	0.33	0.65	4.32	3.02
	Final	4.01	0.14	0.28	4.29	3.73

7. Field Sampling and Analysis

7.1 Techniques and Methods

All samples were taken and submitted to the analytical laboratory within the guidelines provided in the QAPP. All samples delivered to the laboratory were accompanied by chain-of-custody (COC) to serve as a record of the analytical request and transfer of possession. Installation tailings samples were random grab samples taken at approximately 1-foot intervals in 1-gallon Ziploc bags as the cells were being loaded. A composite sample was taken for metals analysis. A sample of the sand and soil cover sample from each of the test cells was also obtained for analysis.

Field data was collected during the monitoring phase of the project from aqueous samples that consisted of 1) plant water used for irrigation of the cells, and 2) cell leachate samples. Field parameters taken on the fresh aliquots of the aqueous samples were pH, SC, oxidation-reduction potential (ORP), and dissolved oxygen. These measurements were made with the YSI 556 Multi Probe System.

Leachate pumped from the test cells was collected in a graduated tank or weighed to determine the volume produced from each of the cells.

Irrigation water was analyzed for the parameters of dissolved metals, sulfate, alkalinity, and chlorine. Leachate samples were analyzed for total metals, pH, and sulfate. Alkalinity and sulfate samples were taken in 500-mL HDPE bottles. Total metals were taken in 500-mL HDPE bottles and dissolved metals in 250-mL HDPE bottles. Chlorine was taken in a 250-mL HDPE bottle with minimal headspace. All HDPE sample bottles were rinsed three times with the subject

water before collecting the aliquot for analysis. Samples were stored at 4 °C until delivery to the analytical laboratory.

Core samples were taken using a Geoprobe at the completion of testing, immediately prior to dismantling the test cells. Three cores were taken from each cell in random locations and stored in sealed 1-inch plastic tubes under refrigeration until analyzed.

7.2 Field Sample Analysis and Data Recording

Parameters measured in the field were:

- pH;
- SC;
- ORP;
- dissolved oxygen; and
- leachate volumes.

All calibration and sample measurement data was recorded in a bound all-weather notebook with waterproof ink. To ensure that field measurements were of acceptable quality, all parameters were required to meet the calibration check criteria stated in the QAPP (Section 5). The results of the calibration checks were likewise recorded in the project logbook for reference.

7.3 Instrument Accuracy

The accuracy of the field measurements is directly related to the accuracy of the instrumentation and procedures used in obtaining the values. The YSI 556 Multi Probe System was calibrated in compliance with the requirements set forth in the QAPP. The requirements are outlined in Table 7-1.

Table 7-1. Calibration Requirements for Process Field Measurements

Parameter	Measurement Classification	Process Instrument	Calibration Procedure	Frequency of Calibration	Expected Range/ Expected Accuracy
pH	Primary	pH Electrode	2 points	Daily	-2.00 to 16.00 ±0.1
SC	Nonprimary	Conductivity Electrode	Calibrate with 1412 microSiemens per centimeter (uS/cm) standard	Daily	5,000 to 20,000 µS/cm ±1%
Dissolved Oxygen	Nonprimary	Dissolved Oxygen Electrode	Per Manufacturer's Specification	Daily	0 to 10 milligrams per liter (mg/L) ±2%
ORP	Nonprimary	Redox Electrode	Zobell's Solution	Daily	-250 to +250 millivolts (mV) ±20 mV
Leachate Volumes	Primary	5-gallon tank	Manufacturer's Graduation	NA	3 to 10 gallons, 0.1 gallon

8. Discussion

The ENPAR project was originally setup to be conducted at the GSM. Concerns with the amount of precipitation, suitability of demonstration location and loss of interest on the behalf of GSM, the demonstration was moved to the MSE facility.

The analytical results of samples taken are presented within the following sections. Data is presented in graphs and a summary of the quality assurance activities from the project specific QAPP are contained in Appendix C.

8.1 Field Installation

The experimental setup consisted of two treatment cells and two control cells (Figure 4-1). The natural precipitation was augmented by a sprinkler system that was intended to provide enough moisture to maintain an artificial water table in the cells and provide ample water to generate leachate sampling, and accelerate the weathering processes in the tailings (Figure 4-2).

8.1.1 Acid-Base Accounting

The demonstration cells were sampled during installation while they were being filled with tailings at approximately 1-foot intervals. Samples of the sand, used in the saturated zone of the cells, and soil cover were obtained during the installation process. The ABA results for these samples are tabulated in Appendix A. The field installation samples were analyzed for size distribution, saturated paste pH, and total metals. Tabulation of these results is found in Appendix A.

8.1.1.1 Data Interpretation

The average sulfur content of the tailings in the test cells ranged from 4.78% to 9.44% sulfur, with the overall average being 6.42%. Cell T1, which had the highest total sulfur concentration, exhibited the largest variability amongst the analysis of the 1-foot interval samples, and it was notable that results from this cell displayed the largest variation in the referee sample results. The majority of the sulfur content in the tailings samples was initially present as pyritic sulfur. The

sand and soil cover had very little, if any, sulfur present, which is shown by the results being below or at the method detection limits.

8.1.2 Other Installation Analysis

Saturated paste pH, total metals, and sieve analysis were performed on the composite samples taken during the demonstration installation. The results are tabulated in Appendix A. The analysis shows the particle size to be relatively consistent between the test cells. The mean particle size is 0.22 to 0.23 millimeters in diameter (Appendix A) and the saturated paste pH is nearly neutral for each test cell. Metals are similar between the test cells. All metals show little variation in concentration between the composite samples, as depicted by a relative standard deviation of less than 15%.

8.2 Monitoring Period Leachate Analyses

The majority of the data was taken during the monitoring phase of the demonstration. The purpose of the data obtained during this phase was to detect changes in the physical or chemical parameters of the leachate. These data could provide insight into the progress of oxidation within the treatment and control cells. A requirement during this phase was to establish an artificial water table within the test cells so there would be an aqueous reservoir from which to obtain aqueous samples. Leachate samples were pumped from the artificial water table within the test cells. The water depth in each test cell is presented in Figure 8-1.

8.2.1 Voltage and Current Measurements

The voltage and current were monitored throughout the demonstration to insure the galvanic couple, between the cathodic covering and the anode in the treatment cells, was established at installation and maintained until dismantling. The galvanic couple is a required element to provide electrons for the chemical reactions necessary in the operation of the electrochemical technology. The treatment cells voltage and current data is presented in Figures

8-2 and 8-3. The graphs show that indeed an electrical connection, galvanic couple, was present throughout the project.

8.2.2 Leachate Analysis

The leachate from each test cell was analyzed for pH, sulfate, SC, and total metals during the monitoring phase. The leachate volume produced was also recorded.

8.2.2.1 Leachate Volume

The leachate volume accumulated over the duration of the demonstration is shown in Figure 8-4. The data indicates that more leachate was pumped out of the control cells compared to the treatment cells. Reasons for the disparity in the leachate production remain unclear, since the soil physical properties appear to be relatively similar between all the test cells from the installation data. One potential cause is that the electrochemical cover may have impeded migration of the irrigation water and precipitation; possibly retaining water in the upper portions of each cell, therefore allowing more evaporation in the treatment cells.

8.2.2.2 Test Cell pH

The pH of the test cell leachates at sampling, are shown in Figure 8-5. No significant difference between the control and treatment cells was noted for the duration of the demonstration. In all the test cells, the pH remained constant, with the exception of the samples taken on October 6, 2004. The pH readings from October 6, 2004 are questionable, since tank collection water had siphoned back into the wells. The test cells' pH was relatively neutral, with the pH still above 6.3 at the conclusion of the demonstration.

8.2.2.3 Sulfate

Sulfate was a primary parameter for determining the effectiveness of the electrochemical cover sulfide oxidation. The leachate sulfate concentration over time is shown in Figure 8-6. The cumulative mass amount of sulfate leached from the test cells is displayed in Figure 8-7. Sulfate production is one measure of oxidation of sulfides in the tailings. If the cover technology is

effective, it can be projected that the treatment cells would yield less sulfate over time than the control cells. From observation of Figures 8-6 and 8-7, for leachate sulfate and cumulative sulfate, no significant differences between the treatment and control cells are readily apparent. Generally, leachate sulfate concentration in all the cells was initially high and then dropped quickly to a relatively steady value, or slightly decreasing level. The initial peak in leached sulfate from the test cells, which is common between all the cells, indicates that the initial sulfate content within the cells has been flushed from the tailings. The apparent stabilization of the leached sulfate, near the conclusion of the demonstration, may reflect the true rate of sulfate production. The rate (i.e., kinetics) of sulfide mineral oxidation may be too slow to cause significantly measurable differences between the treatment and control cells in leached sulfate during the timeframe of this demonstration. It cannot be concluded whether sulfate is retained preferentially in the treatment cell versus the control cell from the sulfate data obtained in this demonstration.

8.2.2.4 Specific Conductance

Specific conductance results are shown in Figure 8-8. Consistent with expectations, the test cell leachate SC started out relatively high and decreased gradually during the demonstration. Initially, the soluble salts were washed from the tailings, followed by the slightly soluble salts, which was reflected by the decrease in leachate SC. A significant secondary rise in conductance was not observed. It was projected that oxidation of sulfide in the tailings in the control cells would generate more soluble salts; hence, a rise in the SC. Throughout the demonstration, the treatment cells' conductance remained higher than the control cells. This may be partially explained by dilution effects. If similar amounts of dissolved salts were leached from all the test cells, cells with greater leachate production (i.e., control cells) would have a lower SC when compared to the treatment cells, producing less leachate.

8.2.2.5 Total Metals

Total metals summary is shown in Table 8-1. There does not appear to be any significant differences in metals concentration trends between the control and treatment cells. Distinct observations that can be made about all of the test cells involve calcium, iron, and sodium. Throughout the demonstration, the calcium concentration was constant, the iron concentration increased, and the sodium concentration decreased within each test cell. The mechanisms behind the concentration changes are likely independent of each other. Leaching of soluble salts over time will cause the decrease in sodium. The increase in the iron leachate concentration may be due to oxidation of the sulfides in the tailings. The data from the October 6, 2004 samples was biased because tank water was siphoned back into the wells. This event correlated with an anomalously high pH in the leachate, which impacted the leachate by causing the iron to be precipitated. Calcium was remarkably constant suggesting that the calcium solubility in the leachate may be controlled by a stable mineral phase.

8.2.3 Irrigation Water

Several parameters were monitored in the irrigation water that was used to augment the meteoric water during the demonstration. The field and laboratory results are available in Appendix A. The irrigation water physical and chemical parameters were relatively consistent.

