

# **Selenium Treatment/Removal Alternatives Demonstration Project**

## **Mine Waste Technology Program Activity III, Project 20**

by

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Butte, Montana 59702

IAG DW89938870-01-0

National Risk Management Research Laboratory  
Office of Research and Development  
U.S. Environmental Protection Agency  
Cincinnati, Ohio 45268

and

Federal Energy Technology Center  
U.S. Department of Energy  
Pittsburgh, Pennsylvania 15236  
contract No. DE-AC22-96EW95405

National Risk Management Research Laboratory  
Office of Research and Development  
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## **Notice**

The information in this document has been funded in part by the U.S. Environmental Protection Agency under IAG DW89938870-01-0 and the Department of Energy Contract DE-AC22-96EW96405 to MSE Technology Applications, Inc., Butte, Montana 59702. EPA made comments and suggestions on the document intended to improve the scientific analysis and technical accuracy of the document. These comments are included in the report. However, the views expressed in this document are those of MSE Technology Applications, Inc. and EPA does not endorse any products or commercial services mentioned in this publication.

## Foreword

The mining and mineral processing industries are developing and modifying technologies that will enable these industries to operate more efficiently. If improperly dealt with, the waste generated by these industries can threaten public health and degrade the environment. The U.S. Environmental Protection Agency (EPA) is charged by the Congress of the United States with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, EPA strives to formulate and implement actions leading to a balance between human activities and the ability of natural systems to support and nurture life. These laws direct EPA to perform research to define and measure the impacts and search for solutions to environmental problems.

The National Risk Management Research Laboratory (NRMRL) of EPA is responsible for planning, implementing, and managing research, development, and demonstration programs to provide an authoritative, defensible engineering basis to support the policies, programs, and regulations of EPA with respect to drinking water, wastewater, pesticides, toxic substances, solid and hazardous wastes, and Superfund-related activities. The National Energy Technology Laboratory (NETL) of the U.S. Department of Energy (DOE) has responsibilities similar to NRMRL in that NETL is one of several DOE centers responsible for planning, implementing, and managing research and development programs. This document is a product of the research conducted by these two Federal organizations.

This document is the final report for EPA's Mine Waste Technology Program (MWTP) Activity III, Project 20, Selenium Treatment/Removal Alternatives. MWTP is a program developed through an Interagency Agreement between EPA and DOE. MSE Technology Applications, Inc., manages MWTP and is responsible for the field demonstration and reporting activities. The information generated under this program provides a vital communication link between the researcher and the user community.

One of the objectives of MWTP is to identify the types of mining wastes impacting the nation and the technical issues that need to be addressed. Other objectives of the program are: 1) address these technical issues through application of treatment technologies; 2) determine the candidate technologies that will be tested and evaluated; and 3) determine the candidate sites where these evaluations will take place.

E. Timothy Oppelt, Director  
National Risk Management Research Laboratory

## Acknowledgments

This document, *Final Report—Selenium Treatment/Removal Alternatives Demonstration Project*, was prepared for the U.S. Environmental Protection Agency (EPA) National Risk Management Research Laboratory (NRMRL) and the U.S. Department of Energy (DOE) National Energy Technology Laboratory by MSE Technology Applications, Inc. (MSE) under contract DE-AC22-96EW96405. The Selenium Treatment/Removal Demonstration Project was conducted under the Mine Waste Technology Program (MWTP) and funded by EPA with in-kind support contributions from Kennecott Utah Copper Corporation (KUCC). MWTP is jointly administered by EPA and DOE through an Interagency Agreement. MSE manages MWTP and owns/operates the MSE Testing Facility in Butte, Montana.

Roger Wilmoth from NRMRL served as EPA's MWTP Program Manager, and Melvin Shupe from DOE served as DOE's Technical Program Officer. Mary Ann Harrington-Baker served as MSE's Program Manager, Helen Joyce served as MSE's Project Manager, and Jon Cherry served as the Project Manager for KUCC. KUCC was a major contributor to the project through in-kind services including: permitting, laboratory analysis, influent tank rental, transfer of water from Garfield Wetlands-Kessler Springs to the MSE Demonstration Site, site-specific safety training, warehouse services, and miscellaneous supplies and chemicals. Dr. Larry Twidwell from Montana Tech of the University of Montana was the technology provider of the catalyzed cementation process and also served as technical consultant for the chemical processes demonstrated. Dr. D. J. Adams and Tim Pickett of Applied Biosciences served as technology providers for the biological selenium reduction technology and the enzymatic selenium reduction technology. The organization and execution of this project was a collaborative effort between the participants mentioned above. Without these contributions, this project could not have been completed.

In addition to the people listed above, the following agency and contractor personnel contributed their time and energy by participating in the Selenium Treatment/Removal Alternatives Demonstration Project and preparing this document.

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## Acronyms

AA	atomic absorption
AB	Applied Biosciences Corporation
BASBR	baffled anaerobic solids bed reactors
BDAT	best demonstrated available technology
BSeR™	biological selenium reduction
EPA	U.S. Environmental Protection Agency
ICP	inductively coupled plasma
KEL	Kennecott Environmental Laboratory
KUCC	Kennecott Utah Copper Corporation
MCL	maximum contaminant level
MSE	MSE Technology Applications, Inc.
MWTP	Mine Waste Technology Program
ORP	oxidation-reduction potential
std dev	standard deviation
TCLP	toxicity characteristic leaching procedure
TNPV	total net present value

## Executive Summary

This document is the final report for the U.S. Environmental Protection Agency's (EPA) Mine Waste Technology Program (MWTP) Activity III Project 20—Selenium Treatment/Removal Alternatives Demonstration Project. MWTP is a program developed through an Interagency Agreement (IAG) between EPA and the U.S. Department of Energy. MSE Technology Applications, Inc. (MSE) manages MWTP and owns/operates the MSE Testing Facility in Butte, Montana. MSE proposed and was granted funding for the Selenium Treatment/Removal Demonstration Project during the April 1999 IAG Management Committee Meeting.

Selenium contamination originates from many sources including mining operations, mineral processing, abandoned mine sites, petroleum processing, and agricultural run-off. Kennecott Utah Copper Corporation's (KUCC) Garfield Wetlands-Kessler Springs site has a well characterized selenium contaminated artesian flow and was selected as the site for demonstrating various selenium treatment technologies. The contamination is of a low-level, high-volume nature that makes most treatment options expensive.

The objective of the Selenium Treatment/Removal Alternatives Demonstration Project was to test and evaluate technologies capable of removing selenium from Garfield Wetlands-Kessler Springs water to below 50 micrograms per liter ( $\mu\text{g/L}$ ), the National Primary Drinking Water Regulation Maximum Contaminant Level for selenium established by EPA. Several technologies with the potential to treat this water were presented in MWTP, Activity I, Volume VII, *Issues Identification and Technology Prioritization Report—Selenium*.

Three technologies were selected for field demonstration during this project:

- EPA's Best Demonstrated Available Technology (BDAT)—ferrihydrite precipitation with concurrent adsorption of selenium

onto the ferrihydrite surface (ferrihydrite adsorption) optimized by MSE;

- a catalyzed cementation process developed by Dr. Larry Twidwell of Montana Tech of the University of Montana with assistance from MSE; and
- a biological selenium reduction (BSeR™) process developed by Applied Biosciences Corporation (AB) of Salt Lake City, Utah.

Because ferrihydrite adsorption is considered EPA's BDAT for selenium removal from solution, it was considered the baseline technology and was used as a basis for comparison with the innovative selenium removal processes. All work was performed under an EPA-approved Quality Assurance Project Plan.

All three of the processes were able to achieve the target level for selenium in effluent samples under optimized conditions. Table ES-1 summarizes the results from the field demonstration for each technology and also includes results from additional testing of the catalyzed cementation process that occurred at MSE's testing facility following the field demonstration.

The BSeR™ process performed most consistently during the demonstration. During the 187 days of evaluation, all but four effluent samples from the BSeR™ process were below 10  $\mu\text{g/L}$ , and greater than 70% of the effluent samples were below detection (2  $\mu\text{g/L}$ ).

A secondary objective of the project was to perform an economic analysis for scale-up of the processes to treat 300 gallons per minute (gpm) flow at the Kessler Springs site. The retrofit of a vacant water treatment plant/associated equipment at the Kessler Springs site was used as the basis for the capital costs.

Table ES-2 is a summary of the outputs of the economic analysis for the selected technologies treating groundwater with 2 mg/L selenium operating at

300 gpm. The figures are the total net present value for each process that was demonstrated in the field. The figures used represent an order of magnitude cost estimate. The BSeR™ process was the most economically attractive technology demonstrated during this project.

A fourth technology—enzymatic selenium reduction—was demonstrated on a bench scale by AB. Enzymatic systems have the following advantages over live microbial systems: 1) the potential for greatly increasing kinetics; 2) nutrients are not required; and 3) the effects of toxic process solutions can be eliminated. Methods to economically prepare stable enzyme preparations and enzyme preparations from different microorganisms were investigated. Several immobilization polymers were evaluated to increase operational longevity. Calcium alginate performed the best in regards to ease of handling, toxicity, cost, and performance. Problems with stability or possibly the loss of an electron donor system were problematic throughout the testing. The stability or electron donor systems of the preparations tested was not sufficiently reproducible to warrant pilot-scale tests during this project.

These and other selenium treatment technologies were also reviewed under a Comprehensive Environmental Response, Compensation, and Liability Act feasibility study at the KUCC site. The BSeR™ process technology has been identified by KUCC as the preferred treatment for Garfield Wetlands-Kessler Springs water if KUCC is unable to recycle the selenium-bearing water into the existing process water circuit. Currently, KUCC is recycling 100% of the Garfield Wetlands-Kessler Springs flow back into various operations as make-up water. If the process water circuit is shut down, the BSeR™ process technology has been identified as the technology capable of treating the Garfield Wetlands-Kessler Springs water.

**Table ES-1.** Demonstration results summary.

<b>Ferrihydrite Adsorption Results</b>		
Treatment Condition	Mean Selenium Effluent Concentration ±Standard Deviation (n = sample size)	Minimum Selenium Concentration
Low iron (~1400 mg/L iron)	304 µg/L ±69 (n = 27)	115 µg/L
Medium iron (~3000 mg/L iron)	201 µg/L ±103 (n = 13)	42 µg/L (at midpoint of process)
High iron (~4800 mg/L iron)	90 µg/L ±28 (n = 5)	35 µg/L (at midpoint of process)
Ferrous/ferric (~1200 mg/L ferrous/1200 mg/L ferric iron)	563 µg/L ±280 (n = 5)	409 µg/L
Recycle Sludge (~2340 to 13,290 mg/L iron)	387 µg/L ±58 (n = 12)	77 µg/L
<b>Catalyzed Cementation Results</b>		
Treatment Condition	Mean Selenium Effluent Concentration (µg/L) ±Standard Deviation (n = sample size)	Minimum Selenium Effluent Concentration (µg/L)
Catalyzed Cementation	834 µg/L ±204 (n = 42)	193 µg/L
Catalyzed Cementation with Increased Oxidation/Decreased pH in the reactor tank	35 µg/L (n = 2)	26 µg/L
Additional Testing of Catalyzed Cementation at MSE	3 µg/L <sup>1</sup> ±4.4 (n = 5)	<1 µg/L
<b>BSeR™ Process Results</b>		
Residence Time	Mean Selenium Effluent Concentration (µg/L) <sup>2</sup> ± Standard Deviation (n - sample size)	Minimum Selenium Effluent Concentration (µg/L)
12 hrs (Series 1)	8.8 µg/L ±10.2 (n = 17)	< 2 µg/L
11 hr (Series 2)	4.9 µg/L ±4.9 (n = 16)	< 2 µg/L
8 hr (Series 3)	< 2 µg/L ±2.6 (n = 12)	< 2 µg/L
5.5 hr (Series 2)	< 2 µg/L ±2.1 (n = 26)	< 2 µg/L

<sup>1</sup> Nondetects were substituted with 50% of detection limit (0.5 µg/L).  
<sup>2</sup> Nondetects were substituted with 50% of detection limit (1 µg/L).

**Table ES-2.** Comparative economic analysis of demonstrated technologies.

Cost	Ferrihydrite Adsorption	Catalyzed Cementation	BSeR™ Process
Capital	\$1,026,835 (includes system design, demolition, building modifications, equipment purchase and installation, construction, system start-up, commissioning, and project closeout)	\$1,083,285 (includes additional research and development work, system design, demolition, building modifications, equipment purchase and installation, construction, system start-up, commissioning, and project closeout)	\$603,999 (includes biofim support material, inoculum, system design, building modifications, equipment purchase and installation, construction, commissioning, and project closeout)
Annual Operating and Maintenance Cost	\$2,084,559 (includes reagent costs, manpower, maintenance, and power for equipment use)	\$1,165,358 (includes reagent costs, manpower, maintenance, and power for equipment use)	\$135,029 (includes nutrient costs, manpower, maintenance, and power for equipment use)
Net Present Value of Annual Operating and Maintenance Costs	\$16,992,127	\$9,499,323	\$1,100,682
Total Net Present Value	\$18,017,962	\$10,582,608	\$1,704,681
Net Present Value of \$/1,000 gallons treated	\$13.90	\$8.17	\$1.32

# 1. Introduction

## 1.1 Project Overview

This Final Report was prepared specifically for the Mine Waste Technology Program (MWTP), Activity III, Project 20—Selenium Treatment/Removal Alternatives Demonstration Project, which addresses the U.S. Environmental Protection Agency's (EPA) technical issue of *Mobile Toxic Constituents—Water*.

The Selenium Treatment/Removal Alternatives Demonstration Project consisted of demonstrating one standard process and three innovative processes for selenium removal from Garfield Wetlands-Kessler Springs Water at Kennecott Utah Copper Corporation (KUCC) in Magna, Utah.

## 1.2 Project Purpose

The purpose of the Selenium Treatment/Removal Alternatives Demonstration Project was to test and evaluate technologies capable of removing selenium from Garfield Wetlands-Kessler Springs water to below 50 µg/L, the National Primary Drinking Water Regulation maximum contaminant level (MCL) for selenium. Garfield Wetlands-Kessler Springs water has a selenium concentration of approximately 2,000 µg/L. Several technologies with the potential to treat this water were presented in MWTP, Activity I, Volume VII, *Issues Identification and Technology Prioritization Report—Selenium* (Ref. 1).

Three technologies were selected for field demonstration during Phase 1 of this project:

- EPA's Best Demonstrated Available Technology (BDAT) (Ref. 2)—ferrihydrite precipitation with concurrent adsorption of selenium onto the ferrihydrite surface (ferrihydrite adsorption) optimized by MSE Technology Applications, Inc. (MSE);

- a catalyzed cementation process developed by Dr. Larry Twidwell of Montana Tech of the University of Montana with assistance from MSE; and
- biological selenium reduction (BSeR™) process developed by Applied Biosciences (AB) of Salt Lake City, Utah.

Because ferrihydrite adsorption is considered EPA's BDAT for selenium removal from solution, it was considered the baseline technology and was used as a basis for comparison with the innovative selenium removal processes.

The demonstrations of the ferrihydrite and catalyzed cementation technologies were conducted at KUCC during October and November 1999. These two technologies were demonstrated in the MWTP demonstration trailer that was constructed as part of MWTP Activity III, Project 9—Arsenic Removal Demonstration Project. The BSeR™ process was designed by AB and constructed with assistance from KUCC. The BSeR™ process demonstration was conducted from October 1999 through April 2000.

Phase 2 of this project included additional testing of the catalyzed cementation process under optimized conditions identified during the field demonstration and bench-scale testing of an enzymatic selenium reduction process developed by AB. The additional testing of the catalyzed cementation process was conducted at MSE's testing facility in Butte, Montana, during March and April 2000. The bench-scale testing of the enzymatic selenium reduction technology was conducted at AB's testing facility in Utah from March 2000 through January 2001.