8.3 Post-Demonstration Measurements

Post-demonstration data was acquired from three separate cores taken at random locations from each test cell. There were no differences in coloration between control and treatment cell samples to provide any visual indication of oxidation in the cells. If oxygen penetration into the test cells is limited, any chemical changes resulting from oxidation would occur near the surface of the cell, and the effects should be

detectable in the upper portion of the core. Samples from the top of the core would have highest probability for showing the effects of oxidation; therefore, only the top 3 inches of each of the 1-inch cores was submitted for analysis to the MSE Laboratory (formerly HKM Laboratory). A larger sample would normalize the analysis if oxidation were limited to the surface of the test cell.

8.3.1 Acid-Base Accounting

ABA analysis of the core samples determined the amount and form of the sulfur remaining in the test tailings at the completion of the demonstration. There were significant differences between the treatment and control cell post-test ABA results. The post-test ABA laboratory analytical data is presented in Table 8-2. The results between cores taken from the same test cells show good precision, as well as good agreement between treatments. Total sulfur in the treatment cells was approximately 4.5%, with about 88% being unoxidized pyritic sulfur. In contrast, the total sulfur remaining in the control cells was only 0.04% to 0.05%. This is possibly an indication that the electrochemical cover used in the treatment cells was effective in inhibiting oxidation when compared to the control cells.

8.3.2 1:1 pH

Due to the limited sample size submitted, 1:1 pH was determined in lieu of a saturated paste pH. The 1:1 pH results are also shown in Table 8-2. The control cell tailings react significantly different from the treatment cell tailings when mixed with deionized water. The 1:1 pH of the control cells was around 8, while the 1:1 pH of the treatment cells was approximately 5.6. The lower relative 1:1 pH from treatment cells may have been caused by the oxidation/hydrolysis of the remaining pyritic sulfur or the development of acidic, secondary phases in the treatment cells during the sample preparation process.

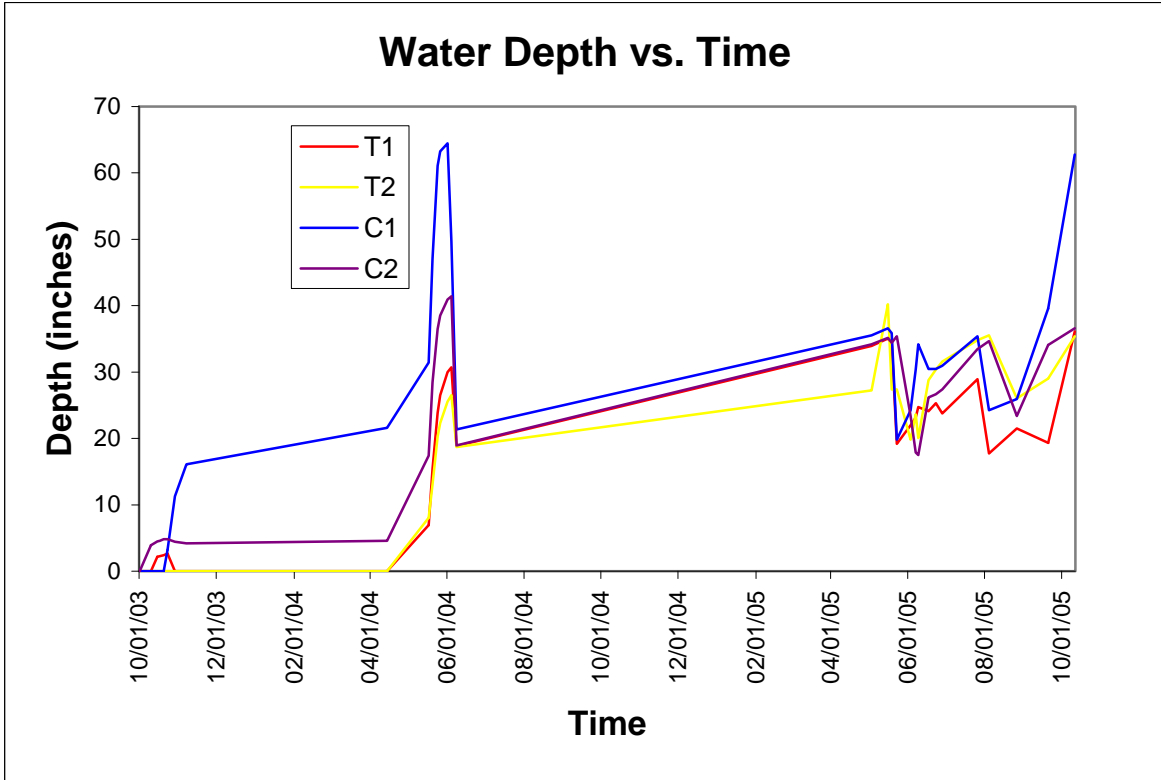


Figure 8-1. Water depth in the test cells over time.

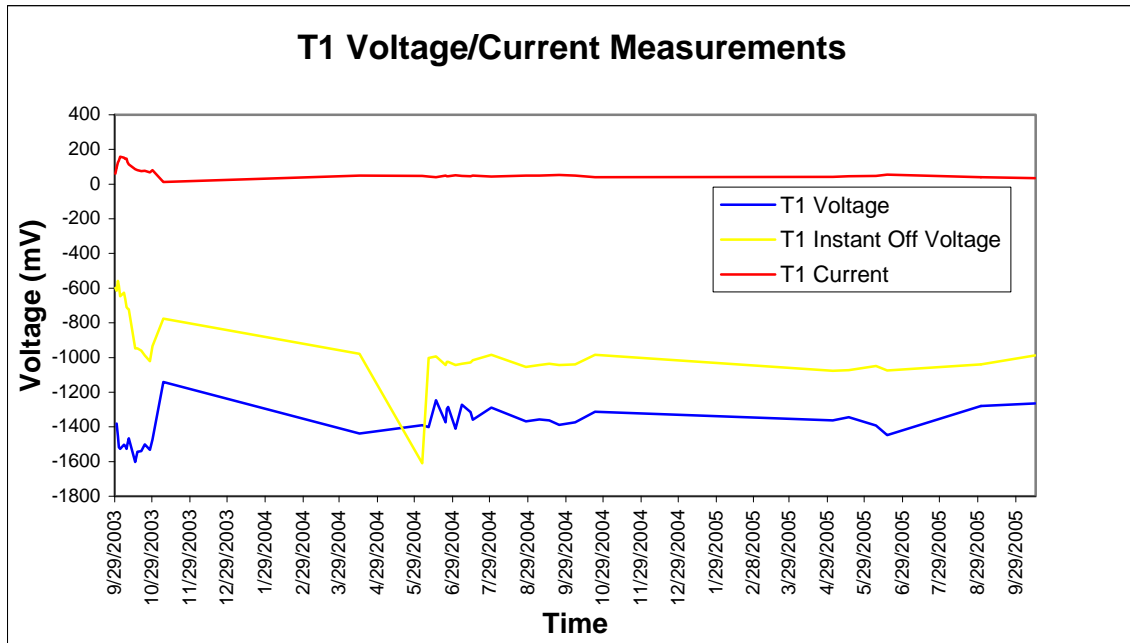


Figure 8-2. Treatment cell T1 voltage and current measurements.

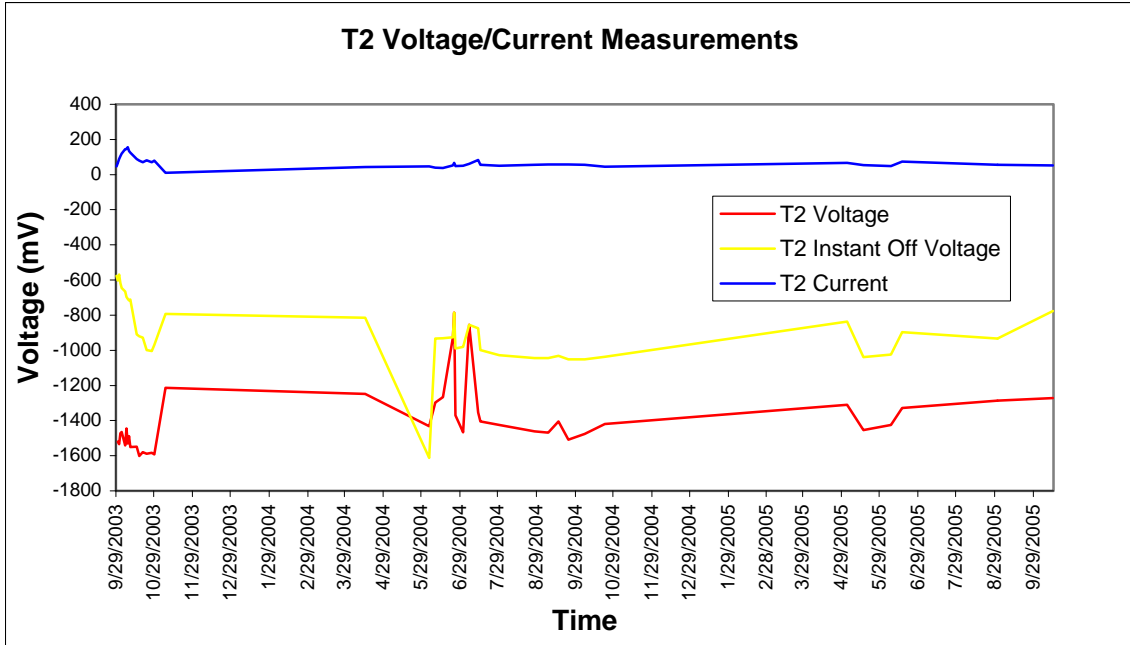


Figure 8-3. Treatment cell T2 voltage and current measurements.

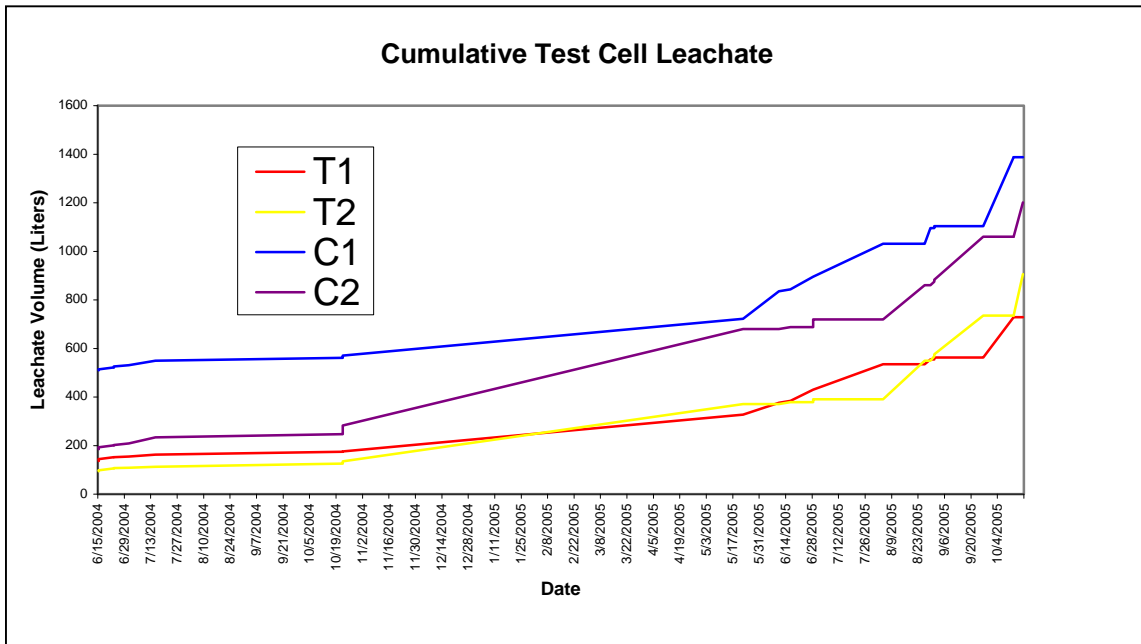


Figure 8-4. Test cell leachate production.

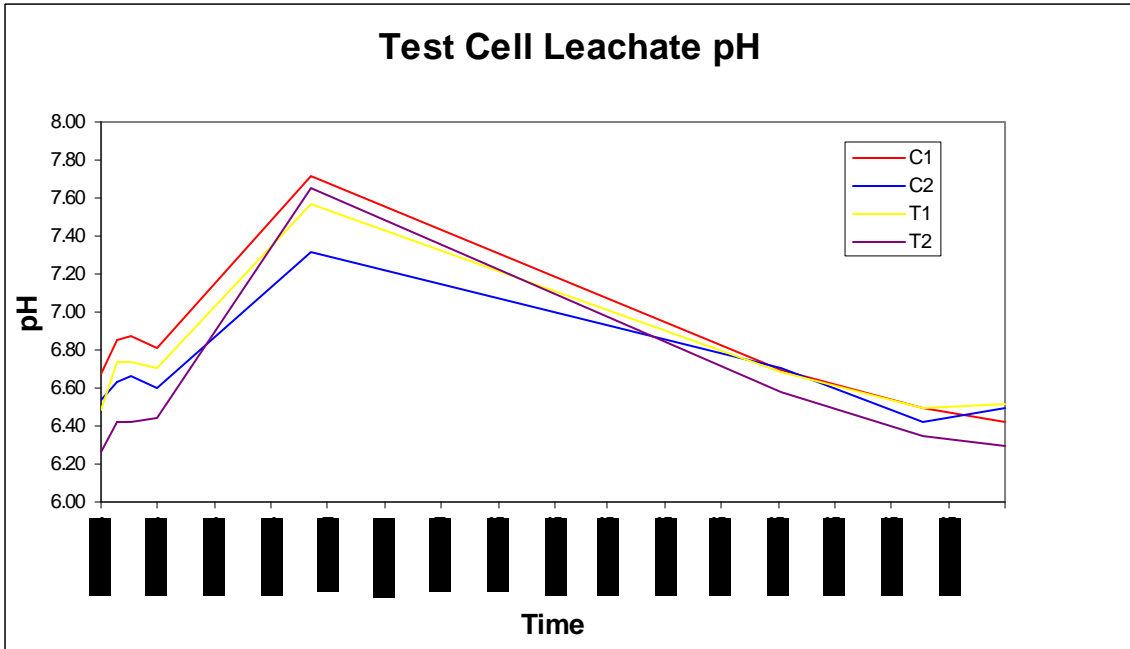


Figure 8-5. Test cell leachate pH.

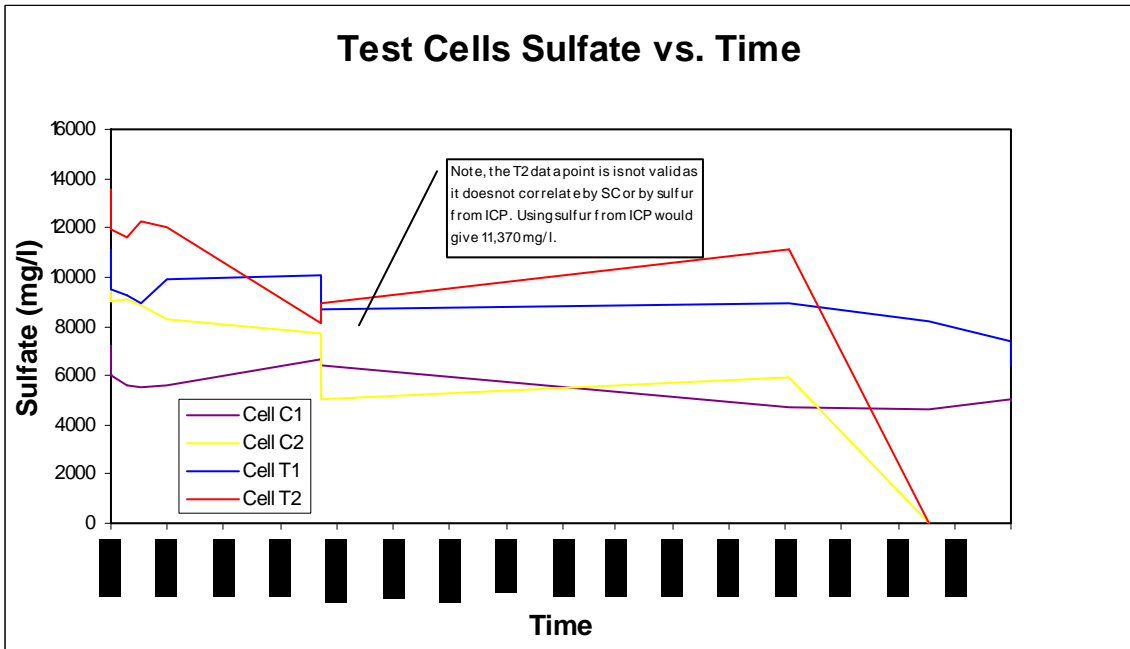


Figure 8-6. Test cell leachate sulfate.

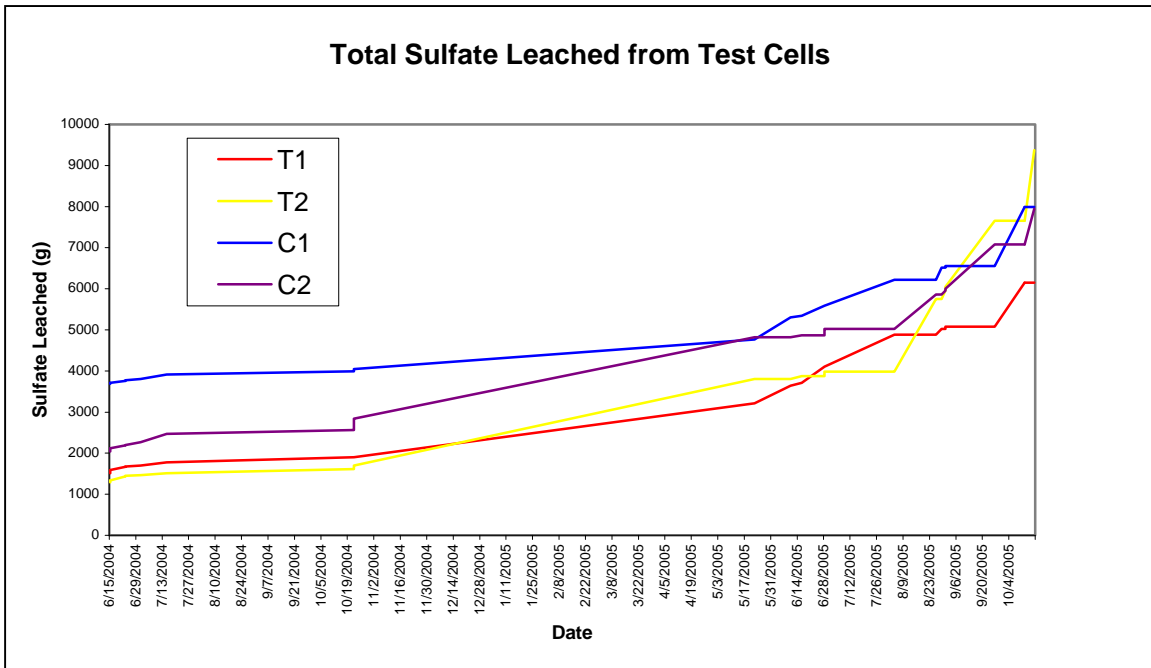


Figure 8-7. Total sulfate leached from the test cells.

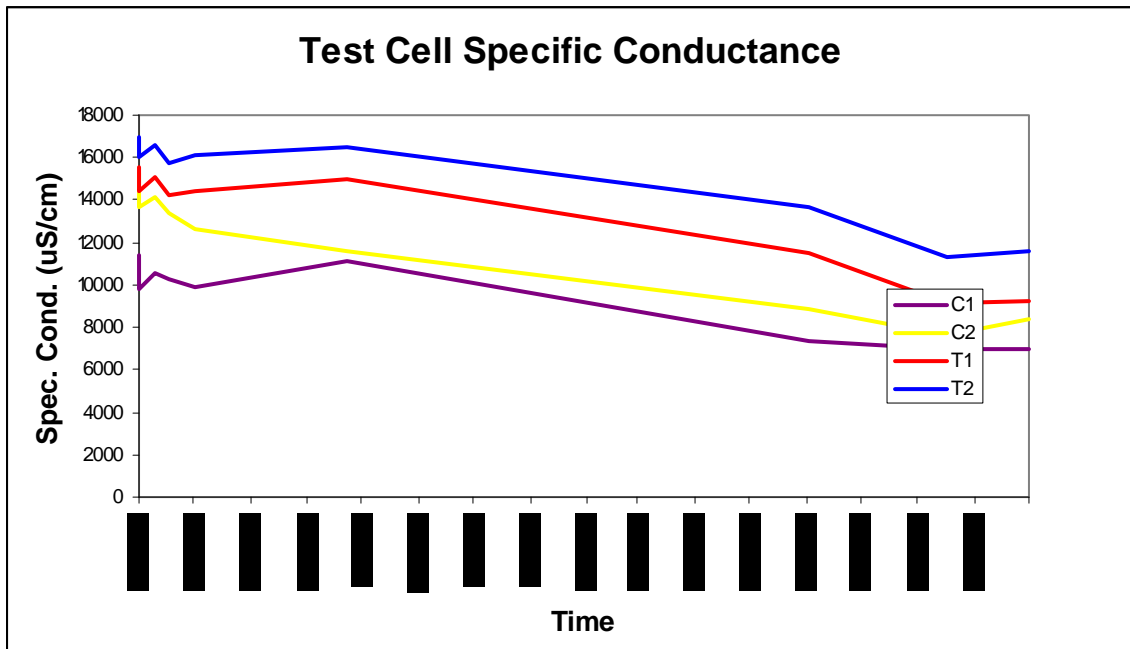


Figure 8-8. Leachate SC.