## 1.3 Scope of the Problem

Selenium is a problem in many wastewaters and is a common water contaminant throughout the world. Selenium contamination represents a major environmental problem in at least nine western U.S. states. This contamination originates from many sources including mining operations, mineral processing operations, abandoned mine sites, petroleum processing, agricultural runoff and natural groundwater. For mining waste, the principal sources of selenium contamination are copper- and uranium-bearing ores and sulfur deposits. Selenium is commonly found in mining wastewaters in concentrations ranging from 3 to >12,000 µg/L (Ref. 1). The National Primary Drinking Water Standard MCL is 50 µg/L for selenium. The National Fresh Water Quality Standard is 5 µg/L for selenium. The U.S. Fish and Wildlife Service has recommended that the national fresh water quality standard be lowered to 2 µg/L to protect fish, waterfowl, and endangered aquatic species. Questioning of this standard has arisen because some laboratory and field studies indicate that water borne selenium concentrations as low as 2.0 µg/L may bio-accumulate in aquatic food chains to toxic levels.

## 1.4 Site Description

KUCC's Garfield Wetlands-Kessler Springs site has a well defined selenium contaminated artesian flow with the following characteristics:

- groundwater containing selenate ranging from <50 to 10,000 µg/L;
- artesian flows 250–500 gpm, with selenium concentrations from 200 to 2,000 µg/L; and
- varying site water quality with some naturally occurring total dissolved solids concentrations greater than 5,000 mg/L.

Selenium, the primary contaminant of concern at this site, is present as selenate in the site's groundwater. Groundwater formerly surfaced from two main sources within the site into a large wetlands area on the boundary of the Great Salt Lake. Selenium contaminated artesian flow is currently captured and routed into KUCC's process water circuit. The contamination is of a low-level, high-volume nature that makes most treatment options expensive.

KUCC co-chairs a technical review committee with EPA, State organizations, and public groups to evaluate remediation/treatment strategies to substantially lower the release of selenium into the Garfield Wetlands and the Great Salt Lake. The Garfield Wetlands site is well characterized with site water and solids chemistry data available. A Garfield Wetlands site assessment indicated that natural selenium reduction is occurring at limited locations in the wetlands. Additionally, laboratory treatability testing of site waters indicated that these waters were at least somewhat difficult to treat, even though they appear by chemical analysis to only contain selenium as the major contaminant. A chemical profile of the Garfield Wetlands-Kessler Springs water is presented in Table 1-1.

This site provided an excellent opportunity to test the selected selenium removal technologies under MWTP. The BSeR™ process was constructed near Garfield Wetlands-Kessler Springs. The portion of the water emanating from the springs was fed directly to the biological process. The MWTP demonstration trailer was located near a vacant water treatment facility at KUCC approximately 2 miles from the Garfield Wetlands-Kessler Springs site. A photograph of the MWTP demonstration trailer and associated equipment at the demonstration site is shown in Figure 1-1. Feed water for the catalyzed cementation and the ferrihydrite precipitation processes was transported from Garfield Wetlands-Kessler Springs by a water truck and placed in a large bulk storage tank at that location.

**Table 1-1.** Composition of Garfield Wetlands-Kessler Springs Water

Analyte	Units	Sampled 5/5/99
Conductivity	µmho/cm	2,720
pH	standard units	7.08
Temperature	°C	13
Alkalinity	mg/L as CaCO <sub>3</sub>	315
Hardness	mg/L as CaCO <sub>3</sub>	601
Total Dissolved Solids	mg/L	1,520
Total Suspended Solids	mg/L	<3
Calcium	mg/L	145
Chloride	mg/L	496
Potassium	mg/L	11.6
Magnesium	mg/L	58
Sodium	mg/L	380
Sulfate	mg/L	294
Silver	µg/L	<1
Aluminum	µg/L	<5
Arsenic	µg/L	140
Barium	µg/L	34
Cadmium	µg/L	<1
Chromium	µg/L	<10
Copper	µg/L	29
Iron	µg/L	<300
Manganese	µg/L	<10
Molybdenum	µg/L	100
Nickel	µg/L	<40
Lead	µg/L	<5
Selenium	µg/L	1,950
Selenate	µg/L	1,870
Selenite	µg/L	49
Zinc	µg/L	<10

All field testing of these processes was conducted by MSE and AB with assistance from KUCC personnel as necessary. All sampling and field work was performed according to procedures outlined in the project specific quality assurance project plan and existing standard operating procedures.

All chemical analyses for collected samples were conducted at the Kennecott Environmental Laboratory (KEL) located at KUCC. KEL is certified by the State of Utah and audited annually by EPA. Confirmatory analyses were performed on 10% of samples at the HKM Analytical Laboratory located in Butte, Montana. A comparison of the KEL analyses and the HKM confirmatory analyses is presented in Appendix A—Summary of Quality Assurance Activities.

## 1.5 Technology Descriptions

The following technologies were demonstrated during Phase 1 of this project:

- BDAT–ferrihydrite adsorption of selenium;
- catalyzed cementation of selenium; and
- BSeR™ process.

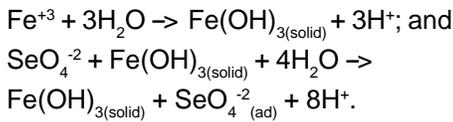
A brief description of each technology is provided in the following sections. During Phase 2 of the project, an enzymatic selenium reduction technology was evaluated, and additional data was collected for the catalyzed cementation technology.

### 1.5.1 Ferrihydrite Adsorption of Selenium

Ferrihydrite precipitation with concurrent adsorption of selenium onto the ferrihydrite surface (ferrihydrite adsorption) is EPA's BDAT for treating selenium-bearing waters. For adsorption of selenium using ferrihydrite to occur, the ferric ion (Fe<sup>+3</sup>) must be present in the water. Selenate (Se<sup>+6</sup>) is most effectively

removed from the water at pH levels below 4.

The chemical reactions for ferrihydrite precipitation of selenium are:



The selenium-iron solid product must be separated from the treated water before the process of selenium removal is complete. During the demonstration, solid-liquid separation was accomplished using a settler and filter press.

The selenium process water was delivered to the test site by a small tank truck and then transferred to a bulk storage tank. From the storage tank, the process water was pumped to the ferrihydrite adsorption process and the catalyzed cementation process. This arrangement provided the capability for operating both systems simultaneously.

Detailed in Figure 1-2 is the mechanical configuration of the ferrihydrite precipitation process system as tested during the pilot scale demonstration at a flow rate of approximately 5 gpm. Starting from the bulk storage tank, Garfield Wetlands-Kessler Springs water was introduced to the front end of the system. A digital programmable peristaltic metering pump controlled the flow rate of the process water through the treatment system. Following the pump, a turbine flow meter recorded the flow rate and the total volume of water processed.

The ferric chloride reagent was introduced next just in front of a static mixer. The static mixer ensured a homogeneous mix, thus, reducing reaction time.

From the static mixer, the process water was fed directly into an 80-gallon tank where a lime slurry was injected to increase the pH of the process water. A pH probe and controller monitored and adjusted the pH to an operator-selected set point. Additionally, the oxidation-reduction potential (ORP) of this tank was monitored and recorded. The overflow from the pH adjustment tank was collected in the transfer tank. A flocculent

was added to the second 80-gallon tank to assist with solid separation in the 1,000-gallon thickener. A level transmitter and level controller regulated the process water level in the transfer tank by adjusting the pumping rate of the transfer pump. At a flow rate of 5 gpm, the residence time of the thickener was about 200 minutes. This was adequate time for the solids to settle in the cone of the thickener tank.

The treated process water was removed from the top of the thickener and gravity fed to an 80-gallon batch transfer tank. To bring the pH of the water to neutral, a small amount of lime slurry was added to the transfer tank prior to final filtering and discharge. A pH probe and controller regulated the proper amount of lime slurry injected. The discharge pump operation was controlled by a level switch system that forced the water through a three-stage bag filter system. The filter system was a precaution against carryover of thickener solids in the event of an upset in the system.

Solids that accumulated in the bottom of the thickener were periodically removed by a diaphragm pump. This sludge slurry was then dewatered using a filter press. The liquid separated from the solids was returned to the thickener. The filter cake solids were removed from the filter press and prepared for analysis or disposal by placing them in appropriate containers. A photograph of the ferrihydrite adsorption process inside the MWTP demonstration trailer is presented in Figure 1-3.

### **1.5.2 Catalyzed Cementation of Selenium**

Catalyzed cementation is a process that was developed to remove arsenic and other heavy metals such as thallium and selenium from water. The term catalyzed cementation describes the process's ability to remove heavy metals from solution by cementation on the surface of the iron particles. It was anticipated that the catalyzed cementation process would have the ability to treat and remove selenium from solution regardless of its valence state (+6 or +4). To optimize the cementation process, proprietary catalysts are added to the process

to increase the selenium removal efficiency.

Detailed in Figure 1-4 is the configuration of the catalyzed cementation process system as tested during the pilot-scale demonstration. Starting from the bulk storage tank, Garfield Wetlands-Kessler Springs water was introduced to the front end of the system at approximately 1 gpm. A digital programmable peristaltic metering pump controlled the flow rate of the process water to the treatment system. Following the pump, a turbine flow meter was used to record the flow rate and the total volume of water processed. The catalyst reagent was introduced next, just in front of the first static mixer. The static mixer ensured a homogeneous mix and reduced the reaction time. Next, sulfuric acid was injected to lower the pH of the process water to the desired level. A second static mixer was used to speed-up the pH adjustment before the process water entered the elemental iron reactor. This reactor was a specialized tank designed to fluidize the iron particles. Additionally, pH and ORP were both closely monitored and recorded within this reactor. Iron particles that carried over were trapped in a small, cone-bottom tank and pumped back to the reactor for reuse.

Under gravity flow, the process water from the top of the small, cone-bottom tank was routed to a second 80-gallon reactor. Here, the pH of the water was raised with a lime slurry and an oxidizer was added to complete the required reaction. Flocculent was also added to this reactor to assist with solid separation. A level transmitter and level controller regulated the process water level in the reactor tank by adjusting the pumping rate of the transfer pump. At a flow rate of 1 gpm, the residence time of the thickener was about 15 hr. This was adequate time for the solids to settle in the cone of the thickener tank.

The treated process water was removed from the top of the thickener and gravity fed to an 80-gallon batch transfer tank. The operation of the discharge pump was controlled by a level switch system that forced the water through a three-stage bag filter system. The filter system was a precaution against carryover of thick-

ener solids in the event of an upset in the system.

Solids that accumulated in the bottom of the thickener were periodically removed by a diaphragm pump. This sludge slurry was then processed by a filter press. The sludge liquid separated from the solids was returned to the thickener. The filter cake solids removed from the filter press were prepared for analysis or disposal by placing them in appropriate containers. A photograph of the catalyzed cementation process in the MWTP demonstration trailer is shown in Figure 1-5. In addition to the ferrihydrite adsorption and catalyzed cementation processes, the BSeR™ process was also demonstrated.

### 1.5.3 Biological Reduction of Selenium

To accomplish biological selenium reduction, researchers at AB of Salt Lake City, Utah, have developed the BSeR™ process using anaerobic solids bed reactors (BASBR). Selenium (selenate and selenite) was reduced to elemental selenium by specially developed biofilms containing specific proprietary microorganisms. This process produces a precipitate of elemental selenium. With the aid of backflushing, 97% of the selenium reduced in the system can be removed from the bioreactors. This process was designed by AB and constructed with assistance from KUCC.

The BSeR™ process was demonstrated using a defined mixture of *Pseudomonas* and other microbes for removing selenium from Garfield Wetlands-Kessler Springs water. A block flow diagram of the BSeR™ process is shown in Figure 1-6. A photograph of the BSeR™ process at the Garfield Wetlands-Kessler Springs site is shown in Figure 1-7.

Garfield Wetlands-Kessler Springs water was pumped to the BSeR™ process at a flow rate of approximately 1 gpm using a solar pump. A flow meter/totalizer recorded the actual flow rate and the total volume of water processed by the BSeR™ process. The Garfield Wet-

lands-Kessler Springs water then entered a series of 500-gallon bioreactors containing carbon/biosolids/biofilm combination or carbon/biofilm, depending on the test series. Nutrients were supplied to the reactors at three locations in the process. When the water had flowed through the appropriate number of bioreactors, it was filtered by a slow sand filter before discharge.

Testing done previous to the pilot-scale demonstration produced the patent pending BSeR™ process that is demonstrated to reduce selenate and selenite in mining process solutions, petroleum wastewaters, and agricultural run-off using both single microbes and site-specific selenium-reducing bacteria. Initial batch and continuous bioreactor tests demonstrated selenium removal up to 97% in wastewaters containing up to 33.1 mg/L selenium in 4 to 6 hr with high-density microbial and microbial cocktail biofilms. In additional laboratory tests using a semi-fluidized bed reactor, live microbial and microbial cocktail biofilms have demonstrated selenium reduction rates of approximately 40 mg/L per 6 hr (Refs. 3 through 6).

The BSeR™ process implementation/configuration approach was to characterize and optimize naturally occurring microbial and like proprietary laboratory strains for each site-specific application. Using known, tested microbial strains and enhanced biofilm establishment techniques prevented the nonintentional incorporation of pathogens, undesirable indigenous nonselenium reducing microbes, and helped to ensure optimum selenium removal rates.

### 1.5.4 Enzymatic Reduction of Selenium

AB has isolated an optimized mixture of naturally occurring bacterial enzymes from heterotrophic bacteria previously isolated from selenium contaminated mining waters and soils. The bacterial enzymes reduce selenate and selenite in mining wastewaters to elemental selenium. Advantages of these cell-free systems over live bacterial systems in-

clude: (1) the potential for greatly increasing kinetics; (2) nutrients are not required; and (3) the effects of toxic process solutions can be eliminated. Bench-scale testing was performed to evaluate the enzymatic selenium reduction process and to make a decision whether to scale-up the process to pilot-scale for field demonstration. The enzymatic selenium reduction process was not recommended for scale-up due to the instability of the enzyme system matrix; therefore, a process flow diagram is not included for this technology.

## 1.6 Project Objectives

The primary objective of the field demonstration project was to assess the effectiveness of the processes being tested for removing selenium from Garfield Wetlands-Kessler Springs Water. More specifically, the objective that was defined for the project was to reduce the concentration of dissolved selenium in the effluent waters to a level under the National Primary Drinking Water Regulation MCL for selenium (50 µg/L) established by the EPA.

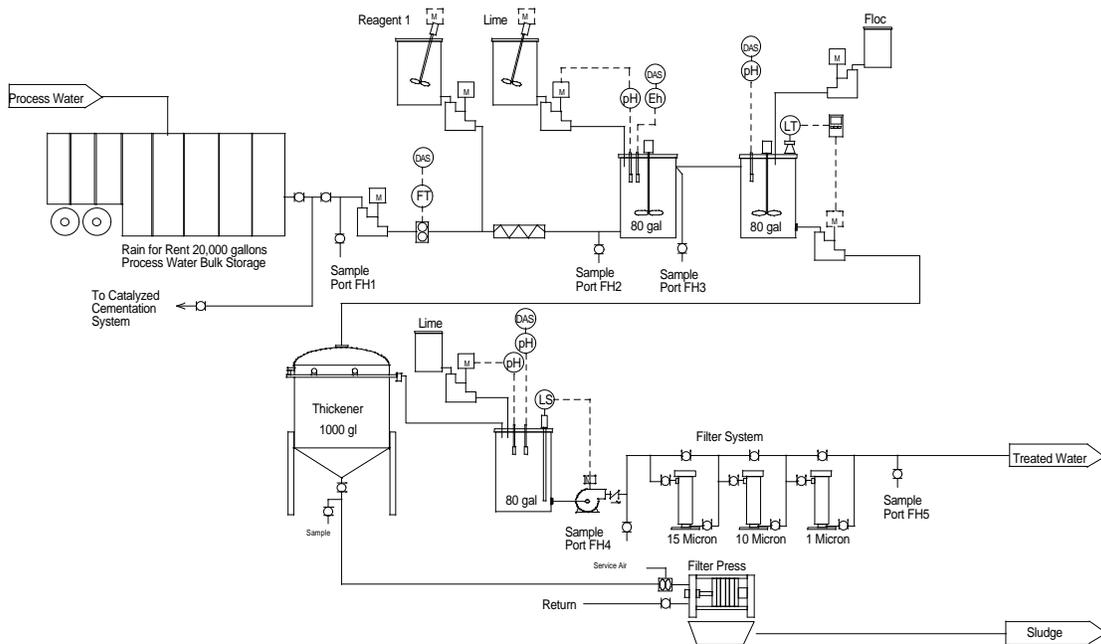
A secondary objective for the products from the catalyzed cementation and ferrihydrite precipitation processes was to render them environmentally stable by demonstrating that selenium results will be below the Maximum Concentration for Toxicity Characteristic using toxicity characteristic leaching procedure (TCLP) of 1.0 mg/L.