Table 8-1. Total Metals in Test Cell Leachate

Test Cell ID	FIELD ID	Date Sampled	Ca (mg/L)	Fe (mg/L)	Mg (mg/L)	Mn (mg/L)	K (mg/L)	Si (mg/L)	Na (mg/L)	S (mg/L)
C1	ENPAR-C1-061504	6/15/2004	419	6.66	436	7.09	364	6.31	959	NA
C1	ENPAR-C1-071504	7/15/2004	386	8.79	420	7.36	346	6.52	895	1,790
C1	ENPAR-C1-100604	10/6/2004	372	0.44	502	5.26	397	7.90	1,150	2,100
C1	ENPAR-C1-061605	6/16/2005	403	15.8	381	6.61	284	6.74	554	1,410
C1	ENPAR-C1-083105	8/31/2005	436	22.7	613	10.8	239	8.29	348	1,520
C1	ENPAR-C1-101405	10/14/2005	464	18.4	782	14.2	211	8.46	226	1,700
C2	ENPAR-C2-061504	6/15/2004	390	3.18	1,030	12.3	455	6.49	1,310	NA
C2	ENPAR-C2-071504	7/15/2004	348	3.03	884	11.8	386	6.38	1,040	2,630
C2	ENPAR-C2-100604	10/6/2004	344	0.19	850	10.0	379	7.39	869	2,370
C2	ENPAR-C2-061605	6/16/2005	373	7.40	739	8.00	306	6.63	617	1,970
C2	ENPAR-C2-083105	8/31/2005	415	9.73	843	12.4	229	8.16	356	1,830
C2	ENPAR-C2-101405	10/14/2005	416	12.4	886	13.3	280	8.28	447	1,980
T1	ENPAR-T1-061504	6/15/2004	385	3.68	918	8.16	465	6.15	1,670	NA
T1	ENPAR-T1-071504	7/15/2004	348	4.22	863	7.81	422	6.12	1,540	2,980
T1	ENPAR-T1-100604	10/6/2004	316	1.71	907	5.31	426	6.37	1,750	3,130
T1	ENPAR-T1-061605	6/16/2005	349	6.85	813	6.11	384	6.03	1,070	2,490
T1	ENPAR-T1-083105	8/31/2005	396	12.1	933	11.3	319	7.64	610	2,200
T1	ENPAR-T1-101405	10/14/2005	394	13.7	871	9.81	322	7.97	527	2,040
T2	ENPAR-T2-061504	6/15/2004	366	2.45	1,410	19.6	497	6.33	1,400	NA
T2	ENPAR-T2-071504	7/15/2004	327	2.56	1,400	18.5	450	6.22	1,540	3,750
T2	ENPAR-T2-100604	10/6/2004	298	0.53	1,260	13.6	446	6.43	1,740	3,790
T2	ENPAR-T2-061605	6/16/2005	336	6.71	1,210	12.8	439	6.60	1,350	3,320
T2	ENPAR-T2-083105	8/31/2005	384	8.81	1,370	17.7	358	7.63	765	3,040
T2	ENPAR-T2-101405	10/14/2005	386	11.2	1,430	18.1	364	8.69	644	3,030

NA – parameter not measured

Table 8-2. Post-Test Acid-Base Accounting and 1:1 pH Results

Test Cell ID	Client Sample ID	Pyritic Sulfur -		Insoluble	Sulfate -	Residual	Neutralization Potential (t CaCO ₃ /1000t)	Maximum Potential Acidity (t/1000t)	Net Neutralization Potential (t/1000t)	pH - 1:1 (SU)
		Total Sulfur (%)	HNO ₃ Extractable (%)	Sulfide - HCl Extractable (%)	Water Soluble (%)	Sulfur - Non-Extractable (%)				
C1	ENPAR-C1-1-120506	0.04	0.02	0.01	0.01 U	0.01 U	73.0	1.3	72	7.9
	ENPAR-C1-2-120506	0.06	0.04	0.01 U	0.01 U	0.01	100.0	2.0	100	8.0
	ENPAR-C1-3-120506	0.05	0.03	0.01 U	0.01	0.01 U	95.0	1.4	93	8.1
	Average	0.05	0.03	0.01	0.01	0.01	89.3	1.6	88.3	8.0
	Standard Deviation	0.01	0.01				14.4		14.6	0.1
	Rel. Std. Dev.	20.0	33.3				16.1		16.5	1.2
C2	ENPAR-C2-1-120506	0.04	0.01	0.02	0.01 U	0.01 U	120.0	1.2	120	8.1
	ENPAR-C2-2-120506	0.04	0.03	0.01 U	0.01 U	0.01 U	120.0	1.1	120	8.0
	ENPAR-C2-3-120506	0.04	0.02	0.01 U	0.01	0.01 U	110.0	1.2	110	8.2
	Average	0.04	0.02	0.01	0.01	0.01	116.7	1.2	117	8.1
	Standard Deviation	0.00	0.01				5.8		5.8	0.1
	Rel. Std. Dev.	0.0	50.0				4.9		4.9	1.2
T1	ENPAR-T1-1-120506	4.69	4.32	0.06	0.19	0.12	3.8	147	-140	5.7
	ENPAR-T1-2-120506	4.34	3.77	0.15	0.29	0.14	7.6	136	-130	5.5
	ENPAR-T1-3-120506	4.50	3.84	0.27	0.25	0.14	1.3	141	-140	5.6
	Average	4.51	3.98	0.16	0.24	0.13	4.2	141.3	-137	5.6
	Standard Deviation	0.18	0.30				3.2		5.8	0.1
	Rel. Std. Dev.	3.9	7.5				74.9		-4.2	1.8
T2	ENPAR-T2-1-120506	4.35	3.99	0.01 U	0.32	0.06	5.0	136	-130	5.9
	ENPAR-T2-2-120506	4.60	4.16	0.07	0.29	0.09	8.2	144	-140	5.5
	ENPAR-T2-3-120506	4.38	3.88	0.01 U	0.43	0.13	8.8	137	-130	5.6
	Average	4.44	4.01	0.03	0.35	0.09	7.3	139.0	-133	5.7
	Standard Deviation	0.14	0.14				2.0		5.8	0.2
	Rel. Std. Dev.	3.1	3.5				27.9		-4.3	3.7

9. Conclusions and Recommendations

The MWTP, Activity III, Project 40, *Electrochemical Tailings Cover* demonstration, has provided some evidence that the electrochemical tailings cover can reduce the oxidation of sulfide minerals in sulfide-containing mine waste. The reduction in oxidation of sulfur in the tailings was best shown by the post-test ABA analysis. The electrochemically treated cells retain total sulfur and pyritic sulfur at higher levels than in the control cells that had no special treatment. In fact, treatment cell T2 retained over 90% of its original sulfur content. Nearly 50% of the sulfur was retained in the other treatment cell, T1; however, there was a large degree of variation in the initial sulfur data for this treatment cell, which seemed somewhat suspect due to initial high total sulfur content when compared to the three other test cells. Interestingly, both cells with the electrochemical treatment retained about 4.5% total sulfur while the untreated cells contained less than 0.05% sulfur at the conclusion of the demonstration. It is apparent that the sulfur was readily oxidized and leached away from the top few inches of tailings in the untreated control cells, since initial total sulfur in the cells ranged from 4.78 to 9.44%.

Disappointingly, the leachate water samples failed to provide any real conclusive evidence about the effectiveness of the electrochemical tailings cover. No significant trends in leachate sulfate, total sulfate, or pH could be denoted. The oxidation rate of the sulfur in the tailings may be responsible for the inconclusive findings from the leachate sulfate and pH. The duration of the demonstration may have been of insufficient length for the oxidation of sulfide to dominate the leachate chemistry.

Other factors may have influenced the chemistry of the leachate as it migrated downward through the tailings on its path to collection in the base of the test cells. The calcium and sulfate concentrations remained relatively constant in the leachate throughout the demonstration. This

indicates that their concentration in the leachate may have been controlled by a stable mineral phase in the sediment. It is quite likely that gypsum, $\text{CaSO}_4 + 2\text{H}_2\text{O}$, may be responsible for the influence on the calcium and sulfate, since both are present at considerable concentrations in the leachate. If indeed gypsum was controlling the solubility of calcium and sulfate, any sulfate arising from oxidation of sulfide in the tailings would be attenuated before it reaches the reservoir of water at the base of the test cells; thus, obscuring the sulfate data as a useful indicator of sulfide oxidation.

9.1 Lessons Learned

When planning any testing demonstration, especially when it entails a large-scale demonstration, it is highly important to attempt to understand the entire system and potential effects on the analytical data to be collected. In this demonstration, it was expected that leachate sulfate and pH would provide the data necessary to evaluate the effectiveness of the electrochemical tailings cover. However, the influence of the tailings and stability of mineral phases may have confounded the results from these two parameters. Fortunately, several types of data were collected, and the ABA analysis provided some data that allowed an alternative for evaluation of the electrochemical cover technology.

Quantification of the amount of sulfate being leached from the cells was difficult in the experiment. An alternative leachate collection format may have improved the accuracy of determining the amount of sulfur, and other constituents leached from the tailings. Dedicated collection barrels for each cell would provide accurate volume determinations and a reservoir from which to sample for sulfate and other analytes of concern. Samples from the collection reservoir would provide a more accurate determination of the leachate constituents.

10. References

1. MSE Technology Applications, Inc., Mine Waste Technology Program, *Quality Assurance Project Plan – Electrochemical Tailings Cover*, Activity III, Project 40, October 2003.