For AB's BSeR™ process, the product was expected to be marketable, and the secondary objective was to determine the purity and marketability of the product, and the impact the product had on process economics.

Another secondary objective was to perform an economic analysis for the scale-up of the processes tested to treat 300 gpm flow at the Garfield Wetlands-Kessler Springs site. The economic analysis for this project is presented in Section 3 of this report and represents an order of magnitude cost estimate.



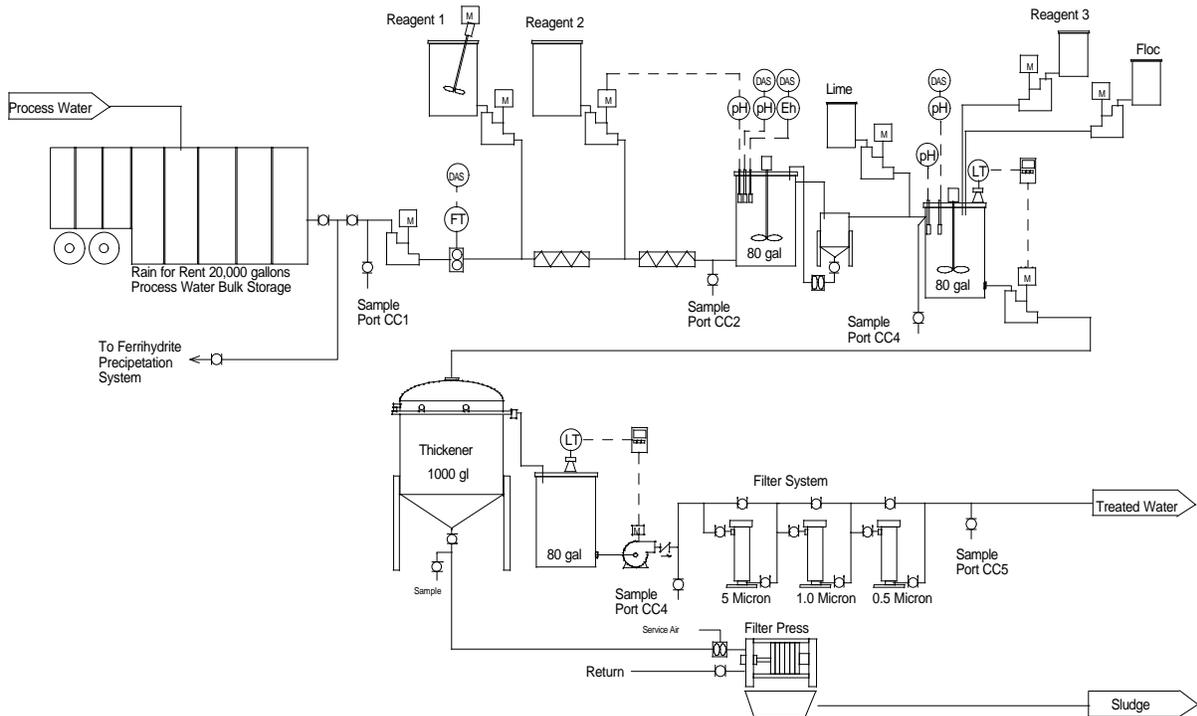
**Figure 1-1. MWTP demonstration trailer at the field site.**



**Figure 1-2. Ferrihydrite precipitation process flow diagram.**



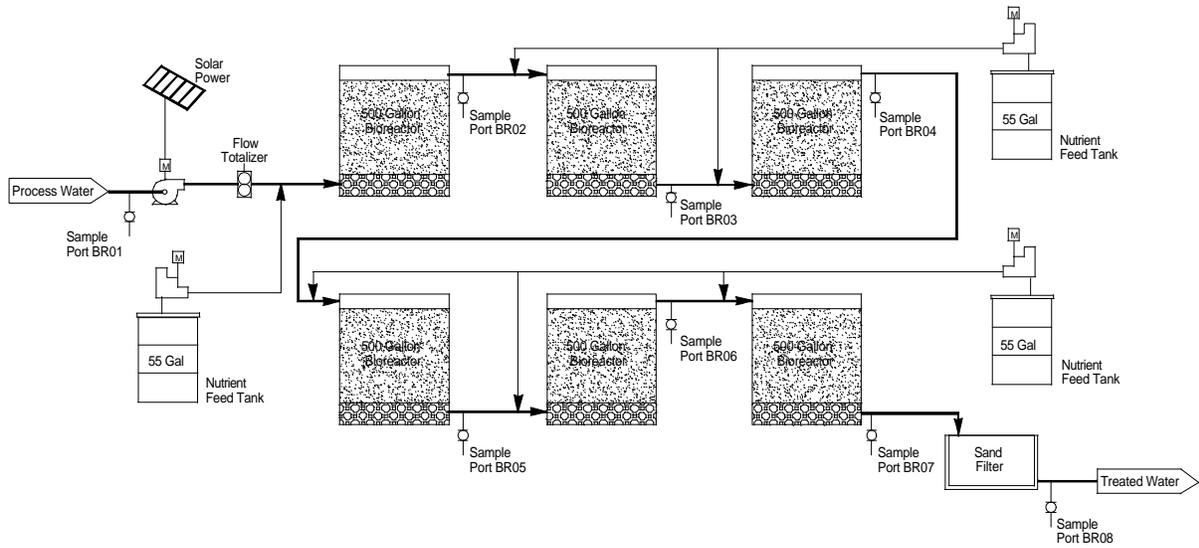
**Figure 1-3. Ferrihydrite adsorption process in MWTP demonstration trailer.**



**Figure 1-4. Catalyzed cementation process flow diagram.**



**Figure 1-5. Catalyzed cementation process in MWTP demonstration trailer.**



**Figure 1-6. BSeR™ process flow diagram.**



**Figure 1-7. Field-scale BSeR™ process reactor.**

## 2. Demonstration Description and Results

The following sections provide a description of the pilot-scale demonstration and any additional work for each technology as well as a brief discussion of the demonstration results. Field and laboratory data associated with each pilot-scale and bench-scale technology demonstration are contained in Appendix B. The sampling and analysis schedules for each pilot-scale technology demonstration are contained in Appendix C.

The achievement of the primary project objective for each process was determined by analyzing effluent samples for dissolved selenium concentration. Appropriate statistical tests were performed to determine the effectiveness of each process for selenium removal. Procedures outlined in *Guidance for Data Quality Assessment* (Ref. 6) were used to determine whether the data from each process was statistically below the action level of 50 µg/L dissolved selenium. During the demonstration of the ferrihydrite precipitation and catalyzed cementation processes, several different testing conditions were necessary before the processes removed selenium below the action level. Eventually, all three processes did remove selenium to below the action level of 50 µg/L; however, the ferrihydrite adsorption and the catalyzed cementation processes did not remove selenium to below 50 µg/L on a consistent basis. To determine if the primary project objective had been met, a Wilcoxon Signed Rank Test was performed on the effluent data set for

each process. The Wilcoxon Signed Rank Test was selected because each of the distributions were non-normal. Data QUEST software was used to test for normality. Filibens statistic ( $n > 50$ ) was used for the BSeR™ process and the ferrihydrite adsorption process, while the Shapiro-Wilks test ( $n < 50$ ) was used for the catalyzed cementation process. Non-normality was detected for all three distributions at a 5% significance level. The null hypothesis for the Wilcoxon Signed Rank Test was  $H_0$ : mean  $\geq 50$  ppb, and the alternative hypothesis was  $H_a$ : mean  $< 50$  ppb. The calculated sum of the Ranks for each process was compared to the critical value ( $w$ ) at  $\alpha = 0.05$ . Because the number of samples was greater than 20, a large sample approximation to the Wilcoxon Signed Rank Test was performed by calculating the  $z$  statistic for each process and comparing it to the critical value of  $z_{1-\alpha}$ . The results of the inferential analysis for all three processes are presented in Table 2-1. The BSeR™ process was the only technology that could reject the null hypothesis at a 5% significance level; thus, the effluent data from the BSeR™ process effluent suggests that the alternative hypothesis is more likely. The only process that was shown to statistically reduce selenium below the action level of 50 µg/L was the BSeR™ process. In fact, all of the effluent data from all BSeR™ process tests were less than 50 µg/L with the exception of some samples collected during start-up phases as the biofilm was maturing.

### 2.1 Ferrihydrite Adsorption Demonstration and Results

The ferrihydrite precipitation process was optimized by MSE for the demonstration. During the demonstration, several different tests were run to obtain the lowest possible concentration of selenium in the effluent water.

The effluent samples from the ferrihydrite precipitation processes were characterized to determine how effectively each treatment condition removed selenium from the Garfield Wetlands-Kessler Springs water. The solid products from the ferrihydrite precipitation process were analyzed for TCLP constituents as well as total constituents of interest.

Ferrihydrite precipitation is considered EPA's BDAT for selenium removal. Several tests were performed to determine the iron concentration necessary to remove selenium to below the target level of 50 µg/L. The various tests included:

- low iron condition (~1400 mg/L iron);
- medium iron condition (~ 3000 mg/L iron);
- high iron condition (~4800 mg/L iron);
- ferrous/ferric condition (~1200 mg/L ferrous/1200 mg/L ferric); and
- sludge recycle conditions (~2340 to 13290 mg/L iron).

**Table 2-1.** Summary of Results for Wilcoxon Signed Rank Test

Process	R calculated	$w_{\alpha}$ critical	z calculated	$z_{1-\alpha}$ critical	Result
Ferrihydrite Adsorption	0	1,211	-6.846	1.645	*
Catalyzed Cementation	3	636	-21.85	1.645	*
BSeR™ Process	2,256	1,565	5.603	1.645	Reject the null hypothesis at a 5% significance level because z calculated $> z$ critical.

\* There is not enough evidence to reject the null hypothesis at a 5% significance level because  $z$  calculated  $< z$  critical.

A graph of the results from the various test conditions is presented in Figure 2-1. The influent data represents Garfield Wetlands-Kessler Springs water, FH3 results were from midpoint in the system, and the effluent data are the discharge from the process. FH3 data are included because several times during the testing, results from midpoint in the process were less than the results at the effluent location. This may have been due to iron suppression of the selenium signal during inductively coupled plasma mass spectrometer analysis of the samples. The only conditions that removed selenium below 50 µg/L were the medium and high iron conditions, and this was only on a limited number of samples at the midpoint (FH3) of the process. Table 2-2 summarizes the results for each treatment condition.

### 2.1.1 Low Iron Test Results

The ferrihydrite demonstration was initiated in the MWTP demonstration trailer. The average pH during the low iron testing period was 3.9. The initial target iron concentration in the first 80-gallon tank in the process was approximately 1,400 mg/L iron (Fe/Se ratio, 900:1). Garfield Wetlands-Kessler Springs water was fed to the system at approximately 5 gpm. The mean selenium effluent concentration during the low iron tests was 303 µg/L [standard deviation (std dev), 69.4], well above the target of 50 µg/L. The minimum effluent selenium concentration during the low iron period was 115 µg/L.

### 2.1.2 Medium Iron Test Results

Because selenium removal was not at target levels, the target iron concentration was increased to 3,000 mg/L iron

(Fe/Se ratio, 2000:1). The average pH values recorded during this testing period was 4.1. The mean selenium effluent concentration during the medium iron concentration tests was 201 µg/L (std dev 103). The minimum effluent concentration achieved during this testing period was 42 µg/L selenium. Lower selenium results were achieved in the effluent samples with an increase in iron concentration from the low iron tests to the medium iron tests, so the iron concentration was further increased during the high iron concentration tests.

### 2.1.3 High Iron Test Results

The high iron test was initiated with iron concentrations of 4,800 mg/L (Fe/Se ratio, 3200:1). The mean selenium effluent concentration for this testing period was 90 µg/L (std dev 28), and the average pH value was 3.8. The minimum selenium effluent concentration achieved was 35 µg/L. Because reagent consumption (ferric chloride) was excessive during this period, high iron testing was suspended, and the system was set up to run a mixture of ferrous/ferric iron.

### 2.1.4 Ferrous/Ferric Test Results

To determine if the presence of ferrous iron in the system would positively impact selenium removal, a treatment condition using both ferrous and ferric iron was established. The amount of ferrous iron was increased in the system using ferrous sulfate. For this testing period, ferrous iron was approximately 1,200 mg/L, and ferric iron was approximately 1,200 mg/L. This process modification was not successful. The mean effluent selenium concentration during this test period was 563 µg/L (std dev 280). Once

these high selenium results were received from the laboratory, testing of this configuration was suspended.

### 2.1.5 Sludge Recycle Tests

The sludge generated from previous process tests was recycled during this test period. The iron used to attain the medium and high iron concentration conditions was in excess stoichiometrically so the sludge was recycled to take advantage of additional, available adsorption sites. To attain the desired iron concentration while minimizing reagent consumption, the sludge was recycled to the initial 80-gallon tank in the process. The mean selenium effluent concentration during this testing period was 387 µg/L (std dev 58). The minimum concentration of selenium in the effluent achieved during this testing period was 77 µg/L.

### 2.1.6 TCLP Results

To determine if the secondary objective had been achieved, filter cakes produced by the ferrihydrite adsorption process were subjected to TCLP analysis. The results are summarized in Table 2-3. While both filter cake samples failed TCLP for selenium (i.e., >1 mg/L), the total metal results presented in the last column of the table should be at least 20 times greater than the TCLP results but are instead less than detection. Therefore, TCLP results are questionable for the ferrihydrite adsorption process because the TCLP results for selenium do not correlate with the total selenium values. In the presence of excess iron, selenium is very difficult to detect in small concentrations.

Approximately 19,090 gallons of Garfield Wetlands-Kessler Springs water were processed during the ferrihydrite precipitation portion of the demonstration. The processed water was routed into KUCC's process water circuit and any wastes generated from the project were placed in KUCC's on site Comprehensive Environmental Response, Compensation, and Liability Act repository. Three days after the ferrihydrite tests were initiated, the catalyzed cementation process testing was initiated.