Appendix A

Laboratory and Field Data

Metals in Test Cell Tailings

Results per dry weight basis

SAMPLE ID	FIELD ID	Test Cell ID	Al (mg/Kg)	As (mg/Kg)	Cd (mg/Kg)	Ca (mg/Kg)	Cu (mg/Kg)	Fe (mg/Kg)	Pb (mg/Kg)	Mg (mg/Kg)	Mn (mg/Kg)	K (mg/Kg)	Si (mg/Kg)	Na (mg/Kg)	Zn (mg/Kg)
CRDL			40.0	14.4	2.7	1,000	5.0	20.0	23.4	1,000	3	1000	5.98	1000	4
IDL	ICP-AES		6.22	7.2	1.354	1.48	0.28	1.8	11.7	10.84	0.6	4.22	2	0.8	1.3
0310200041	ENPAR-C1-TAILSCOMPOSITE-0925	C1	820	29.1	1.3 U	1,880	133	43,000	16.1 B	4,640	77.9	1,010	575	237 B	70.7
0310200042	ENPAR-C2-TAILSCOMPOSITE-0925	C2	829	32.4	1.3 U	2,400	153	46,600	14.5 B	5,270	96.3	1,030	648	263 B	94.7
0310200043	ENPAR-T1-TAILSCOMPOSITE-0925	T1	801	32.5	1.3 U	2,310	167	46,600	12.3 B	5,000	91.8	993	637	260 B	94.0
0310200044	ENPAR-T2-TAILSCOMPOSITE-0925	T2	804	33.9	1.3 U	2,150	141	42,700	18.1 B	4,780	83.4	969	498	245 B	86.4
Inter-Cellular Statistics															
	Average		814	32.0	1.3 U	2,185	149	44,725	15.3 B	4,923	87.4	1,001	589.5	251	86.5
	Standard Deviation		13.3	2.0	N/A	228	14.8	2169	2.5	275	8.3	25.9	68.9	12.3	11.2
	Relative Standard Deviation (RSD)		1.6%	6.4%	N/A	10.4%	10.0%	4.8%	16.1%	5.6%	9.5%	2.6%	11.7%	4.9%	12.9%

Legend:

- IDL - Instrument Detection Limit
- CRDL - Contract Required Detection Limit
- U - analyte undetected
- B - analyte detected, less than CRDL

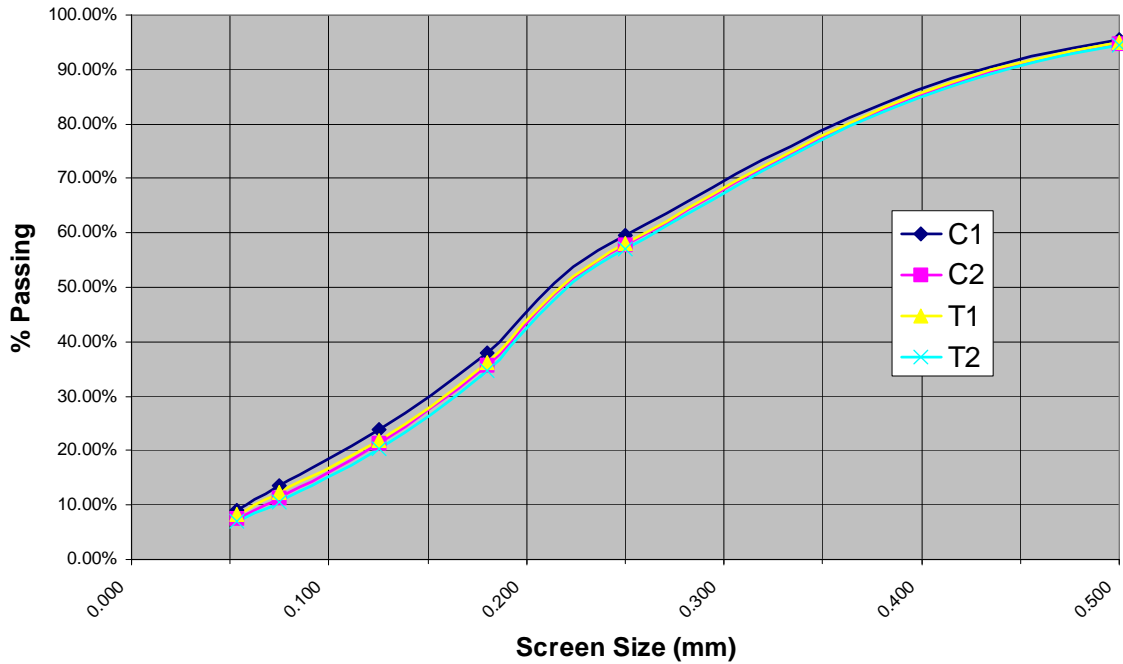
Test Cell Tailings Acid-Base Accounting Results

Fraction	Total Sulfur (%)	Pyritic Sulfur (%)	Soluble Sulfur (%)	Insoluble Sulfur (%)	Organic Sulfur (%)	Neutralization Potential (t/100t)
Method of Extraction	None	Hot HNO ₃ Leach	Hot Water Leach	HCl Leach	Residual	
Cell - C1						
Avg.	5.40	4.45	0.37	0.10	0.51	14.9
Std. Dev.	0.64	0.61	0.12	0.07	0.16	1.9
RSD	11.9	13.8	31.8	69.9	30.7	12.5
Cell - C2						
Avg.	6.07	4.88	0.37	0.25	0.67	19.6
Std. Dev.	0.66	0.65	0.12	0.19	0.13	3.6
RSD	10.9	13.3	32.5	76.4	19.7	18.5
Cell - T1						
Avg.	9.44	7.83	0.62	0.33	0.67	18.4
Std. Dev.	1.40	1.52	0.43	0.27	0.17	1.6
RSD	14.8	19.5	70.6	84.2	25.8	8.9
Cell - T2						
Avg.	4.78	3.67	0.40	0.15	0.58	25.3
Std. Dev.	0.23	0.33	0.20	0.13	0.17	2.0
RSD	4.7	8.9	50.6	82.8	29.8	8.1
Overall averages						
	6.42	5.21	0.44	0.21	0.61	19.6

Test Cell Soil & Sand Acid-Base Accounting Results

Fraction	Total Sulfur (%)	Pyritic Sulfur (%)	Soluble Sulfur (%)	Insoluble Sulfur (%)	Organic Sulfur (%)	Neutralization Potential (t/100t)
Method of Extraction	None	Hot HNO ₃ Leach	Hot Water Leach	HCl Leach	Residual	
Cell - C1						
Soil	0.02	0.02	<0.01	<0.01	<0.01	135.4
Sand	<0.01	<0.01	<0.01	0.02	<0.01	<1.0
Cell - C2						
Soil	0.05	0.04	<0.01	0.01	<0.01	146.0
Sand	0.01	0.01	0.01	<0.01	<0.01	3.7
Cell - T1						
Soil	0.03	0.01	0.02	<0.01	<0.01	143.2
Sand	0.01	<0.01	<0.01	0.01	<0.01	<1.0
Cell - T2						
Soil	0.03	0.02	0.02	<0.01	<0.01	157.2
Sand	0.01	<0.01	0.01	<0.01	<0.01	9.4

Test Cell Tailings Sieve Analysis



Test Cell Tailings Saturated Paste pH

SAMPLE ID	FIELD ID	Test Cell ID	Date Collected	pH (SU)
0310200041	ENPAR-C1-TAILSCOMPOSITE-0925	C1	9/25/2003	7.0
0310200042	ENPAR-C2-TAILSCOMPOSITE-0925	C2	9/25/2003	7.1
0310200043	ENPAR-T1-TAILSCOMPOSITE-0925	T1	9/25/2003	7.2
0310200044	ENPAR-T2-TAILSCOMPOSITE-0925	T2	9/25/2003	7.1
Inter-Cellular Statistics				
	Average			7.1
	Standard Deviation			0.1
	Relative Standard Deviation (RSD)			1.1%

Irrigation Water (Plant Water) Chemical Analysis

FIELD ID	Date Collected	Dissolved Metals (ug/L)														Alkalinity (mg CaCO ₃ /L)	Cl (mg/L)	Cl ₂ Res. (mg/L)	Sulfate (mg/L)			
		Al	As	Cd	Ca	Cu	Fe	Pb	Mg	Mn	P	K	Si	Na	S					Zn		
ENPAR-PLANTWATER	10/27/2003	52.5 B	0.50 U	0.06 U	13900	13.3 B	242	0.94 U	5270	273	NR	2000 B	5785	3330 B	NR	7.4 B	36		<	0.05	27	
ENPAR-PLANTWATER 060304	6/3/2004	33.5 U	0.5 U	0.06 U	14800	20.4 B	198.0	0.9 U	5870	42.2	NR	2140 B	6767	3380 B	NR	8 U	41				0.15	28
ENPAR-PLANTWATER-071504	7/15/2004	41.7 B	29.9 U	8.1 U	12900	31.8	142	44.6 U	5120	32.3	NR	2030 B	6740	3150 B	8250	21.6	38	<	5	<	0.05	29
ENPAR-PLANTWATER-100604	10/6/2004	41.7 B	29.9 U	8.1 U	11700	59.8	289	44.6 U	4710 B	48.2	NR	1920 B	6610	3070 B	8150	10.7 B	30		NR	<	2	NR
ENPAR-PLANTWATER-061605	6/16/2005	40.6 U	24.6 U	9.1 U	14000	60	736	52.3 U	5630	78.1	NR	2220 B	6740	3320 B	7720	92.3	42	<	5	<	2	25
ENPAR-PLANTWATER-083105	8/31/2005	40.6 U	24.6 U	9.1 U	14500	115	195	52.3 U	5280	41.1	34.9 U	2080 B	7470	3670 B	6760	9.07 B	47	<	5		0.26	30
ENPAR-PLANTWATER-101405	10/14/2005	40.6 U	24.6 U	9.1 U	17000	58.8	439	52.3 U	5930	81.1	34.9 U	2270 B	7330	4030 B	7650	10.4 B	51	<	5		0.05	29

NR - parameter not requested

Appendix B

Data Collection Schedule During Field Installation and Monitoring and After Test Completion

Table B-1: Data Collected During Field Installation

Location	Parameter	Classification	Frequency	Matrix	Field Measurement or Laboratory Analysis
Test cells C1, C2, T1, T2 Tailings	Size Distribution	Nonprimary	One composite sample each cell	Solid	Laboratory
Test cells C1, C2, T1, T2 Tailings	Acid-Base Accounting	Primary	Seven composite samples each cell, one split duplicate each cell	Solid	Laboratory
Test cells C1, C2, T1, T2 Tailings	Paste pH	Nonprimary	One composite sample each cell	Solid	Laboratory
Test cells C1, C2, T1, T2 Tailings	Total Metals Analysis	Nonprimary	One composite sample each cell	Solid	Laboratory
Test cells C1, C2, T1, T2 Tailings	Mineralogy	Nonprimary	One composite sample each cell	Solid	Laboratory
Soil cover stockpile	Acid-Base Accounting	Nonprimary	One random grab sample	Solid	Laboratory
Quartz sand	Acid-Base Accounting	Nonprimary	One random grab sample	Solid	Laboratory
<p>Notes:</p> <ol style="list-style-type: none"> 1. The composite sample for size distribution, paste pH, total metals, and mineralogy for each cell will be obtained by taking one scoop of material from each “Bobcat” bucket being added to each cell, and placing it in a 5-gallon bucket. 2. The composite samples for acid-base accounting for each cell will be obtained as follows: After hand distribution of approximately each vertical foot of tailings, one scoop of material will be obtained from each quadrant of the cell. Since the tailings depth will be approximately 7.5 feet, this will provide seven composite samples each cell, each representing a vertical foot. For the top vertical foot, this will be performed twice to provide a field duplicate for each cell. 					