**Table 2-2.** Summary Results for Ferrihydrite Adsorption Tests

Treatment Condition	Mean Se Effluent Concentration ±Standard Deviation (n = sample size)	Minimum Selenium Concentration
Low iron	304 µg/L +69 (n = 27)	115 µg/L
Medium iron	201 µg/L +103 (n = 13)	42 µg/L (at midpoint of process)
High iron	90 µg/L +28 (n = 5)	35 µg/L (at midpoint of process)
Ferrous/ferric	563 µg/L +280 (n = 5)	409 µg/L
Recycle Sludge	387 µg/L +58 (n = 12)	77 µg/L

**Table 2-3.** TCLP/Total Selenium Results for Ferrihydrite Adsorption Filtercake Samples

Sample Description	Col. Date	AG-TCLP 0.1 mg/L	AS-TCLP 0.1 mg/L	BA-TCLP 0.1 mg/L	CD-TCLP 0.01 mg/L	CR-TCLP 0.1 mg/L	HG-TCLP 0.001 mg/L	PB-TCLP 0.1 mg/L	SE-TCLP 0.1 mg/L	SE-Total 0.5 mg/kg
FH Filtercake-221	10/31/1999	0.1	<0.1	0.1	<0.1	<0.1	0.001	<0.1	1.6	<0.5
FH Filtercake-225	11/18/1999	<0.1	<0.1	0.1	0.01	<0.1	<0.001	<0.1	1.1	<0.5

## 2.2 Catalyzed Cementation Process Demonstration

MSE tested several physical/chemical selenium removal technologies on a bench-scale to determine which technology would be tested on a pilot scale. Catalyzed cementation was the best selenium removal technology to emerge as a result of the bench-scale testing. Previous tests performed by Dr. Twidwell along with thermodynamic data strongly indicated that catalyzed cementation would be effective. Bench-scale results indicated that this process could remove selenium to below 50 µg/L. Scale-up to the pilot-scale did not immediately yield the same results.

Garfield Wetlands-Kessler Springs water was fed to the catalyzed cementation process at approximately 1 gpm. Chemistry conditions that were successful on a bench-scale were duplicated to maximize selenium removal. Despite attaining the proper conditions, selenium removal was not very successful for the majority of the tests. During the first 16 days of the test, the mean effluent selenium concentration was 834 µg/L (std dev 204). The minimum selenium concentration attained in the effluent water was 193 µg/L.

Near the end of the testing period, the pH in the cementation reactor was reduced to 3 and an increased oxidation condition was generated following the cementation step in an effort to improve the results. The mean effluent selenium concentration during this testing period was 35 µg/L, and the minimum effluent selenium concentration was 26 µg/L. These results were more promising than the initial portion of the testing, and the testing would have been continued; however, results were not received from the laboratory until the operation of the catalyzed cementation process had been

suspended. A summary of results from the field testing and additional testing of the catalyzed cementation process are summarized in Table 2-4. A graph of the influent and effluent selenium concentrations for the catalyzed cementation process is presented in Figure 2-2. Influent values represent the selenium concentration in Garfield Wetlands-Kessler Springs water, CC3 values represent midpoint of the process, and effluent values represent the discharge stream from the process. Approximately 10,000 gallons of Garfield Wetlands-Kessler Springs water were processed during the catalyzed cementation portion of the demonstration.

Additional testing to duplicate these optimum conditions for selenium removal was performed at MSE's testing facility. Preliminary results indicated that the process consistently removed selenium to below 40 µg/L, the inductively coupled plasma (ICP) detection limit at the HKM Laboratory. All samples below 100 µg/L were reanalyzed by furnace atomic absorption spectroscopy (AA) (detection limit 1 µg/L) to better quantify the selenium removal. The AA analysis yielded sample concentrations rang-

ing from <1 to 28 µg/L with a mean effluent concentration of 3 µg/L.

A process similar to catalyzed cementation is currently being investigated by Dr. Twidwell at Montana Tech of the University of Montana as part of MWTP, Activity IV, Project 19—*Removing Oxyanions of Arsenic and Selenium from Mine Waste Waters Using Galvanically Enhanced Cementation Technology*. The results of the research thus far have been very promising. If this modified cementation technology proves to be effective, it should be considered for pilot-scale testing.

Investigations utilizing agitated iron slurries and columns packed with iron have been performed by Eric Dahlgren (MSc graduate student at Montana Tech of the University of Montana and Dr. Twidwell (thesis advisor). These studies have demonstrated and optimized the cementation process applied to selenium removal from synthetic and actual plant process waters. Their results (Ref. 7) show that detection limit concentrations of selenium (<1 ppb) can be obtained utilizing the iron cementation technology.

**Table 2-4.** Summary of Results for the Catalyzed Cementation Process Demonstration

Treatment Condition	Mean Selenium Concentration (µg/L)±standard deviation (n = sample size)	Minimum Effluent Selenium Concentration (µg/L)
Catalyzed Cementation	834 µg/L ±204 (n = 42)	193 µg/L
Catalyzed Cementation with Increased Oxidation/Decreased pH in the Reactor Tank	35 µg/L (n = 2)	26 µg/L
Additional Testing of Catalyzed Cementation Under Optimized Conditions	3 µg/L <sup>1</sup> ±4.4 (n = 5)	<1 µg/L

<sup>1</sup> Nondetects were substituted with 50% of the detection limit (0.5 µg/L) to determine the mean selenium concentration.

**Table 2-5.** TCLP Results for Catalyzed Cementation Filtercake Samples

Sample Description	Col. Date	AG-TCLP 0.1 mg/L	AS-TCLP 0.1 mg/L	BA-TCLP 0.1 mg/L	CD-TCLP 0.01 mg/L	CR-TCLP 0.1 mg/L	HG-TCLP 0.001 mg/L	PB-TCLP 0.1 mg/L	SE-TCLP 0.1 mg/L
CC Filtercake-221	11/06/1999	<0.1	<0.1	0.1	<0.1	<0.1	0.001	<0.1	0.3
CC Filtercake-225	11/15/1999	<0.1	<0.1	0.1	0.02	<0.1	0.002	<0.1	<0.1

### 2.2.1 TCLP Results

To determine if the secondary objective was achieved, filter cake produced by the catalyzed cementation process was subjected to TCLP analysis. The results are summarized in Table 2-5. Both filter cake samples were below the TCLP threshold value for selenium of 1 mg/L. These results indicate that the catalyzed cementation process produced an environmentally stable precipitate, and therefore achieved the secondary project objective. In addition to the catalyzed cementation and ferrihydrite adsorption technologies, the BSeR™ process was also demonstrated.

### 2.3 Biological Selenium Reduction Process Demonstration

The BSeR™ process was demonstrated at the Garfield Wetlands-Kessler Springs site with a feed flow rate of approximately 1 gpm. Tests with residence times of approximately 12, 11, 8, and 5.5 hr (per reactor) were conducted. The BSeR™ process was demonstrated longer than the other processes to determine the reliability/longevity of the system. The BSeR™ process treatment unit was designed and built by AB with assistance from KUCC. Selenium values for all effluent samples were maintained below the 50 µg/L target for the entire test period. The pH in the individual reactor effluents ranged from 6.3 to 7.5, and the final discharge had an average pH of 7.26 over the entire pilot test period; anaerobic conditions were maintained in the reactors. Three different reactor series were operated in the field, treating a combined total of over 100,000 gallons of Garfield Wetlands-Kessler Springs water:

- Series 1 used 5 reactors in series (carbon/biosolids/biofilm) with a sixth reactor for inoculum and mixing nutrients to feed the reactors;
- Series 2 used 3 anaerobic reactors (carbon/biofilm) in series; and
- Series 3 used 3 anaerobic reactors (carbon/biofilm) in series.

Series 2 and 3 allowed for side-by-side comparison of two identical systems. Laboratory-scale reactors, started in advance of the field demonstration project, were used to help predict and optimize the BSeR™ process field reactors. Laboratory testing results are in Appendix D. An agricultural grade molasses was used as a base for a proprietary nutrient supplement that was mixed with the reactor feed waters to maintain the biofilm and provide energy for selenium reduction. A summary of the results from the BSeR™ process field testing is presented in Table 2-6. The mean selenium concentrations in the effluent for each residence time test were well below the 50 µg/L target concentration. Over 70% of the samples collected during the approximately 6 months of operation were below detection.

### 2.3.1 Series 1–Carbon/Biofilm and Biosolids Biofilm Reactors

The initial test configuration utilized both carbon/biofilm and biosolids/biofilm reactors in series. This test series was at a fixed retention time of 12-hr per reactor. After approximately 1 month of continuous operation, the reactors were decommissioned, and the matrix material was disposed. The five-reactor BSeR™ process system was terminated when the entire system was inadvertently heated to over 55 °C. The system was cleaned up, replumbed for operation as two, three-reactor systems; filled with new activated carbon; and reinoculated. Based on an evaluation of the biosolids matrix material, a decision was made to remove this matrix from future testing. The mean effluent concentration during this test series was 8.8 µg/L, and minimum effluent concentration was <2 µg/L. Figure 2-3 shows the results of these tests. The selenium removal was very good within the initial reactors; therefore, a decision was made that fewer reactors (three rather than five) could be used during subsequent test series.

**Table 2-6.** Summary of Results from BSeR™ Process Field Tests

Residence Time	BSeR™ Process Results	
	Mean Selenium Concentration (µg/L) <sup>1</sup> ±standard deviation (n = sample size)	Minimum Effluent Selenium Concentration (µg/L)
12 hr (Series 1)	8.8 µg/L ±10.2 (n = 17)	<2 µg/L
11 hr (Series 2)	4.9 µg/L ±4.9 (n = 16)	<2 µg/L
8 hr (Series 3)	<2 µg/L ±2.6 (n = 12)	<2 µg/L
5.5 hr (Series 2)	<2 µg/L ±2.1 (n = 26)	<2 µg/L

<sup>1</sup> Nondetects were substituted with 50% of detection limit (1 µg/L) to determine the mean selenium concentrations.

### 2.3.2 Series 2 and 3 Carbon/Biofilm Reactors

Two new series of reactors (three carbon/biofilm reactors each) were reconfigured for operation at the site. This new configuration allowed for side-by-side performance comparisons of two identical systems. In three different runs, systems were operated at retention times of 11, 8, and 5.5 hr (per reactor). Selenium removal, as a function of reactor retention time, is shown in Figure 2-4 combining data from the three reactor retention times (11, 8, and 5.5 hr). The average reactor temperature was about the same as the influent spring water ~16 °C and the pH of the influent and effluent waters ranged from ~7.0 to 7.7 with a general slight lowering of pH through the reactor systems. The heterotrophic facultative anaerobic nature of the selected microbial biofilm allowed effective selenium removal to below MCL levels at ORP values ranging from >200 to <-50 millivolts.

Biofilms capable of reducing both selenate and selenite produced an elemental selenium precipitate that was readily evident in the reactors and connecting tubes after ~48 hr of operation (see Figure 2-5). All but four effluent samples were below 10 µg/L, and greater than 70% of the effluent samples were below detection.

An ICP metals scan was performed on the system effluents to determine the removal efficiencies of other metals present in the Garfield Wetlands-Kessler Springs water. The BSeR™ process system also effectively removed trace levels of arsenic and copper from the system. Arsenic in the Garfield Wetlands-Kessler Springs water was removed from 70 µg/L to below detection, and copper was removed from 26 µg/L to below detection.

Laboratory tests demonstrated that agitation and/or back flushing freed much of the biologically reduced selenium from the biofilm support materials (granular carbon) and that filtration through a filter press would remove approximately 97% of the selenium. The collected elemental selenium/microbial product has a potential market niche as an animal feed supplement. Marketability analysis conducted in collaboration with an international feed supplement distributor indicates that the elemental selenium from the BSeR™ process can be used in various feed supplements. According to the distributor, the microbial biomass associated with the BSeR™ process will contribute an additional value.

### 2.4 Enzymatic Selenium Reduction Bench-scale Evaluation

Applied Biosciences has isolated an optimized mixture of naturally occurring bacterial enzymes from heterotrophic bacteria previously isolated from selenium contaminated waters and soils. The bacterial enzymes, which reduce selenate and selenite to elemental selenium were used to develop the enzymatic selenium reduction process. The enzymatic selenium reduction process was demonstrated at bench-scale by AB. The testing included the following tasks:

- test enzyme extracts from microbes with best demonstrated selenium reduction capabilities;
- optimize selenium enzyme extraction/purification protocols;
- examine immobilization/encapsulation formulations to increase the stability and extend the functional longevity of the enzyme preparations;
- evaluate the immobilized/encapsulated enzyme preparations for du-

rability and enzyme function (kinetics and stability); and

- determine initial bench-scale process operational parameters and any pretreatment recommendations.

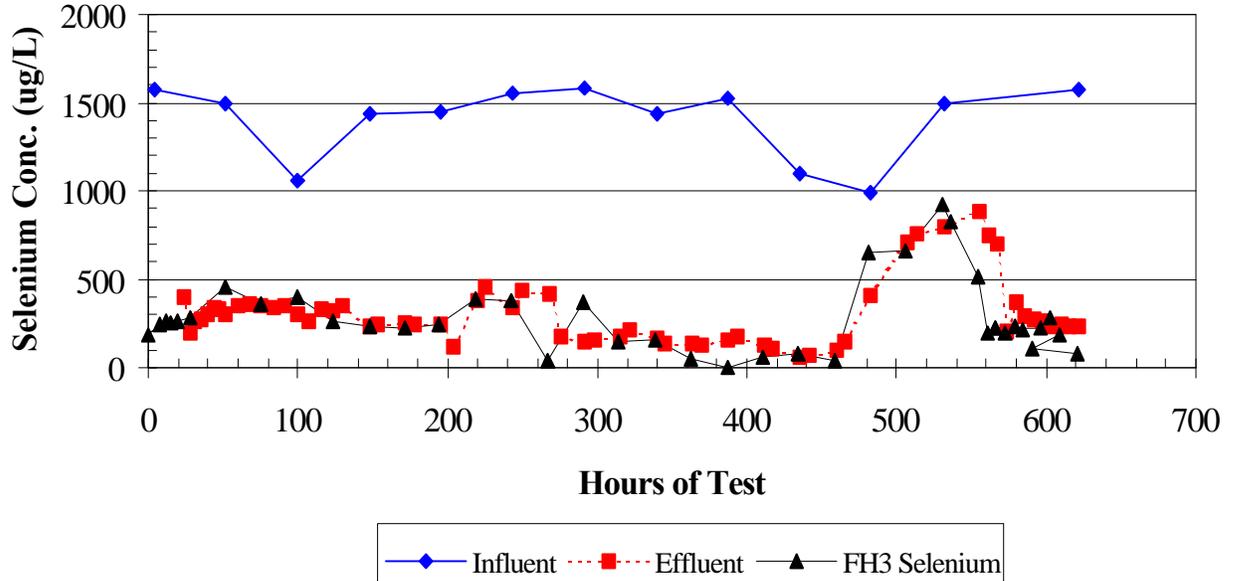
Top performing microbial cultures previously isolated from selenium containing mining wastes and soils were used as the source material for enzyme preparations. The prepared extracts were evaluated and screened over a 2-month period and compared to live cell preparations and appropriate controls. While the enzyme preparations initially exceeded the activity of the live cell preparations, a loss of stability was observed in the enzyme preparations that was not observed in the live cell preparations.

Due to the instability of the enzyme systems tested, the technology was not recommended for pilot-scale testing. The following conclusions were drawn based on the enzymatic selenium reduction bench-scale testing.

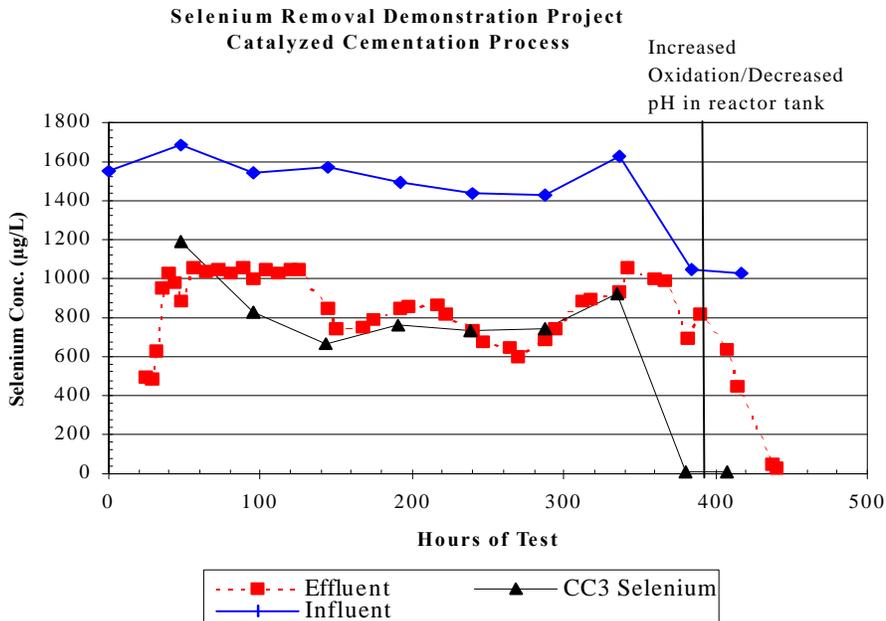
- Microorganisms are an alternative source for inorganic contaminant reducing enzymes.
- Selenium reduction in the presence of cyanide is possible using select enzyme preparations.
- Calcium alginate outperformed other encapsulation polymers in regards to ease of handling, toxicity, cost, and performance. AB's report summarizing the enzymatic bench-scale testing is contained in Appendix E.

Further research is recommended to further develop the electron donor system and enhance the operational longevity of the enzymatic selenium reduction technology. This research and development work is necessary to complete prototype development for this technology.

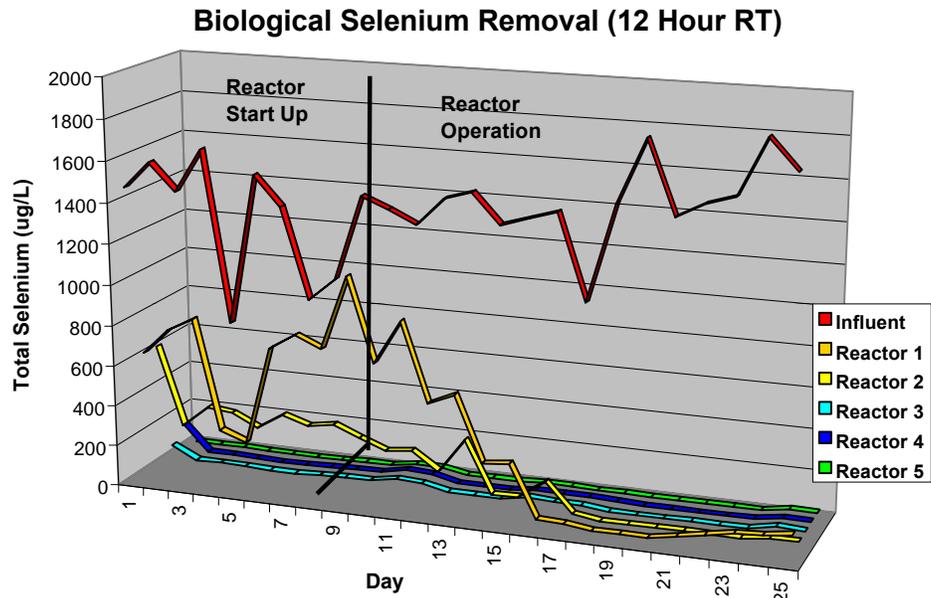
## Selenium Removal Demonstration Project Ferrihydrite Adsorption Process



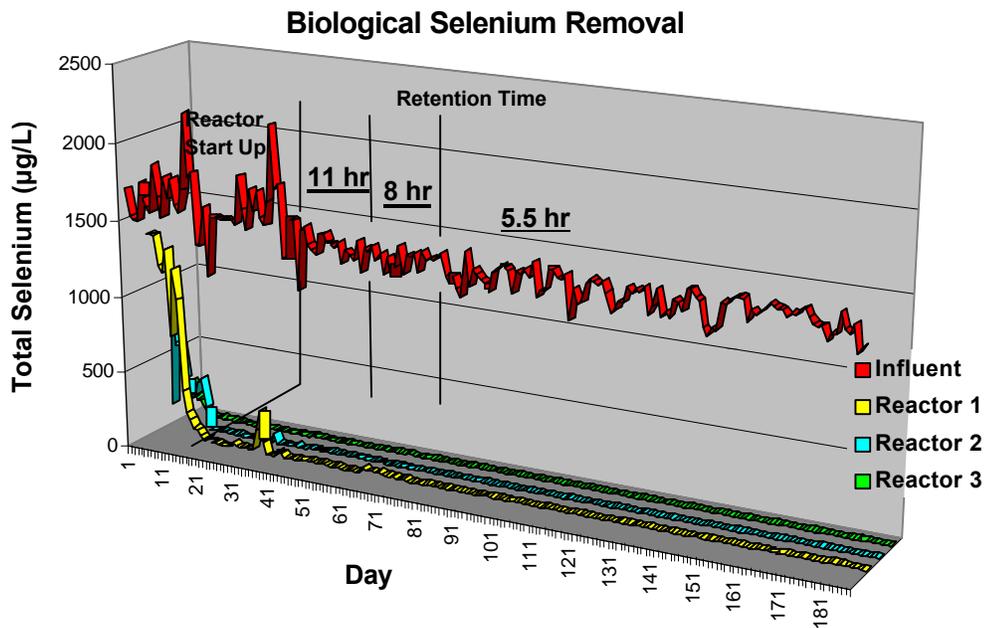
**Figure 2-1. Summary of results from ferrihydrite adsorption tests.**



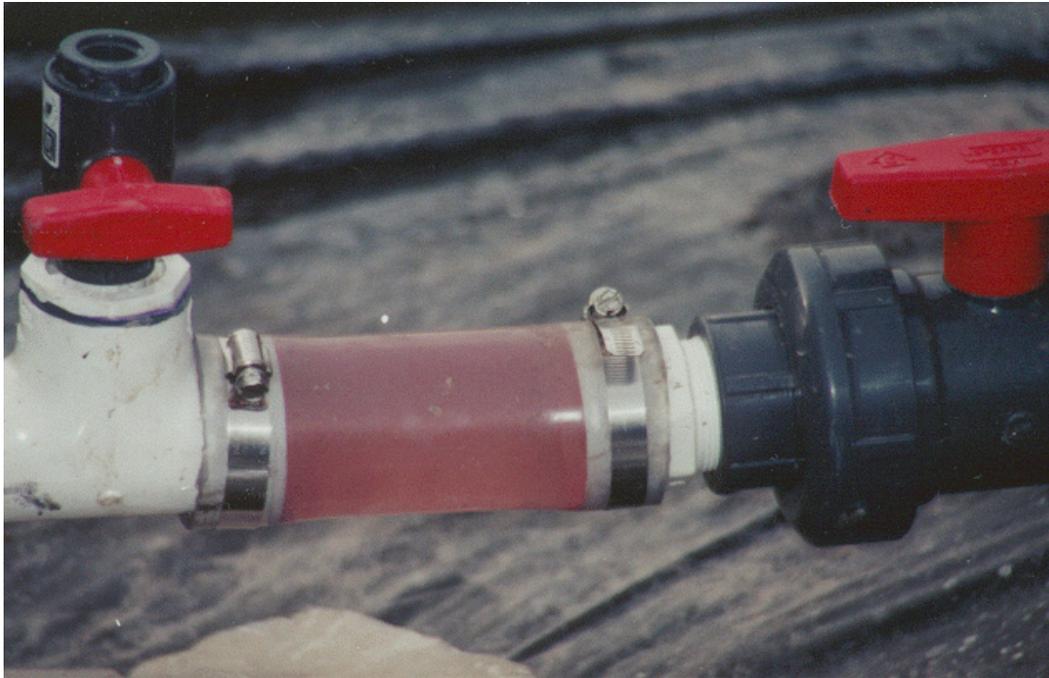
**Figure 2-2. Summary of results for field catalyzed cementation process tests.**



**Figure 2-3. Series 1 Pilot-scale BSeR™ process operation at a 12-hr retention time per reactor.**



**Figure 2-4. BSeR™ process pilot-scale reactor summary graph.**



**Figure 2-5. A red, amorphous, selenium precipitate observed in process piping after 8 hr of operation.**

### 3. Economic Analysis

A secondary objective of this study was to perform an economic analysis of the processes demonstrated. The costs presented are an order of magnitude cost estimate based on each of the treatment flow sheets. Definitions and cost estimation factors are taken primarily from similar work performed under MWTP. Itemized equipment lists were used where available.

Major cost items have been included. Capital costs include minor equipment, instrumentation, process piping, auxiliary engineering, and plant size factors for the ferrihydrite adsorption and catalyzed cementation processes. Capital costs provided by AB for the BSeR™ process included only biofilm support materials and \$40,000 to perform retrofits to the existing water treatment plant.

The following assumptions were made for completing the cost estimates:

- the processes would be installed at KUCC utilizing an existing water treatment facility;
- regulatory permits are in place;

- the Garfield Wetlands-Kessler Springs flow rate is 300 gpm, containing 2 mg/L selenium; and
- depreciation, leases, salvage and taxes were not considered.

A scale-up of each process to treat the entire 300 gpm of Garfield Wetlands-Kessler Springs flow was used as the basis of the economic analysis. Retrofit of equipment located at the existing water treatment facility was used as the basis for the scale-up. Because the field testing of the BSeR™ process and the catalyzed cementation process were only performed at 1 gpm, scaling up of these processes may not be as accurate as scaling up the ferrihydrite adsorption process that was demonstrated at 5 gpm.

#### 3.1 Ferrihydrite Adsorption of Selenium

The cost estimates presented for the scale-up of the ferrihydrite adsorption system are conceptual in nature and would be adjusted when an actual system design was implemented. Initial indications are that the reagent consumption

of this technology when effective (high iron condition) makes it cost prohibitive. The reagent consumption of this technology alone is estimated to be \$15.17/1,000 gallons treated when reagents are purchased in bulk. The estimates are based on information contained in the *Chemical Market Reporter* (Ref. 8). The majority of this cost was due to the high cost of the ferric chloride reagent, which accounts for \$14.31/1,000 gallons treated of the reagent costs. In a full-scale system, these costs would probably be lower if sludge generated was recycled to the reaction tank, thus, minimizing the fresh reagent usage.

Table 3-1 summarizes the capital costs and construction times necessary to retrofit the existing KUCC Waste Water Treatment Plant for ferrihydrite adsorption of selenium (high iron condition). The costs are associated with a system designed to handle a 300-gpm peak flow rate. Due to the difference in flow rate capability between the existing system and that of the scaled-up systems, most pumps and piping will require replacement.

**Table 3-1.** Capital Costs/Construction Schedule for Ferrihydrite Adsorption System Scale-Up

Task	Construction Time	Materials	Labor	Travel Nonlabor	Total
MSE System Design	11.3 weeks		\$145,450	\$11,538	\$156,988
MSE Subcontract Construction Oversight	8 weeks		\$51,530	\$21,568	\$73,274
MSE System Startup, Commissioning, and Project Closeout	5 weeks		\$44,190	\$10,266	\$54,375
Demolition, Building Modifications, Equipment Purchase, and Installation by Subcontract	12 weeks	\$612,107	\$36,079		\$648,850
Total	27.3 weeks				\$933,487
Schedule/Cost Contingency @ 10%	2.7 weeks				\$93,348
<b>TOTAL</b>	<b>30 weeks</b>				<b>\$1,026,835</b>

The cost of a filter press (approximately \$89,000) was also included in this estimate and may not be necessary depending on how the wastestreams from the system would be handled at KUCC. If a filter press was not necessary, the associated savings including shipping, filter press stand, sludge handling equipment, labor for installation, and design labor would be estimated at \$113,000.

### 3.2 Catalyzed Cementation of Selenium

The cost estimates presented for the scale-up of the catalyzed cementation system are conceptual in nature and would be adjusted when an actual system design was implemented. Initial indications are that the reagent consumption of this technology is still high, although approximately half of the reagent costs for the ferrihydrite adsorption system. The reagent consumption of this technology is estimated to be \$8.11/1,000 gallons treated. The majority of this cost is due to the cost of the oxidizing reagent, which accounts for \$5.81/1,000 gallons treated of the reagent costs. One way to reduce this cost would be to substitute the reagent used with a more cost effective alternative.

Table 3-2 summarizes the capital costs and construction times necessary to retrofit the existing KUCC Waste Water Treatment Plant. The costs are associated with a system designed to handle a 300-gpm peak flow rate. Due to the

difference in flow rate capability between the existing system and that of the scaled-up systems, most pumps and piping will require replacement.

The cost of a filter press (approximately \$89,000) was included in this estimate and may not be necessary depending on how the wastestreams from the system would be handled at KUCC. If a filter press was not necessary, the associated savings including shipping, filter press stand, sludge handling equipment, labor for installation, and design labor would be estimated at \$113,000.

Also included in this cost estimate is approximately \$75,000 in the system design task to perform additional research and development work on this process. Additional work is necessary to optimize reactor design, optimize elemental iron selection, optimize the conditions to maximize selenium removal, and optimize reagent additions.

The work of Dahlgren (Ref. 7) has shown that if a reactor is constructed so that very little air infiltration occurs, then the second-stage oxidation of the ferrous iron to ferric iron (with the subsequent ferric hydroxide, ferrihydrite, precipitation) is unnecessary. This is because the cementation process is very effective at removing selenium (<5 ppb) at pH 7–8. When the system is operated at pH 7–8, very little ferrous iron is produced (i.e., only a few ppm of iron dissolves). The ferrihydrite precipitation second stage of the present process is the most cost intensive step in the en-

tire treatment sequence. Therefore, the cost of the catalyzed cementation technology will likely be a cost competitive bioprocess or less than \$1.32 per 1,000 gallons (Ref. 9).

### 3.3 Biological Selenium Reduction (BSeR™) Process

Nutrient costs can be a primary contributor to the long-term operating cost of any biological process. Biotreatability results indicated that efficient short-term selenium reduction could be obtained with several media types; however, long-term selenium removal is dependent on a balanced nutrient mixture formulated to match process, microbial, and site water characteristics. The BSeR™ process has worked effectively in all waters tested with an inexpensive molasses-based nutrient. Nutrient costs can be reduced through careful microorganism selection and managed bioreactor microbial density. As determined in laboratory and pilot-scale tests, operating costs for the BSeR™ process are estimated to be less than \$0.50/1,000 gallons of treated water when nutrients are purchased in bulk quantities.

#### 3.3.1 Nutrient Costs

Nutrient costs for reactor operation at the selected flow rates are shown in Table 3-3. Nutrient costs ranged from \$0.51/1,000 gallons at a reactor retention time of 11 hr to \$0.58/1,000 gallons with a reactor retention time of 5.5 hr and averaged \$0.54/1,000 gallons.