Table B-2: Data Collected During Monitoring Period

Location	Parameter	Classification	Frequency	Matrix	Field Measurement or Laboratory Analysis
Test cells T1, T2	Galvanic voltage	Nonprimary	Monthly	--	Field
	Galvanic current	Nonprimary	Monthly	--	Field
Irrigation water	pH	Nonprimary	Monthly	Aqueous	Field
	Specific conductance	Nonprimary	Monthly	Aqueous	Field
	ORP	Nonprimary	Monthly	Aqueous	Field
	Dissolved oxygen	Nonprimary	Monthly	Aqueous	Field
	Dissolved metals	Nonprimary	Bimonthly	Aqueous	Lab
	Alkalinity forms	Nonprimary	Monthly	Aqueous	Lab
	Chlorine	Nonprimary	Monthly	Aqueous	Lab
	Sulfate	Nonprimary	Monthly	Aqueous	Lab
Test cells C1, C2, T1, T2 leachate	Water Volume	Primary	Weekly	Aqueous	Field
	pH	Primary	Weekly	Aqueous	Field
	Sulfate	Primary	Weekly, one duplicate each sampling event	Aqueous	Lab
	Specific conductance	Nonprimary	Weekly	Aqueous	Field
	Total metals	Nonprimary	Monthly	Aqueous	Lab
Notes:					
1. Samples of plant service water and test cells leachate will be obtained only during time periods when irrigation water is being applied (approximately October 2003, then April through September 2004).					
2. Metals analyte list: (Al, As, Cd, Cu, Ca, Fe, Mg, Mn, Pb, Si, Na, K, Zn)					

Table B-3: Data Collected After Test Completion

Location	Parameter	Classification	Frequency	Matrix	Field Measurement or Laboratory Analysis
Test cells C1, C2, T1, T2	Acid-Base Accounting	Nonprimary	Three cores each cell	Solid	Lab
Test cells C1, C2, T1, T2	Paste pH	Nonprimary	Three cores each cell	Solid	Lab
Test cells C1, C2, T1, T2	Mineralogy	Nonprimary	Three cores each cell	Solid	Lab

Appendix C

Quality Assurance Activities

Quality Assurance and Control Activities

Mine Waste Technology Program Activity III, Project 40 (Electrochemical Tailings Cover)

C.1 REVIEW OF LABORATORY QUALIFICATIONS

The HKM Laboratory performed all the analysis for MWTP Activity III, Project 40. The laboratory is routinely audited by the Montana Department of Public Health & Human Services (MDPHHS) for adherence to appropriate methods and required quality control for the analysis of drinking water in the state of Montana. Additionally, the laboratory must analyze two sets of performance evaluation samples each year to maintain the MDPHHS drinking water certification.

Primary laboratory parameters for the demonstration are leachate sulfate, pH, and ABA of solid samples. HKM Laboratory is certified by the MDPHHS for the analysis of sulfate and pH in drinking water; thus, demonstrating their ability to satisfactorily perform the analysis for this project. There is no certification program in the state of Montana for the analysis of ABA parameters; however, the laboratory routinely analyzes blanks, control samples, and duplicates in performing ABA analysis. The control standard, KZK-1, used for checking the accuracy of the ABA method is a certified reference material for ABA analysis by CANMET-MMSL in Ottawa, Ontario, Canada.

C.1.1 Performance Evaluation Samples

The HKM Laboratory is required to analyze performance evaluation samples twice annually to maintain its drinking water certification by the MDPHHS. The HKM Laboratory achieved satisfactory results on the performance evaluation for sulfate in WS-86 Study from Environmental Resource Associates conducted from September 15, 2003 to October 30, 2003, which was at the beginning of MWTP Activity III, Project 40. The laboratory result for sulfate in the unknown sample was 27 mg/L, which was within the acceptance range of 21.9 to 27.6 mg/L.

There are no performance evaluation studies for ABA analyses; however, strict analytical protocol is followed, which consists of the QA samples previously mentioned in this section.

C.2 FIELD AUDITS

One field audit was performed during the setup of the experimental cell installation. Ken Reick, a quality assurance officer for MSE, performed the technical systems review on September 29, 2003. The scope of the audit was to monitor compliance of loading tailings into the study cells with procedures outlined in the QAPP. There were no findings reported, but two observations were noted:

- The first observation noted that one of the cells was nearly completely loaded without following the alternating 1-foot loading scheme outlined in the QAPP, where 1 foot of tailings would be loaded into a cell and then move onto the next cell and load 1 foot of tailings and sequentially move on to the next cell until the cells contained 7 feet of uncompacted tailings. Since the tailings were considered relatively homogeneous, this was not considered a finding.

- The second observation related to the fact that no permeability testing was performed on the tailings used in loading the test cells. Since no permeability information was available or determined, it could not be ascertained when the cells would begin to show an accumulation of leachate water in the base of the study cell. Obtaining such information would only allow some speculation as to when water would appear in the test cell lysimeters, and provides no absolute basis for appearance of leach water. Additionally, other tests such as water holding capacity would be needed to properly elucidate such a prediction. The extent of such analysis is beyond the scope of this project and was given no further consideration.

C.3 FIELD AND LABORATORY DATA VALIDATION

The stated objective in the QAPP was to inhibit oxidation of sulfides in sulfide-containing tailings to minimize consequential AMD. All of the field and laboratory data was collected between September 2003 and October 2005, with the exception of the post-test ABA and 1:1 pH samples which were submitted for analysis in October of 2006.

The effectiveness of the electrochemical tailings cover was evaluated using the results from the installation and post-test ABA tailings analysis, leachate field measurements, and sulfate and metals analysis from the monitoring phase. The analyses were specified in the QAPP, and each analysis was classified as critical or noncritical. A critical analysis is an analysis that must be performed to achieve project objectives. A noncritical analysis is an analysis that is performed to provide additional information about the demonstration. Critical analyses for this project are summarized below.

- Installation
 - tailings ABA
- Monitoring
 - leachate pH
 - leachate sulfate
 - leachate volume

Noncritical analyses for this project are summarized below.

- Installation
 - size distribution
 - total metals
 - paste pH
 - soils ABA
 - quartz sand ABA
- Monitoring
 - treatment cell
 - galvanic voltage
 - galvanic current
 - irrigation water
 - pH

- SC
- ORP
- dissolved oxygen
- dissolved metals
- alkalinity (forms)
- chlorine
- sulfate
- test cell leachate
 - SC
 - total metals
- Test Completion
 - test cell
 - ABA
 - paste pH

The QA objectives for each of the critical and noncritical analysis were outlined in the QAPP and were compatible with project objectives and methods of determination being used. The QA objectives are accuracy, precision, completeness, and method detection limits (MDL). Requirements for each of these objectives were established in the QAPP. The usability of the data was determined by compliance of the data with the stated QAPP QA requirements.

C.3.1 Validation Procedures

Data generated for all critical and noncritical analyses was validated. The purpose of data validation is to determine the usability of project data. Data validation consists of two separate evaluations: analytical evaluation and program evaluation.

C.3.1.1 Criteria for Analytical Evaluation

Analytical evaluation was performed to determine:

- all analyses were performed within specified holding times;
- calibration procedures were followed correctly by field and laboratory personnel;
- laboratory analytical blanks contain no significant contamination;
- all necessary independent check standards were prepared and analyzed at the proper frequency and all remained within control limits;
- duplicate sample analysis was performed at the proper frequency and all relative percent differences (RPDs) were within specific control limits;
- matrix spike sample analysis was performed at the proper frequency and all spike recoveries (%R) were within specified control limits; and
- the data in the report submitted by the laboratory could be verified from the raw data generated by the laboratory.

Measurements that fall outside of the control limits specified in the QAPP or for other reasons are judged to be outliers, and were flagged appropriately to indicate the data is judged to be estimated or unusable. All QC outliers for all samples are summarized in Table 8-1. In addition to the analytical evaluation, a program evaluation was performed.

C.3.1.2 Criteria for Program Evaluation

Program evaluations include an examination of data generated during the project to determine:

- all information contained in the COC forms is consistent with the sample information in field logs, laboratory raw data, and laboratory reports;
- all samples, including field QC samples, were collected, sent to the appropriate laboratory for analysis, analyzed, and reported by the laboratory for the appropriate analyses;
- all field blanks contained no significant contamination; and
- all field duplicate samples demonstrate precision for field and laboratory procedures by remaining within the established RPD control limits.

Program data that was inconsistent or incomplete and did not meet the QC objectives outlined in the QAPP were viewed as outliers and were flagged appropriately to indicate the usability of the data. Both the analytical and program evaluations consisted of evaluating the data generated in the field as well as in the laboratory.

C.3.2 Analytical Evaluation

The analytical evaluation of field and laboratory data was done in December 2005. Analytical and field data were validated by the MSE Quality Assurance Officer (QAO), Michelle Lee.

C.3.2.1 Field Logbook Evaluation

Field data validation began with an examination of the field logbooks. All project information was recorded in two, bound all-weather transit-style 4.75-inch by 7-inch notebooks. Additional notes, SOPs, and field sheets were retained in a project-dedicated 3-inch, 3-ring binder.

Information about Fieldwork Performed

The general logbooks contained notes on fieldwork performed and process measurements. Voltage and current readings from the treatment cells were periodically recorded, to ensure that the technology in the treatment cells was properly functioning. Water level measurements from each test cell were sporadically recorded, but these measurements were only necessary to indicate that the artificial water table was established. Leachate volumes were difficult to correlate between the logbook records and the sulfate analytical data; however, data correlation was reconstructed based on the dates recorded.

C.3.2.2 Field Data Validation

Field data validation was performed to determine the usability of the data that was generated during field activities. Data usability was determined by verifying correct calibration procedures for field instruments were followed. In addition, the QC parameters of precision and accuracy calculated in the field were compared to those specified in the QAPP. Any data that fell outside of the control limits was considered outlier and was flagged appropriately. The measurements performed in the field were:

- amount of test cell leachate pumped (critical);
- test cell well depth (noncritical);
- leachate pH (critical);
- temperature (noncritical);
- ORP (noncritical);
- dissolved oxygen (noncritical); and

- SC (noncritical).

C.3.2.3 Sample Collection

Samples were collected by the project manager and engineer during the project. The technical systems review performed by Ken Reick in September 2003 during setup of the experimental installation, revealed no sample collection findings.

C.3.2.4 Sample Labeling

Samples were labeled in accordance with the scheme outlined in the QAPP. Labeling followed the described format ensuring that each sample was given a unique sample identification number.

C.3.2.5 Sample Packing and Transport

Due to the proximity of the test installation and the laboratory, samples were hand-delivered to the laboratory, typically within 24 hours of collection. However, in a couple instances, soil samples were delivered to the laboratory several days after collection, but the delay is considered inconsequential, since there is no technical holding time for soils analysis.

Amount of Test Cell Leachate Pumped

The amount of leachate pumped was measured by collection in graduated tanks and/or weighed. The data was not systematically recorded, making it difficult to correlate with the appropriate analytical sulfate results. Due to this shortcoming, the amount of leachate pumped was considered to be of screening use only.

pH

The pH meter was calibrated using two known buffer solutions. Typically, the buffer solutions would bracket the measured pH. The accuracy of the pH meter was verified by measuring a third known buffer within the calibration range. Accuracy was defined as the absolute difference between the measured and known buffer value. Calibration was performed each day pH measurements were taken. All pH data was considered usable.

Temperature

Temperature was recorded on the pumped leachate water samples and irrigation water (i.e., plant water) used to augment the natural precipitation. All temperatures were recorded in the project logbooks. All temperature data was considered usable.

Oxidation-Reduction Potential

ORP was performed on the leachate water and irrigation water samples. The accuracy of the ORP was verified by measuring Zobell's solution of a known ORP. The measured ORP was required to be within 20 mV of the temperature specified value for the Zobell's solution. All readings were within the required accuracy range so all ORP values were considered usable.

Dissolved Oxygen

Dissolved oxygen was measured on the leachate and plant water samples. The dissolved oxygen was calibrated each time the meter was used. There was no known standard solution used to verify the dissolved oxygen measurements. Since the meter was calibrated at the required frequency, all dissolved oxygen measurements were considered usable.

Specific Conductance

The SC was measured on leachate and plant water samples. The SC was calibrated daily as specified in the QAPP. Since the SC was calibrated as specified, all SC data was considered usable.

C.3.2.6 Laboratory Data Validation

Laboratory data validation was performed to determine the usability of the data that was generated by the laboratory for this project. The laboratory was reviewed and validated by the MSE QAO in December 2005, after the dismantling of the demonstration. The analyses performed by the HKM Laboratory were:

- ABA (critical);
- sulfate (critical);
- paste pH (noncritical);
- total and dissolved metals (noncritical);
- alkalinity, forms (noncritical);
- chlorine (noncritical); and
- sieve analysis (noncritical).

Laboratory validation was performed using the quality assurance objectives defined in the QAPP for the critical parameters ABA and sulfate. Evaluation of total and dissolved metals analysis was performed using the quality control requirements of the Contract Laboratory Program (CLP) Statement of Work (SOW) ILMO1.1.

Acid-Base Accounting

Acid-base accounting was performed according to the protocols in Method 3.2.6 from EPA-600/2-78-054. Laboratory and field duplicates were prepared at the frequency required in the QAPP. All of the ABA data was considered usable. Evaluation of the ABA data was based on compliance with duplicate precision, accuracy of laboratory control samples, and analyte levels in preparation blanks. In summary, there were no issues with accuracy or contamination. ABA data was flagged because of lack of precision in laboratory and field duplicates only. The duplicate (precision) control limits assigned in the QAPP are quite stringent; typically, the RPD limit of 35% is used to evaluate solid matrix samples. Table C-1 outlines the samples and the associated flags.

During analysis of the installation tailings samples, the HKM Laboratory encountered some difficulty in ABA analysis. This prompted the project manager to submit a batch of samples to a referee laboratory, Silver Valley Laboratory (SVL) for ABA analysis. A comparison of the ABA results is presented in Table C-2. The results compare reasonably well except for the values from treatment cell T1. HKM Laboratory had high total sulfur results from cell T1 compared to SVL results; however, duplicate analysis results from SVL displayed a large imprecision. It can only be concluded that cell T1 was not as homogeneous as the other test cells. Further scrutiny of the sulfur fractions revealed that the HKM Laboratory showed excellent agreement between the total sulfur and summation of the sulfur fractions; whereas, SVL displayed significant discrepancies between the total sulfur and corresponding fraction summations. The HCl extractable sulfur values from SVL were consistently higher than HKM, while the residual sulfur values from HKM were consistently higher than SVL results.

Leachate Sulfate

Sulfate analysis was performed by the HKM Laboratory. All sulfate data was considered usable. However, one sulfate value that appeared to be discrepant. The sulfate value for cell T2 sampled on October 6, 2004 seems to be low. The value does not correlate well with inductively coupled plasma

(ICP) determined sulfur values. Additionally, there is no corresponding decrease in the SC, as would be expected for such a dramatic drop in the sulfate concentration. All field duplicates complied with the precision requirements of the QAPP, as can be seen in Table C-3.

Paste pH

Saturated paste pH and 1:1 paste pH analysis was performed by the methods found in the Methods of Soil Analysis, Part 2, American Society of Agronomy (1982). All data met criteria established in the QAPP. All soil pH data was considered usable.

Total and Dissolved Metals

Total and dissolved metals data was provided by the HKM Laboratory. Select soils/tailings and waters were analyzed using inductively coupled plasma atomic emission spectroscopy (ICP-AES). All data was validated. Silicon was found in the laboratory preparation blanks; however, no data was flagged for silicon since all sample concentrations were greater than ten times the levels found in the blanks. Aluminum, copper, and silicon data from the installation tailings composite samples were flagged as estimated because of matrix spike recoveries outside the 75-125% recovery limit in the QAPP. Lead was flagged because of low matrix spike recovery in the final leachate samples taken on October 14, 2005. Flagged samples are shown in Table C-1. All metals data was considered usable given the previously cited constraints.

Remaining Analysis

Alkalinity, chlorine residual, and sieve analysis data was also reviewed. All data was considered usable and requires no qualification.

C.3.3 Program Evaluation

Program evaluation focused on:

- COC procedures;
- sampling and data completeness;
- field blanks; and
- field duplicates.

C.3.3.1 COC Procedures

All information provided in the COC forms for this project was complete and accurate, with the following exceptions.

- The tailings composite samples taken on September 25, 2003 had no time sampled provided. The time sampled should accompany every sample.
- The referee samples sent to SVL on May 24, 2004 had no date or time sampled provided. The date and time sampled should always be provided with a referee sample so correct and proper comparisons can be made.
- The cell leachate and irrigation water samples taken on October 6, 2004, did not have a time sampled provided. Again, the time sampled should accompany each sample to aid in identification.

C.3.3.2 Sampling and Data Completeness

Samples were collected and analyzed for all the parameters and during all phases (field installation, monitoring, and after test completion), except for mineralogy. Mineralogy was not a critical parameter and was omitted from both the initial and final phases. Saturated past pH analysis was completed on composite samples for the installation samples. Instead of paste pH on the test completion samples, 1:1 pH was substituted due to the limited amount of sample available in the cores.

The sampling frequency was dramatically relaxed after the decision was made to extend the project for a year. It was determined that the kinetics of the processes would not require the frequency initially stated in the QAPP and no compromise would occur as a result of the reduced sampling frequency.

C.3.3.3 Field QC Samples

All field QC samples were collected at the proper frequency for the tests specified in the QAPP. Sulfate was the analyte of primary concern so several field duplicates were collected throughout the project. A duplicate was not collected each sampling event; however, instructions were given and recorded on the COC for the laboratory to use a specific sample for QA/QC to ensure that each leachate collection location had several QC samples. The field duplicate results for sulfate were all within 20% RPD, so none of the samples were qualified.

C.3.3.4 Field Blanks

Field blanks were collected and analyzed for ABA analysis. Sand was designated as the field blank for ABA analysis. The results for the sand were all at or below the detection for total sulfur and the various sulfur fractions. No leachate sulfate field blanks were taken or defined in the QAPP. The leachate sulfate concentration was sufficiently high to virtually guarantee that the results would not be affected by low-level contamination. Laboratory blanks indicated that there was indeed no low or high-level sulfate contamination introduced by the laboratory. The sulfate values were not qualified.

No field blanks were prescribed in the QAPP or submitted for any of the other analysis.

C.3.3.5 Field Duplicates

Field duplicates for leachate sulfate all showed very good agreement.

Total sulfur for the tailings from the test cell installation ABA results had reasonable agreement (Table C-2). The cell C2 duplicate RPD was 28.5%, which is less than a typical 35% RPD limit for solid matrix samples. Field duplicate results for ABA total sulfur from the referee tailings samples sent to SVL were all acceptable except from treatment cell T1, which had 60.6% RPD.

Analysis of the ABA sulfur fractions (forms) had several results with RPDs in excess of 20%. The large variation in the sulfur fractions is not unusual, since these values are determined by difference.

C.4 QA/QC SUMMARY

Analytical laboratory results for ABA, leachate sulfate, pH, and other analysis are located in Appendix A. The majority of the findings with the analytical data and program evaluation are minor for this project. Several installation tailings ABA results were qualified. Leachate recordkeeping on the leachate volume was inconsistent and not clearly recorded.

C.4.1 ABA Analysis

The ABA analysis provided a challenge for the HKM Laboratory. Preliminary data did not provide acceptable agreement between total sulfur and the forms of sulfur. It was determined that further size reduction of the sample was necessary to achieve acceptable results. In order to obtain satisfactory results, the sample particle size had to be reduced to less than 100 mesh. This is significantly finer than the 60-mesh sample size specified in the method. The delay in receiving results and communications from the laboratory prompted the project manager to seek referee ABA analysis from SVL. Results were variable between the laboratories, but seemed comparable.

The ABA analysis was qualified mainly for imprecision in laboratory and field duplicates. This is the result of two factors, the first being the project precision requirement of < 20% RPD is more limiting than a 35% RPD often assigned soil samples in other QA programs. Secondly, the ABA fractions were often qualified. The general reason for this is due to ABA fractions being calculated from two separate analyses and the error being a function of multiple determinations. Since each determination contributes uncertainty to the final result, the more determinations involved in the result increases the uncertainty; therefore, it is more likely the duplicate result will exceed the RPD criteria.

C.4.2 Leachate Volume

The leachate volume was established as a critical parameter to measure the success of the cover technology being tested in this demonstration. There appeared to be no systematic means to record the volume of leachate being pumped from each test cell. The leachate volume measurements were recorded in field logbook; however, there was no direct correlation between the volume data from the logbook and appropriate laboratory analytical data; therefore, accuracy in calculating the mass of sulfate leaching from each cell was compromised. To help clarify this type of data collection in future projects, it would be advisable to formulate a specific logbook or log sheet that requires the essential information to be recorded for each event.