**Table 3-2.** Capital Costs/Construction Schedule for Catalyzed Cementation System Scale-Up

Task	Construction Time	Materials	Labor	Travel Nonlabor	Total
MSE System Design	13.5 weeks	\$74,580	\$156,670	\$11,487	\$242,737
MSE Subcontract Construction Oversight	7 weeks		\$44,730	\$18,952	\$63,683
MSE System Startup, Commissioning, and Project Closeout	5 weeks		\$44,190	\$10,266	\$54,456
Demolition, Building Modifications, Equipment Purchase and Installation by Subcontract	12 weeks	\$588,342	\$35,587		\$623,929
Total	26.5 weeks				<b>\$984,805</b>
Schedule/Cost Contingency @ 10%	2.7 weeks				\$98,480
<b>TOTAL</b>	<b>29.2 weeks</b>				<b>\$1,083,285</b>

**Table 3-3.** Nutrient Usage and Cost Per 1,000 Gallons as a Function of Retention Time

Retention Time	Flow (gal/min)	Time (days)	Water Treated (L)	Nutrient (g)	Nutrient Use (g/L)	Nutrient (g/1000 gal)	Nutrient (\$/ton)	Nutrient (\$/1000 gal)
11	0.3	14	22982.4	11,000	0.48	1818.8	250	0.51
8	0.4	14	30643.2	15,000	0.49	1860.1	250	0.52
5.5	0.6	7	22982.4	12,500	0.54	2066.8	250	0.58

### 3.3.2 BSeR™ Process Biofilm Support Cost

In a pump-and-treat bioreactor system, it is advantageous to use an optimized support material for biofilm establishment. The BSeR™ process allows for establishing high-density biofilms that result in faster kinetics. The results of this and previous tests, including full-scale bioprocess implementation, continue to validate the use of carbon as a bioreactor support material for the BSeR™ process. Laboratory and field-tests have proven the durability of carbon as a stable biofilm support for long-term BSeR™ process operation. In fact, testing indicates that the biofilm support materials should have a life expectancy of 15+ years. Pilot tests completed at the Garfield Wetlands-Kessler Springs site indicate that the current selenium levels (2.0 mg/L) can be reduced to near or below detection with a retention time of <5.5 hr.

The BSeR™ process normally uses granular carbon as a biofilm support to establish specific biofilms that will endure long-term exposure to contaminated waters containing indigenous

nonselenium reducing microorganisms. This testing allowed additional comparisons and evaluations of other biofilm support materials. Granular carbon (8 x 30, I#900), evaluated in the laboratory along with the granular carbon from the field reactors, in bulk at a cost of \$0.48 per delivered pound, is the best biofilm support material tested to date for the BSeR™ process.

### 3.3.3 BSeR™ Process Capital Costs

Capital costs for the BSeR™ process are dependent on a great variety of factors including tank construction materials, use of available on-site tanks, pump and piping material specifications, and biofilm support materials. These factors all vary and can be adjusted to accommodate various site requirements of reactor materials, varying selenium contamination levels, and short or extended operating times. For example, the flow rates and projected extended operation times at the KUCC Garfield Wetlands-Kessler Springs site dictate a requirement for a durable biofilm support and shorter retention times; this was accommodated by using a biofilm support of granular carbon.

The cost of producing a bulk inoculum is estimated at \$0.75/1,000 gallons (cost dependent on BSeR™ process reactor size) and should only be required at start up. Two, 850,000-gallon clarifiers at the KUCC site would be used for this process. Granular carbon (8 x 30, I#900) costs \$0.48 per delivered pound. Conservatively, an estimated 360,000 lb of carbon support material is required for a 300 gpm BSeR™ process system at a cost of \$172,800. Laboratory and field tests suggest that the carbon can be used for a minimum of 25 reactor back flushing cycles for selenium removal and recovery, or an estimated 15 years at the Garfield Wetlands-Kessler Springs site.

Table 3-4 summarizes the capital costs estimated by MSE for the BSeR™ process system scale-up.

### 3.3.4 Comparative Economic Analysis

The three technologies demonstrated in the field were economically evaluated for a system operating at 300 gpm for 10 years @ 3.9% interest, 300 days per

**Table 3-4.** Capital Costs for BSeR™ Process System Scale-Up

Task	Construction	Materials	Labor	Total
AB System Design	4 weeks		\$53,807	\$53,807
AB Project Management	20 weeks		\$9,699	\$9,699
AB System Startup, Commissioning, and Project Closeout	5 weeks		\$113,875	\$113,875
Demolition, Building Modifications, Equipment Purchase and Installation by Subcontract	11 weeks	\$342,270	\$24,000	\$366,270
Total	20 weeks			\$549,090
Schedule/Cost Contingency @ 10%	2 weeks			\$54,909
<b>TOTAL</b>	<b>22 weeks</b>			<b>\$603,999</b>

year, to treat ground water containing 2 ppm selenium. The technologies were compared using the total net present value (TNPV) for each. The TNPV was determined by the following relationship:

$$\text{TNPV} = (\text{CapitalCost} + \text{NPVO} \& \text{MCost})$$

Where:

- TNPV is the total net present value;
- Capital Cost is the estimated capital cost to install each technolog

in the KUCC Wastewater Treatment Plant; and

- NPVO & MCost is the net present value of the estimated annual operating and maintenance costs.

The NPV function in Excel was used to calculate the NPV Operating Cost for each technology. A summary of the economic analysis of the three technologies is presented in Table 3-5.

Among the three technologies, the BSeR™ process technology dominates both technical and economical perfor-

mance. Catalyzed cementation was the next most cost effective treatment. The baseline technology, ferrihydrite adsorption, was the least attractive alternative from an economic standpoint. The operating and maintenance costs for the ferrihydrite adsorption and catalyzed cementation technology are much higher than the BSeR™ process due to high reagent usage. Optimization of reagent usage coupled with reagent substitution with lower cost reagents would make ferrihydrite adsorption and catalyzed cementation more economically attractive.

**Table 3-5.** Comparative Economic Analysis of Demonstrated Technologies

Cost	Ferrihydrite Adsorption	Catalyzed Cementation	BSeR™ Process
Capital	\$1,026,835 (includes system design, demolition, building modifications, equipment purchase and installation construction, system start-up, commissioning, and project closeout)	\$1,083,285 (includes additional research and development work system design, demolition, building modifications, equipment purchase and installation, construction, system start-up, commissioning, and	\$603,999 (includes biofilm support material, inoculum, system design, building modifications, equipment purchase and installation, construction, commissioning, and project closeout)
Annual Operating and Maintenance Cost	\$2,084,559 (includes reagent costs, manpower, maintenance, and power for equipment use)	\$1,165,358 (includes reagent costs, manpower, maintenance, and power for equipment use)	\$135,029 (includes nutrient costs, manpower, maintenance, and power for equipment use)
Net Present Value of Annual Operating and Maintenance Costs	\$16,992,127	\$9,499,323	\$1,100,682
Total Net Present Value	\$18,017,962	\$10,582,608	\$1,704,681
Net Present Value of \$/1000 gallons treated	\$13.90	\$8.17	\$1.32

## 4. Conclusions/Recommendations

Of the three technologies demonstrated, the BSeR™ process produced the most consistent results. A site-specific optimization is an essential component of any selenium removal process implementation, including the BSeR™ process. This optimization allowed the BSeR™ process to achieve economical removal efficiencies using realistic retention times while minimizing operating costs. Optimization of the BSeR™ process for the KUCC site produced a microbial cocktail that was later confirmed to efficiently remove selenium to near or below detection from Garfield Wetlands-Kessler Springs water using an inexpensive molasses-based nutrient blend and 5.5-hr retention times. The optimized microbial cocktail consisted of site-endemic and other naturally occurring, nonpathogenic microbes, including *Pseudomonas stutzeri* and *RC-large*. The BSeR™ process consistently removed selenium to below the target concentration (50 µg/L) and the majority of the time to below the detection limit of 2 µg/L.

The ferrihydrite adsorption process can also be optimized to achieve the desired level of selenium removal; however, reagent usage is excessive and cost prohibitive. Although this technology is considered the BDAT by EPA, it would not be feasible to utilize this technology to treat Garfield Wetlands-Kessler Springs water on a large scale. Another remaining question about this technology is the stability of the filter cake produced during this demonstration. Filter-cake samples did not pass TCLP for selenium but results were questionable because total metal analyses on the same samples did not correlate with the TCLP results.

The catalyzed cementation technology has also produced promising, albeit, erratic results. Additional testing of this process is necessary to provide more information about this innovative selenium removal technology. Further testing and optimization such as performing a solubility product or kinetic study to determine the optimum parameters for selenium and iron would make selenium removal using catalyzed cementation even more consistent and cost effective. The cementation reactor design may hold the key to the successful implementation of this technology. It is known that cementation of selenium can be accomplished in simple columns and stir tanks (Ref. 10). However, long residence times are required to achieve selenium removal to acceptable levels (Ref. 11). The recent work of Dahlgren (Ref. 7) and the continuation work by Dr. Twidwell (Ref. 9) has shown that iron packed columns are very effective for selenium removal (<1 ppb at pH 7) and require only a relatively short residence time (30 minutes). Current research indicates that novel agitation methods may provide the key to efficient selenium removal from solution. Testing of a system with a unique reactor design to accomplish the correct agitation method is necessary to further develop the catalyzed cementation technology.

The enzymatic selenium reduction technology was tested on a bench-scale during this project. The technology was not demonstrated in the field due to the instability of the enzyme reactor matrix. Plant enzyme preparations are commercially available; however, these plant-based preparations are much too expensive for water treatment applications. The use of microbial enzyme preparations

are expected to eventually reduce these costs. More research is necessary to gain a better understanding of what is occurring in the immobilization of the enzymes and the linking of electron donors within the various immobilization techniques. If the enzyme matrix can be demonstrated to be stable for 6 to 9 months, the process may be an economical treatment alternative. At the current operational longevity of 3 weeks to several months, the treatment costs become prohibitive. It is recommended that additional research be performed on the enzymatic selenium reduction technology because enzyme systems have the potential to outperform live microbial systems in many ways. Enzymatic technologies are still in the prototype development stage but have the potential to revolutionize drinking water and wastewater treatment.

In addition to further testing of the catalyzed cementation technology and enzymatic selenium reduction technology, other newly developed selenium treatment/removal technologies that may be ready for small-scale demonstration have been identified during this project. It is important to demonstrate these new technologies, in addition to the technologies tested during this project, to determine which technologies are effective at treating Garfield Wetlands-Kessler Springs water and also other waters with differing selenium concentrations and more complicated matrices. Further testing of these additional technologies could identify promising/economical technologies that could address the environmental problem of selenium contamination faced by the mining/mineral processing industries as well as the agricultural sector and the petroleum industry.

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## **APPENDIX A**

Summary of Quality Assurance Activities

Kennecott Environmental Laboratory/HKM Laboratory Data Evaluation  
Mine Waste Technology Program  
Activity III, Project 20  
Selenium Treatment/Removal Alternatives

## ACRONYMS

AB	Applied Biosciences Corporation
BDAT	best demonstrated available technology
CCV	continuing calibration verification
COC	chain-of-custody
EPA	U.S. Environmental Protection Agency
IDL	instrument detection limit
KEL	Kennecott Environmental Laboratory
KUCC	Kennecott Utah Copper Corporation
LCS	laboratory control sample
MDL	method detection limit
mg/L	milligrams per liter
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
RPD	relative percent differences
SOP	standard operating procedures
TCLP	toxicity characteristic leachate procedure
Fg/L	micrograms per liter

## 1. BACKGROUND

On October 23, 1999, sampling officially began for the Mine Waste Technology Program (MWTP) Activity III, Project 20—Selenium Treatment/Removal Alternatives at the Kennecott Utah Copper Corporation (KUCC) in Magna, Utah. The intent of the project was to obtain performance data on two chemical removal processes and one biological technology capable of selenium treatment/removal. The demonstration was conducted using Garfield Wetlands-Kessler Springs Water that has an approximate selenium concentration of 2 milligrams per liter (mg/L). The technologies demonstrated included:

- C ferrihydrite precipitation with concurrent adsorption of selenium onto the ferrihydrite surface [U.S. Environmental Protection Agency's (EPA) Best Demonstrated Available Technology (BDAT)] as optimized by MSE Technology Applications, Inc. (MSE);
- C catalyzed cementation process developed by MSE; and
- C biological selenium reduction technology developed by Applied Biosciences Corporation (AB) and implemented by AB with assistance from KUCC.

Because ferrihydrite precipitation is considered EPA's BDAT for selenium removal, it was the baseline technology used as a basis for comparison with the innovative selenium removal technologies.

The stated objective of the project was to reduce the concentration of dissolved selenium in the effluent waters to a level under the National Primary Drinking Water Regulation Maximum Contaminant Level for selenium [50 micrograms per liter (µg/L)] established by the EPA.

Samples were collected according to the schedule outlined in the approved project-specific quality assurance project plan (QAPP) document. The ferrihydrite precipitation and catalyzed cementation technologies were demonstrated for 3 weeks. The biological process was demonstrated for over 5 months. All field and laboratory data available has been evaluated to determine the usability of the data. Dissolved selenium analysis has been classified as a critical analysis for this project. A critical analysis is an analysis that must be performed to determine if project objectives were achieved. Data from noncritical analyses were also evaluated.

## 2. PROJECT REVIEWS

During the project, two evaluations were performed: 1) preproject evaluation of the Kennecott Environmental Laboratory (KEL); and 2) field systems review at KUCC demonstration site and KEL.

### 2.1 PREPROJECT EVALUATION OF KEL

Before the project began, a determination was made as to whether KEL was prepared/qualified to perform selenium analysis for this project. KEL holds accreditation from the following organizations that perform routine external audits:

- C Certified by the State of Utah for Environmental Testing performed under the Safe Drinking Water Act, the Clean Water Act, and the Resource Conservation and Recovery Act. The State of Utah audits KEL twice a year.
- C Accredited by the American Industrial Hygiene Association for all aspects of industrial hygiene analysis including heavy metals, free silica, and asbestos.
- C Participant regularly in Interlaboratory Performance Evaluation Testing for EPA, Proficiency Analytical Testing (four times a year), College of American Pathology, Discharge Monitoring Resource Quality Association, and Environmental Lead Proficiency Analytical Testing.
- C Audited by EPA for the National Pollution Discharge Elimination System once a year.

In addition to the external audits, KEL's quality assurance (QA) department performs internal audits twice a year. A review of the facilities indicated that KEL was prepared and qualified to perform analyses for the project. The unique matrix of the samples due to the high salinity in ground water samples near the Great Salt Lake made KEL a good choice because they routinely analyze these samples.

### 2.2 FIELD SYSTEMS REVIEW AT KUCC

A field systems review was performed on November 3, 1999, at the KUCC demonstration site and KEL. The field systems review included a review of the following items:

- personnel, facilities, and equipment;
- documentation (chain-of-custody and logbooks);
- calibration of equipment; and
- sampling procedures.

No concerns were identified during the audit. Some observations were made for areas not conforming exactly to the project specific QAPP.

### **2.2.1 Personnel, Facilities, and Equipment**

Personnel present during the audit included: Michelle Lee, MSE Project Engineer, and Ken Reick, MSE Project QA Officer. Some equipment for the demonstration was housed in the MWTP process demonstration trailer, while the influent feed tank and other associated equipment was located outside the confines of the trailer. Analysis and preparation of the samples (filtering and preserving) were performed in the sampling area provided inside a water treatment plant. The Project Engineer was knowledgeable about the demonstration and their duties and responsibilities at the demonstration site.

All equipment was calibrated prior to measurements in the MWTP process demonstration trailer or the designated sampling area. All calibration information was available and recorded in the project logbooks.

### **2.2.2 Documentation**

Chain-of-custody forms (COC) were reviewed at the demonstration site, and all COC procedures were being followed. The project logbooks were also reviewed. The sampling logbook was very thorough, and included spaces where specific information was required. Sampling personnel were familiar with the logbook format and COC procedures. The sampling logbook did not conform to the standard operating procedure (SOP) because the pages of the logbook were not numbered consecutively, and the unused portions of the logbook pages were not lined out and dated as stated in the SOP that was attached to the project-specific QAPP.

### **2.2.3 Calibration of Equipment**

Field equipment was used to manually measure pH and oxidation-reduction potential (ORP). This information was recorded in the project logbooks. All meters were properly calibrated prior to performing measurements. Standard operating procedures were available at the demonstration site for reference on how to calibrate/operate the meters. Sampling personnel were familiar with the SOPs and requirements for routine calibration of the various meters.

### **2.2.4 Sampling Procedures**

A review of sampling activities was also performed during the systems review. All sample collection procedures and equipment decontamination procedures were followed by sampling personnel with one exception. The QAPP required that the sample container that is shipped to the laboratory be rinsed three times with the solution to be analyzed. In this case, some of the filtered solution should have been used to rinse the 500-mL sample containers. None of the sample containers for samples collected during the audit were rinsed in this manner. The unfiltered sample containers were triple rinsed.

As a corrective action, the sampler was notified of this deficiency to ensure that compliance with the QAPP would occur at future sampling events. Michelle Lee indicated that the QAPP she had used to prepare for the audit did not indicate a separate rinsing procedure for the filtered sample bottle. This was a draft version of the QAPP. The official, approved version of the QAPP was available in the

trailer, as well as SOPs which indicated the proper rinsing procedure. In addition, other samplers were notified of the problem, and they indicated that they had been following the proper rinsing procedure since project initiation.

As a follow-up corrective action, this lesson learned was reiterated in annual sample collection refresher training for all MWTP personnel to avoid this problem in the future.

### **2.2.5 Analytical Facility Evaluation**

Project personnel delivered samples to KEL in sealed coolers containing blue ice with a COC. The COC was properly filled out, and samples were logged into KEL upon receipt. When samples from the project were delivered, an evaluation of KEL was also performed. No deficiencies with KEL were identified. The auditor described the facility as one of the best equipped inorganic analytical laboratories in the western United States.

### 3. DATA EVALUATION

The data quality indicator objectives for dissolved selenium analysis were outlined in the QAPP and were compatible with project objectives and the methods of determination being used. The data quality indicator objectives are method detection limits (MDL) for accuracy, precision, and completeness. Control limits for each of these objectives are summarized in Table 3-1.

**Table 3-1. Data quality indicator objectives.**

Parameter	Matrix	Unit	MDL <sup>a</sup>	Precision <sup>b</sup>	Accuracy <sup>c</sup>	Completeness <sup>d</sup>
Dissolved Se	Aqueous	µg/L	5	#20%	75-125%	90%
<sup>a</sup> Minimum detection limits are based on what is achievable by the methods, what is necessary to achieve project objectives, and account for anticipated dilutions to eliminate matrix interferences. MDLs will be adjusted as necessary when dilutions of concentrated samples are required. <sup>b</sup> Relative percent difference of analytical sample duplicates. <sup>c</sup> Percent recovery of matrix spike, unless otherwise indicated. <sup>d</sup> Based on number of valid measurements, compared to the total number of samples.						

In addition to the data quality indicators listed in Table 3-1, KEL also analyzes internal quality control (QC) checks, including calibration, calibration verification checks, calibration blanks, matrix spike duplicates, blank spikes, method blanks, and laboratory control samples. These QC checks have also been evaluated for the purposes of this data review.

## 4. VALIDATION PROCEDURES

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

### 4.1 ANALYTICAL EVALUATION

An analytical evaluation is performed to determine that:

- 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 remained within control limits;
- duplicate sample analysis was performed at the proper frequency and all relative percent differences (RPDs) were within specified control limits; and
- matrix spike sample analysis was performed at the proper frequency and all spike percent recoveries were within specified control limits;

Measurements that fall outside of the control limits specified in the QAPP, or for other reasons, were judged to be outlier and were flagged appropriately to indicate that the data is judged to be estimated or unusable.

An analytical evaluation was performed to determine the usability data that was generated by the KEL and the HKM Laboratory for the project. Laboratory data validation was performed using *USEPA Contract Laboratory Program National Functional Guidelines for Inorganics Data Review* (USEPA, 1994) (Ref. 1) as a guide. The QC criteria outlined in the QAPP were also used to identify outlier data and to determine the usability of the data for each analysis. A summary of QC check results for the critical selenium analysis and the noncritical total and TCLP selenium analyses is presented in Table 4-1. All data requiring flags is summarized in Table 4-2. In addition to the analytical evaluation, a program evaluation was performed.

### 4.2 PROGRAM EVALUATION

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

- all samples, including field QC samples, were collected, sent to the appropriate laboratory for analysis, and were analyzed and reported by the laboratory for the appropriate analyses;
- all field blanks contain no significant contamination; and
- all field duplicate samples demonstrate precision of field as well as laboratory procedures by remaining within control limits established for RPD.

**Table 4-1. Summary of QC checks for critical selenium analysis and noncritical total selenium and TCLP analysis.**

Analysis	Mean RPD for Sample Duplicates	Range of RPDs for Sample Duplicates
Dissolved Selenium	-0.38%	-5.4% to 2.9%
Selenium Hydride	0.8	-1.6% to 4.3%
	Mean Matrix Spike Recovery	Range of Matrix Spike Recoveries
Dissolved Selenium	100	80% to 120%
Selenium Hydride	98.4%	76% to 124%
Total Selenium (solid)	103%	100% to 110%
TCLP Selenium	100%	90% to 120%
	Mean Matrix Spike Duplicate Recovery	Range of Matrix Spike Duplicate Recoveries
Dissolved Selenium	98.6%	80% to 108%
Selenium Hydride	101.5%	76% to 120%
	Mean Matrix Spike/Matrix Spike Duplicate RPD	Range of Matrix Spike/Matrix Spike Duplicate RPDs
Dissolved Selenium	-0.07%	-5.1% to 3.4%
Selenium Hydride	-1.8%	-11.1% to -4.9%

**Table 4-2. Summary of qualified data for MWTP Activity III, Project 20.**

Date <sup>1</sup>	Sample ID	Analysis	QC Criteria	Control Limit	Result	Flag <sup>2</sup>	Comment
10/23/99 10/31/99 11/12/99 11/14/99 11/14/99	FH1-201 CC5-137 FH2-309 CC1-215 FH1-315	Iron Speciation	Holding Time	Analyze Immediately	48 hr	R	The data is considered unusable because samples were not brought to the laboratory for immediate analysis. A study was performed by KEL to determine the effect of the holding time on these samples; and as expected, the ferrous iron was significantly impacted. This data should be removed from consideration.
11/14/99	FH5-319 FH4-318 FH3-317 FH2-316 FH1-315	Barium Copper	Field Blank Field Blank	< 10 Fg/L < 10 Fg/L	78 Fg/L 538 Fg/L	U U	Samples with less than 10 times the contamination concentration in the blank, but above the MDL, should be flagged "U".
11/14/99	FH5-319 FH4-318 FH3-317 FH2-316 FH1-315	Barium	Field Duplicate	±20 Fg/L	68 Fg/L	J	Because samples were #5 times the instrument detection limit (IDL) for barium, the normal precision control limit of #20% RPD does not apply. An alternative control limit of ±2 times the IDL was applied and resulted in the arsenic data being flagged "J", as estimated.
11/15/99 11/16/99	FH3-321 FH5-322 FH3-323 FH5-324 FH3-325 FH5-326 FH3-327 FH5-328	Cadmium Lead Zinc	Continuing Calibration Verification	90-110% Recovery	Out of control on chart	J	Flag samples "J" for out-of-control continuing calibration verification (CCV).

**Table 4-2. Summary of qualified data for MWTP Activity III, Project 20.**

Date <sup>1</sup>	Sample ID	Analysis	QC Criteria	Control Limit	Result	Flag <sup>2</sup>	Comment
11/13/99	CC3-217	All analytes	All	N/A	Dissolved greater than total for all analytes	X	The dissolved portion of this sample was considerably darker than the total metal sample. This sample should be removed from consideration.
11/14/99	CC8-219	All analytes	Field Blank	No significant contamination	Contamination for barium, copper, and molybdenum	X	This field blank was obviously contaminated and was removed from consideration.
11/14/99	CC5-219	Selenium Speciation	Field Blank	< 2 time IDL (4 ppb)	15 ppb (selenium) 12ppb (selenite)	U	Samples with less than 10 times the contamination concentration in the blank, but above the MDL, should be flagged "U".
11/18/99	CC5-348	Selenium Speciation	Field Duplicate	< 35% RPD	50% RPD (selenium) 93% RPD (selenite)	J	Flag results "J" as estimated due to suspect field duplicate.
10/27/99	CC2-102	Selenium	T=870 D=990	Total should be greater than dissolved	Total results less than dissolved	J	Flag results "J" as estimated for suspect dissolved versus total results.
10/26/99	FH2-233	Iron	T=1340000 D=1800000				
11/11/99	CC2-190	Iron	T=409000 D=955000				
11/16/99	CC1-215	Selenium	T=974 D=1030				
10/31/99	FH Filter Cake-221	Selenium	TCLP=1.6 ppm Total = <0.5 ppm	TCLP results should be less than total metals results	TCLP results 2 to 4 times higher than total metals results	J	Flag results "J" as estimated for suspect TCLP versus total metals results.
11/18/99	FH Filter Cake-225		TCLP=1.1 ppm Total = <0.5 ppm				
1/10/00	BX-001 BX-002 BX-003 BX-004	Selenium	CCV  LCS	90-110% recovery  80-120% recovery	Out-of-control on chart  Out-of-control on chart	J	Flag samples "J", as estimated for out of control CCV and laboratory control sample (LCS).
<sup>1</sup> Date the samples were collected. <sup>2</sup> Data-qualifier definitions. U- The material was analyzed for, but was not detected above the level of the associated value (quantitation or detection limit). J- The sample results are estimated. R- The sample results are unusable. UJ- The material was analyzed for, but was not detected. The associated value is estimated.							

Program data that was inconsistent or incomplete and did not meet the QC objectives outlined in the QAPP were viewed as program outliers and were flagged appropriately to indicate the usability of the data. Both the analytical and program evaluations consisted of evaluating the data available as of June 1, 2000, from KEL and HKM Laboratory, which performed confirmatory analysis on 10% of the project samples.

#### **4.2.1 Field QC Samples**

In addition to internal laboratory checks, field QC samples were collected to determine overall program performance.

#### **4.2.2 Field Blanks**

None of the field blanks collected for the project showed significant contamination for dissolved selenium analysis, with two exceptions. The field blank (FH9-319) collected on November 14, 1999, did show significant contamination for barium and copper, which resulted in five samples—FH5-319, FH4-318, FH3-317, FH2-316, and FH1-315—receiving a “U” flag for these analytes. A “U” flag indicates the data is undetected below the associated value. Another field blank, CC8-219, collected on November 14, 1999, showed significant contamination for selenium speciation analysis, which resulted in the selenium and selenite values for sample CC5-219 receiving a “U” flag. The fact that both of these contaminated field blanks were collected on the same day may indicate a problem with sampling and/or laboratory procedures on that date.

#### **4.2.3 Field Duplicates**

All field duplicates collected were within control limits for all analyses, with the two exceptions. A field duplicate, FH8-319, was out of control for barium analysis. While EPA does not specify control limits for field duplicates, the data reviewer is allowed discretion when evaluating field duplicates. For this project, precision control limits of #35% RPD were used for field duplicates. As a result, the following samples were flagged “J”, as estimated: FH5-319; FH4-318; FH3-317; FH2-316; and FH1-315. A field duplicate collected on November 18, 1999 (CC8-348) was out of control for selenium speciation analyses, resulting in sample CC5-348 being flagged “J” for selenium and selenite values.

In addition to the collection of field duplicates, HKM Laboratory performed confirmatory selenium analysis on 10 % of the samples collected for the project. A comparison of the results from KEL and HKM Laboratory are presented in Table 4-3.

Basically, samples analyzed by the two laboratories were comparable. The results in Table 4-2 summarize all of the data that was flagged for various reasons throughout the project.

**Table 4-3. Comparison of results from KEL and HKM Laboratory.**

Sample ID	Date of Collection	KEL Result (Fg/L)	HKM Laboratory Result (Fg/L)	Relative Percent Difference
FH1-201	10/23/99	1570	1590	1.3%
CC1-101	10/27/99	1530	1390	9.6%
CC5-118	10/28/99	977	827	16.6%
FH5-257	10/31/99	115	88	26.6%
CC5-157	11/04/99	44	90	68.7%
FH5-304	11/10/99	64	58	10.5%
CC1-215	11/14/99	1030	1370	28.3%
CC5-219	11/14/99	105	119	12.5%
CC8-219	11/14/99	29	60	69.2%
CC9-219 (blank)	11/14/99	< 10	< 0.75	N/A
FH8-319	11/14/99	825	642	24.9%
FH9-319 (blank)	11/14/99	< 10	< 1.4	N/A
FH5-319	11/14/99	800	603	28.1
FH1-315	11/14/99	1500	1340	11.3

#### 4.3 IRON SUPPRESSION ON SELENIUM

The samples submitted for the ferrihydrite process had high iron interference, which suppressed the selenium spectra significantly. KEL's analyst talked to the manufacturer about inter-element correction calculations that could be made through the software. Suggested corrections were made; however, the suppression of the selenium spectra continued. The majority of the problems were encountered on samples from sample ports FH2 and FH3 (midpoints of the ferrihydrite system). Effluent samples did not have enough iron to cause problems with the laboratory analysis or data analysis.

#### 4.4 DISSOLVED METALS VERSUS TOTAL METALS

On several occasions, the dissolved metal results were higher than the totals. KEL reanalyzed the samples a second time for verification, and the dissolved results were still higher than the totals. Dissolved results should be less than or equal to the total metal results. These results may indicate a problem with sampling techniques such as contaminated filter paper/apparatus, insufficient decontamination procedures, or mislabeling of containers.

#### 4.5 TCLP VERSUS TOTAL METALS

There were also inconsistencies in TCLP versus total metal results on the filter-cake samples collected from the ferrihydrite adsorption process. Total metal results should be greater than or equal to the total metal results because the TCLP represents at least a 20 times dilution of the total metals.

## 5. SUMMARY

All data from KEL and HKM Laboratory has been validated according to EPA guidelines and the project specific QAPP. Some of the data was flagged for various reasons and is summarized in Table 4-2.

Two major findings are listed below.

- C When a difficult matrix water must be analyzed for a project, it is recommended that the laboratory receive samples to perform analysis on and determine the presence of interferences so that interference can be dealt with before it results in qualification of data.
- C Miscommunication between MSE and KEL personnel resulted in data for iron speciation flagged "R" as unusable. KEL had requested that the samplers notify the laboratory the day before ferrous samples would arrive so KEL could be prepared to analyze them promptly. MSE agreed to do this but did not follow through. There were eleven sampling events for iron speciation, and only four of the eleven times the holding time was met. KEL's analyst did a mini experiment to see the effect of the holding time on the sample. On sample CC5-193, ferrous iron was 37 mg/L on the day the sample was delivered and only 10 mg/L two days later, illustrating the importance of the holding time on iron speciation analysis. Holding time requirements should be communicated better to the sampling team to avoid this problem on future projects.

MWTP, Activity III, Project 20 presented unique challenges for the sampling and analytical team. While several of the data points were flagged for various reasons, none of the critical data was discarded during the data evaluation/validation process.

## 6. REFERENCES

1. U.S. Environmental Protection Agency, "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review," EPA-540/94-013, February 1994.