Table C-1. Summary of QA Outliers

Date	Sample ID	Lab ID	Analysis	QC Criteria	Control Limit	Result	Flag*	Comment
9/25/2003	ENPAR-C2-TAILS-1-0925	031020O001	Hot Water Extractable Sulfur	Lab Duplicate	20% RPD	60.0% RPD	J	Control limit established in the QAPP
	ENPAR-C2-TAILS-1D-0925	031020O002						
	ENPAR-C2-TAILS-2-0925	031020O003						
	ENPAR-C2-TAILS-3-0925	031020O004						
	ENPAR-C2-TAILS-4-0925	031020O005						
	ENPAR-C2-TAILS-5-0925	031020O006						
	ENPAR-C2-TAILS-6-0925	031020O007						
	ENPAR-C2-TAILS-7-0925	031020O008						
	ENPAR-C-SOIL-0926	031020O009						
	ENPAR-C-SAND-0925	031020O010						
9/25/2003	ENPAR-T1-TAILS-1-0925	031020O021	HCl Extractable Sulfur	Lab Duplicate	20% RPD	52.4% RPD	J	Control limit established in the QAPP
	ENPAR-T1-TAILS-1D-0925	031020O022						
	ENPAR-T1-TAILS-2-0925	031020O023						
	ENPAR-T1-TAILS-3-0925	031020O024						
	ENPAR-T1-TAILS-4-0925	031020O025						
	ENPAR-T1-TAILS-5-0925	031020O026						
	ENPAR-T1-TAILS-6-0925	031020O027						
	ENPAR-T1-TAILS-7-0925	031020O028						
	ENPAR-T1-SOIL-0926	031020O029						
	ENPAR-T1-SAND-1-0925	031020O030						
9/25/2003	ENPAR-T1-TAILS-1-0925	031020O021	Residual Sulfur	Lab Duplicate	20% RPD	31.9% RPD	J	Control limit established in the QAPP
	ENPAR-T1-TAILS-1D-0925	031020O022						
	ENPAR-T1-TAILS-2-0925	031020O023						
	ENPAR-T1-TAILS-3-0925	031020O024						
	ENPAR-T1-TAILS-4-0925	031020O025						
	ENPAR-T1-TAILS-5-0925	031020O026						
	ENPAR-T1-TAILS-6-0925	031020O027						
	ENPAR-T1-TAILS-7-0925	031020O028						
	ENPAR-T1-SOIL-0926	031020O029						
	ENPAR-T1-SAND-1-0925	031020O030						

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	Date	Sample ID	Lab ID	Analysis	QC Criteria	Control Limit	Result	Flag*	Comment	
C-11	9/25/2003	ENPAR-C1-TAILSCOMPOSITE-0925	031020O041	Aluminum in soil	Matrix Spike	75-125 % recovery (%R)	374.5 %R	J	Control limit established in the QAPP	
		ENPAR-C2-TAILSCOMPOSITE-0925	031020O042							
		ENPAR-T1-TAILSCOMPOSITE-0925	031020O043							
		ENPAR-T2-TAILSCOMPOSITE-0925	031020O044							
	9/25/2003	ENPAR-C1-TAILSCOMPOSITE-0925	031020O041	Copper in soil	Matrix Spike	75-125 %R	127.5 %R	J	Control limit established in the QAPP	
		ENPAR-C2-TAILSCOMPOSITE-0925	031020O042							
		ENPAR-T1-TAILSCOMPOSITE-0925	031020O043							
		ENPAR-T2-TAILSCOMPOSITE-0925	031020O044							
	9/25/2003	ENPAR-C1-TAILSCOMPOSITE-0925	031020O041	Silicon in soil	Matrix Spike	75-125 %R	157.7 %R	J	Control limit established in the QAPP	
		ENPAR-C2-TAILSCOMPOSITE-0925	031020O042							
		ENPAR-T1-TAILSCOMPOSITE-0925	031020O043							
		ENPAR-T2-TAILSCOMPOSITE-0925	031020O044							
	10/14/2005	ENPAR-T1-101405	051017J002	Lead	Matrix Spike	75-125 %R	69.7 %R	J	Control limit established in the QAPP	
		ENPAR-T2-101405	051017J003							
		ENPAR-C1-101405	051017J004							
		ENPAR-C2-101405	051017J005							
	10/26/2005	ENPAR-EFFTANK-102605	0510270650							
	9/25/2003	ENPAR-C1-TAILS-1-0925	031020O031	HCl Extractable Sulfur, HNO3 Extractable Sulfur, & Residual Sulfur	Field Duplicate	20% RPD	64.0% RPD, 24.6% RPD, & 53.3% RPD	J	Control limit established in the QAPP	
		ENPAR-C1TAILS-1D-0925	031020O032							
		ENPAR-C1-TAILS-2-0925	031020O033							
	ENPAR-C1-TAILS-3-0925	031020O034								
	ENPAR-C1-TAILS-4-0925	031020O035								
	ENPAR-C1-TAILS-5-0925	031020O036								
	ENPAR-C1-TAILS-6-0925	031020O037								
	ENPAR-C1-TAILS-7-0925	031020O038								
	ENPAR-C1-SOIL-0926	031020O039								
	ENPAR-C1-SAND-0925	031020O040								

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Date	Sample ID	Lab ID	Analysis	QC Criteria	Control Limit	Result	Flag*	Comment
9/25/2003	ENPAR-C2-TAILS-1-0925	031020O001	Total Sulfur, Hot	Field Duplicate	20% RPD	28.5% RPD,	J	Control limit established in the QAPP
	ENPAR-C2TAILS-1D-0925	031020O002	Water			44.4% RPD,		
	ENPAR-C2-TAILS-2-0925	031020O003	Extractable			25.6% RPD,		
	ENPAR-C2-TAILS-3-0925	031020O004	Sulfur, HCl			34.5% RPD, &		
	ENPAR-C2-TAILS-4-0925	031020O005	Extractable			23.9% RPD		
	ENPAR-C2-TAILS-5-0925	031020O006	Sulfur, HNO3					
	ENPAR-C2-TAILS-6-0925	031020O007	Extractable					
	ENPAR-C2-TAILS-7-0925	031020O008	Sulfur, &					
	ENPAR-C2-SOIL-0926	031020O009	Residual Sulfur					
	ENPAR-C2-SAND-0925	031020O010						
9/25/2003	ENPAR-T1-TAILS-1-0925	031020O021	Hot Water	Field Duplicate	20% RPD	68.1% RPD,	J	Control limit established in the QAPP
	ENPAR-T1TAILS-1D-0925	031020O022	Extractable			104.4% RPD,		
	ENPAR-T1-TAILS-2-0925	031020O023	Sulfur, HCl			& 24.0% RPD		
	ENPAR-T1-TAILS-3-0925	031020O024	Extractable					
	ENPAR-T1-TAILS-4-0925	031020O025	Sulfur, &					
	ENPAR-T1-TAILS-5-0925	031020O026	Residual Sulfur					
	ENPAR-T1-TAILS-6-0925	031020O027						
	ENPAR-T1-TAILS-7-0925	031020O028						
	ENPAR-T1-SOIL-0926	031020O029						
	ENPAR-T1-SAND-0925	031020O030						
9/25/2003	ENPAR-T2-TAILS-1-0925	031020O011	Hot Water	Field Duplicate	20% RPD	42.4% RPD, &	J	Control limit established in the QAPP
	ENPAR-T2TAILS-1D-0925	031020O012	Extractable			64.4% RPD		
	ENPAR-T2-TAILS-2-0925	031020O013	Sulfur, &					
	ENPAR-T2-TAILS-3-0925	031020O014	Residual Sulfur					
	ENPAR-T2-TAILS-4-0925	031020O015						
	ENPAR-T2-TAILS-5-0925	031020O016						
	ENPAR-T2-TAILS-6-0925	031020O017						
	ENPAR-T2-TAILS-7-0925	031020O018						
	ENPAR-T2-SOIL-0926	031020O019						
	ENPAR-T2-SAND-0925	031020O020						

* J – estimated

Table C-2. ABA Field Duplicate Summary/Comparison, HKM Lab vs. SVL

Cell	Lab	Field ID	Total Sulfur (%)	HNO ₃ Extractable Sulfur (Sulfide Sulfur-%)	Hot Water Extractable Sulfur (Sulfate Sulfur-%)	HCl Extractable Sulfur (Insoluble Sulfide Sulfur-%)	Residual Sulfur (Organic Sulfur-%)	Sum of Sulfur Fractions (%)	Sum of Sulfur Fractions – Total Sulfur (%)
C1	HKM	ENPAR-C1-TAILS-1-0925	5.80	4.52	0.38	0.22	0.68	5.80	0.00
		ENPAR-C1TAILS-1D-0925	6.68	5.79	0.38	0.11	0.39	6.68	0.00
		RPD	14.1%	24.6%	0.4%	64.0%	53.3%	14.1%	
	SVL	C1-3-A	5.55	4.67	0.83	1.57	0.05	7.12	1.57
		C1-3-B	5.01	3.85	1.12	2.01	0.04	7.02	2.01
		RPD	10.2%	19.2%	29.7%	24.6%	22.2%	1.4%	
C2	HKM	ENPAR-C2-TAILS-1-0925	4.70	3.75	0.19	0.11	0.65	4.70	0.00
		ENPAR-C2TAILS-1D-0925	6.26	5.31	0.29	0.14	0.51	6.26	0.00
		RPD	28.5%	34.5%	44.4%	25.6%	23.9%	28.5%	
	SVL	C2-2-A	4.40	3.88	0.48	1.30	0.04	5.70	1.30
		C2-2-B	4.98	4.48	0.47	1.60	0.03	6.58	1.60
		RPD	12.4%	14.4%	2.1%	20.7%	28.6%	14.3%	
T1	HKM	ENPAR-T1-TAILS-1-0925	7.70	6.25	0.67	0.19	0.60	7.70	0.00
		ENPAR-T1TAILS-1D-0925	8.56	7.16	0.34	0.60	0.47	8.56	0.00
		RPD	10.6%	13.5%	66.1%	104.1%	24.0%	10.6%	
	SVL	T1-1D-A	4.47	4.09	0.35	1.63	0.03	6.10	1.63
		T1-1D-B	8.36	7.26	1.01	1.80	0.09	10.16	1.80
		RPD	60.6%	55.9%	97.1%	9.9%	100.0%	49.9%	
T2	HKM	ENPAR-T2-TAILS-1-0925	4.67	3.50	0.30	0.10	0.76	4.67	0.00
		ENPAR-T2TAILS-1D-0925	4.85	3.91	0.46	0.09	0.39	4.85	0.00
		RPD	3.8%	11.0%	42.4%	17.9%	64.4%	3.8%	
	SVL	T2-3-A	5.18	4.82	0.32	1.26	0.04	6.44	1.26
		T2-3-B	4.42	4.33	0.05	0.64	0.04	5.06	0.64
		RPD	15.8%	10.7%	145.9%	65.3%	0.0%	24.0%	

Table C-3. Leachate Sulfate Field Duplicates

Sample ID	Field ID	Test Cell ID	Date Collected	Time Collected	Sulfate (mg/L)	RPD
041007L001	ENPAR-C1-100604	C1	10/6/2004		6641	4.1%
041007L005	ENPAR-C1-D-100604		10/6/2004		6376	
051017J004	ENPAR-C1-101405	C1	10/14/2005	14:15	5065	2.2%
051017J006	ENPAR-C1-D-101405		10/14/2005	14:40	4953	
040706M007	ENPAR-C2-070104	C2	7/1/2004	10:45	9115	2.8%
040706M010	ENPAR-C2-D-070104		7/1/2004	10:45	8862	
040628K001	ENPAR-T1-062304	T1	6/23/2004	9:25	9476	2.1%
040628K005	ENPAR-T1-D-062304		6/23/2004	9:25	9283	
050616Q004	ENPAR-T1-061605	T1	6/16/2005	13:10	8672	2.8%
050616Q006	ENPAR-T1D-061605		6/16/2005	13:10	8918	
050831O004	ENPAR-T1-083105	T1	8/31/2005	10:20	8214	11.0%
050831O006	ENPAR-T1-D-083105		8/31/2005	10:20	7354	
040715P005	ENPAR-T2-071504	T2	7/15/2004	8:45	12258	1.8%
040715P006	ENPAR-T2-D-071504		7/15/2004	8:45	12035	