## **APPENDIX B**

Test Data

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**Table B-1. Ferrihydrite adsorption process demonstration field data record**

BACKGROUND DAYS										
WEEK 1										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR-			pH, ORP		7.1	185				
WEEK 1 (CONTINUOUS)										
DAY 1 INITIAL 10/23/99 Time Zero=13:00 hours										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR - 0		101	pH		3.7			14:00	JM	
HOUR - 0		102	pH, ORP		3.92	605				
HOUR - 0		103	pH		7.5					
HOUR - 0		FIT	Total Flow	115						
WEEK 1 (CONTINUOUS)										
DAY 1 INITIAL										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR - 4		101	pH		3.85		1370	17:00	JB	
HOUR - 4		102	pH, ORP		4.01	420				
HOUR - 4		103	pH							
HOUR - 4		FIT	Total Flow	221.32						
HOUR- 8		101	pH		3.85					
HOUR- 8		102	pH, ORP		4	565	1450	21:00	JB	
HOUR - 8		103	pH							
HOUR- 8		FIT	Total Flow	--						
HOUR- 12		101	pH		3.88		1280	1:00	RZ	
HOUR -12		102	pH, ORP		4.05	425				
HOUR -12		103	pH		--					
HOUR -12		FIT	Total Flow	494.2						
HOUR -16		101	pH		3.9		1290	5:00	RZ	
HOUR -16		102	pH, ORP		4.1	451				
HOUR -16		103	pH		--					
HOUR -16		FIT	Total Flow	670						
HOUR -20		101	pH		4.04		1290	9:00	KN	
HOUR -20		102	pH, ORP		4.16	422				
HOUR -20		103	pH		4.98					
HOUR -20		FIT	Total Flow	4.3						
HOUR -24		101	pH		4.18			13:00	KN	
HOUR -24		102	pH, ORP		4.29	409	1420			
HOUR -24		103	pH		5.93					
HOUR -24		FIT	Total Flow	117.38						
HOUR -24		FH5	pH		6.6	405				

**Table B-1. Ferrihydrite adsorption process demonstration field data record**

WEEK 1 (CONTINUOUS)										
DAY 2										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR - 4		101	pH		3.97			17:00	JB	
HOUR - 4		102	pH, ORP		4.16	555	1310			
HOUR - 4		103	pH		6.15					
HOUR - 4		FIT	Total Flow	254.8						
HOUR- 8		101	pH		3.87					
HOUR- 8		102	pH, ORP		4	430	1370	21:00	JB	
HOUR - 8		103	pH		6.09					
HOUR- 8		FIT	Total Flow	390.09						
HOUR- 12		101	pH		3.78			1:45	RZ	Sampling delayed due to pump problems
HOUR- 12		102	pH, ORP		4.11	435	1260			
HOUR -12		103	pH		6					
HOUR -12		FIT	Total Flow	559.34						
HOUR -16		101	pH		3.84		1240	5:00	RZ	
HOUR -16		102	pH, ORP		4.17	432				
HOUR -16		103	pH		6.04					
HOUR -16		FIT	Total Flow	662.95						
HOUR -20		101	pH		3.91					
HOUR -20		102	pH, ORP		4.14	426		9:00	MGL	
HOUR -20		103	pH		3.06					
HOUR -20		FIT	Total Flow	808.83						
WEEK 1 (CONTINUOUS)										
DAY 2										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR -24		101	pH		4.1			13:00	KN	
HOUR -24		102	pH, ORP		4.2	400				
HOUR -24		103	pH		6.08					
HOUR -24		FIT	Total Flow	991						
HOUR -24		FH5	pH		6.6	430				
WEEK 1 (CONTINUOUS)										
DAY 3										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR- 8		101	pH		3.88			21:00	JB	
HOUR- 8		102	pH, ORP		4.1	473	1290			
HOUR - 8		103	pH		596					
HOUR- 8		FIT	Total Flow	1213.6						
HOUR -16		101	pH		3.82					
HOUR -16		102	pH, ORP		4.1	455	1210	5:00	RZ	
HOUR -16		103	pH		6.27					

**Table B-1. Ferrihydrate adsorption process demonstration field data record**

HOUR -16		FIT	Total Flow	1492.2						
HOUR -24		101	pH		4.04		1250	13:00	MGL	
HOUR -24		102	pH, ORP		4.12	--				
HOUR -24		103	pH		6.39					
HOUR -24		FIT	Total Flow	1777.95						
HOUR -24		FH5	pH		6.55					
<b>WEEK 1 (CONTINUOUS)</b>										
<b>DAY 4</b>										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR- 8		101	pH		3.77			21:00	JB	
HOUR- 8		102	pH, ORP		4	471	1310			
HOUR - 8		103	pH		6.18					
HOUR- 8		FIT	Total Flow	2039.2						
HOUR -16		101	pH		3.85			5:00	RZ	
HOUR -16		102	pH, ORP		4.08	492				
HOUR -16		103	pH		6.07					
HOUR -16		FIT	Total Flow	2340.56			1240			
HOUR -24		101	pH		3.96	675		12:00	KN	
HOUR -24		102	pH, ORP		3.96	462				
HOUR -24		103	pH		5.97	535				
HOUR -24		FIT	Total Flow	2557						
HOUR -24		FH5	pH		--	--				
<b>WEEK 1 (CONTINUOUS)</b>										
<b>DAY 5</b>										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR- 8		101	pH		3.87		1250	21:00	JB	FH2 Sample Port
HOUR- 8		102	pH, ORP		4.1	515				
HOUR - 8		103	pH		6.14					
HOUR- 8		FIT	Total Flow	2840						
HOUR -16		101	pH		3.94		1140	5:00	RZ	FH2 Sample Port
HOUR -16		102	pH, ORP		4.1	514				
HOUR -16		103	pH		6.02					
HOUR -16		FIT	Total Flow	3091.24						
HOUR -24		101	pH		4		1550	13:00	MGL	FH2 Sample Port
HOUR -24		102	pH, ORP		4.11	399				
HOUR -24		103	pH		6.13					
HOUR -24		FIT	Total Flow	3233						
HOUR -24		FH5	pH		--	--				

**Table B-1. Ferrihydrite adsorption process demonstration field data record**

WEEK 1 (CONTINUOUS)										
DAY 6										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR - 6		101	pH		3.87		1160	19:00	JB	FH2 Sample Port
HOUR - 6		102	pH, ORP		4.1	403				
HOUR - 6		103	pH		6.1					
HOUR - 6		FIT	Total Flow	3380						
WEEK 1 (CONTINUOUS)										
DAY 7										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR -6		101	pH		3.83					
HOUR -6		102	pH, ORP		4.22	--				
HOUR -6		103	pH		7.12					pump 106 is stopped/probe uncovered/caused excess lime
HOUR -6		FIT	Total Flow	4243						
HOUR -24		FH5	pH		5.92	505				
WEEK 2 (CONTINUOUS)										
DAY 1										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR -6		101	pH		3.84			19:00	JB	
HOUR -6		102	pH, ORP		4	419				
HOUR -6		103	pH		6.33					
HOUR -6		FIT	Total Flow	5030						
HOUR -24		FH5	pH		6.06	495				
WEEK 2 (CONTINUOUS)										
DAY 2										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR - 6		101	pH		3.43			20:00	JB	18:00 samples taken 20:00 (plugged filters/problems w/filter cake)
HOUR - 6		102	pH, ORP		4.3	493				
HOUR - 6		103	pH		6.41					
HOUR - 6		FIT	Total Flow	5889						
WEEK 2 (CONTINUOUS)										
DAY 3										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR -6		101	pH		4.04			18:00	JB	
HOUR -6		102	pH, ORP		4.01	396				
HOUR -6		103	pH		6.21					
HOUR -6		FIT	Total Flow	6614						
HOUR -24		FH5	pH		--	--				

**Table B-1. Ferrihydrite adsorption process demonstration field data record**

WEEK 2 (CONTINUOUS)										
DAY 4										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR - 6		101	pH		3.93		2290	18:00	JB	
HOUR - 6		102	pH, ORP		3.9	464				
HOUR - 6		103	pH		6.59					
HOUR - 6		FIT	Total Flow	7403						
HOUR -24		FH5	pH		6.82	200				
WEEK 2 (CONTINUOUS)										
DAY 5										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR -6		101	pH		3.89			18:00	JB	
HOUR -6		102	pH, ORP		3.8					
HOUR -6		103	pH		608					
HOUR -6		FIT	Total Flow	8183						Acid Leak
HOUR -24		FH5	pH		--	--				
WEEK 2 (CONTINUOUS)										
DAY 6										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR - 6		101	pH		4.25			20:30	JB	Data collected at 20:30 due to error
HOUR - 6		102	pH, ORP		--	537	3310			FH2 Sample Port
HOUR - 6		103	pH		6.14					
HOUR - 6		FIT	Total Flow	9040						
HOUR -24		FH5	pH		5.89	480	2.68			FH4 Sample Port
WEEK 2 (CONTINUOUS)										
DAY 7										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR -6		101	pH		4.42			18:00	JB	
HOUR -6		102	pH, ORP		--	537	2750			FH2 Sample Port
HOUR -6		103	pH		651					
HOUR -6		FIT	Total Flow	9608						
HOUR -24		FH5	pH		6.29	290				
WEEK 3 (CONTINUOUS)										
DAY 1										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR -6		101	pH		4.11			18:10	JB	
HOUR -6		102	pH, ORP		3.9	526	2510			FH2 Sample Port
HOUR -6		103	pH		5.62					
HOUR -6		FIT	Total Flow	310						
HOUR -24		FH5	pH		6.1	355				

**Table B-1. Ferrihydrite adsorption process demonstration field data record**

WEEK 3 (CONTINUOUS)										
DAY 2										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR - 6		101	pH		4.13			18:00	JB	
HOUR - 6		102	pH, ORP		4.2	522	2400			
HOUR - 6		103	pH		568					
HOUR - 6		FIT	Total Flow	1050						
HOUR -24		FH5	pH		6.7	510				
WEEK 3 (CONTINUOUS)										
DAY 3										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR -6		101	pH		3.98			16:30	JB	
HOUR -6		102	pH, ORP		--	549				
HOUR -6		103	pH		6.07		2210			
HOUR -6		FIT	Total Flow	1816						
HOUR -24		FH5	pH		6.06	295		no time	JM	
WEEK 3 (CONTINUOUS)										
DAY 4										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR - 6		101	pH		3.61			18:10	JB	
HOUR - 6		102	pH, ORP		--	455				
HOUR - 6		103	pH		5.95		4020			FH2 Sample Port
HOUR - 6		FIT	Total Flow	2577						
HOUR -24		FH5	pH		6.25	290				
WEEK 3 (CONTINUOUS)										
DAY 5										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR -6		101	pH		3.68			19:30	JB	
HOUR -6		102	pH, ORP			455	3280			FH2 Sample Port
HOUR -6		103	pH		621					
HOUR -6		FIT	Total Flow	3394						
HOUR -24		FH5	pH		7.3	300				
11/12/99		101	pH		7.56			18:00	JM	No sample taken because no water to sample
		102	pH, ORP		2	640				
		103	pH		4.13					
		FIT	Total Flow							
WEEK 3 (CONTINUOUS)										
DAY 6										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR - 6		101	pH		3.64			16:00	JM	
HOUR - 6		102	pH, ORP		--	456				

**Table B-1. Ferrihydrite adsorption process demonstration field data record**

HOUR - 6		103	pH		7.3					
HOUR - 6		FIT	Total Flow	4059.2						
HOUR -24		FH5	pH		--	--				
<b>WEEK 3 (CONTINUOUS)</b>										
<b>DAY 7 FINAL</b>										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR -6		101	pH							Hour 6 samples not collected
HOUR -6		102	pH, ORP							
HOUR -6		103	pH							
HOUR -6		FIT	Total Flow							
HOUR -24		101	pH		--	--		12:00	MGL	
HOUR -24		102	pH, ORP		--	--				
HOUR -24		103	pH		--	--				
HOUR -24		FIT	Total Flow	--						
<b>WEEK 3 (CONTINUOUS)</b>										
<b>DAY 7 FINAL</b>										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR -24		FH5	pH	--				12:00	MGL	
<b>WEEK 4 (CONTINUOUS)</b>										
<b>DAY 1 -- 11/14/99</b>										
Sample Time	Sample Number	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Sampled Time	Initials	Comments
HOUR -6		101	pH		--			18:00	KN	
HOUR -6		102	pH, ORP		4.05	455				
HOUR -6		103	pH		5.87					
HOUR -6		FIT	Total Flow	6432						
HOUR -24		FH5	pH							

**Table B-2. Selenium demonstration test—ferrihydrite process analytical data summary**

Lab #	Sample Description	Collection Date	Collection Time	Submission Date	Analyte CRDL Units	TDS 20 mg/L	TSS 3 mg/L	Iron 0.3 mg/L	Ferrous 0.5 mg/L	Ferric 0.5 mg/L	Calcium 1 mg/L	Magnesium 1 mg/L	Sodium 1 mg/L	Nitrate 0.2 mg/L	Sulfate 5 mg/L	Arsenic 10 ug/L	Barium 10 ug/L	Copper 10 ug/L	Iron 300 ug/L	Molybdenum 10 ug/L	Selenium 10 ug/L	Hydride 2 ug/L	Selenate 2 ug/L	Selenite 2 ug/L	
Low Iron Test; Fe:Se mole ratio = 921:1																									
AH26360	MSE\FH1-001	10/21/99	8:55	10/21/99				5.9			111	46.7	360	4.1	267	16	44	15		130	1550				
AH26361	MSE\FH1-001	10/21/99	8:55	10/21/99				0.9	0.7	< 0.5	109	46.7	360			13	44	< 10		130	1550	1633	586	509	
AH26583	MSE\FH1-201	10/23/99	14:00	10/25/99				0.4			120	48	342	4.2	263	22	52	14		144	1570				
AH26584	MSA\FH1-201	10/23/99	14:00	10/25/99				< 0.3	< 0.5	< 0.5	120	48	338			17	50	< 10		122	1570	1840	473	172	
AH26585	MSA\FH2-202	10/23/99	14:00	10/25/99							117	47	336			< 10	89	210	180000 0	68	1510				
AH26586	MSA\FH2-202	10/23/99	14:00	10/25/99							115	47	334			< 10	85	189	180000 0	61	1510				
AH26587	MSA\FH3-203	10/23/99	14:00	10/25/99							1310	51	356			< 10	135	233	200000 0	20	350				
AH26588	MSA\FH3-203	10/23/99	14:00	10/25/99							1310	51	356			< 10	129	208	655	< 10	186				
AH26589	MSA\FH3-206	10/23/99	17:00	10/25/99																	247				
AH26590	MSA\FH3-207	10/23/99	21:00	10/25/99																	266				
AH26591	MSA\FH3-208	10/24/99	1:00	10/25/99																	254				
AH26592	MSA\FH3-209	10/24/99	5:00	10/25/99																	262				
AH26593	MSA\FH5-210	10/24/99	9:00	10/25/99																	402				
AH26594	MSA\FH3-211	10/24/99	13:00	10/25/99																	278				
AH26595	MSA\FH5-212	10/24/99	13:00	10/25/99																	194				
AH26596	MSA\FH5-213	10/24/99	17:00	10/25/99																	265				
AH26597	MSA\FH5-214	10/24/99	19:00	10/25/99																		243	133	30	
AH26598	MSA\FH5-215	10/24/99	21:00	10/25/99																	267				
AH26599	MSA\FH5-216	10/25/99	1:00	10/25/99																	297				
AH26600	MSA\FH5-217	10/25/99	5:00	10/25/99																	336				
AH26656	MSE\FH5-218	10/25/99	9:00	10/26/99																	330				
AH26657	MSE\FH1-219	10/25/99	13:00	10/26/99							122	49.1	355	4.1	266	13	52	18	513	135	1500				
AH26658	MSE\FH1-219	10/25/99	13:00	10/26/99							122	48.7	355			13	51	< 10	< 300	118	1500				
AH26659	MSE\FH2-220	10/25/99	13:00	10/26/99							120	48	350			< 10	150	233	139000 0	75	959				
AH26660	MSE\FH2-220	10/25/99	13:00	10/26/99							120	48	350			< 10	145	180	139000 0	75	885				
AH26661	MSE\FH3-221	10/25/99	13:00	10/26/99							730	340	340			< 10	98	185	129000 0	60	1150				
AH26662	MSE\FH3-221	10/25/99	13:00	10/26/99							730	340	340			< 10	74	77	< 300	< 10	458				
AH26663	MSE\FH4-222	10/25/99	13:00	10/26/99							834	296	340			< 10	80	113	4110	< 10	300				
AH26664	MSE\FH4-222	10/25/99	13:00	10/26/99							834	292	339			< 10	80	47	< 300	< 10	300				
AH26665	MSE\FH5-223	10/25/99	13:00	10/26/99							840	296	340	5.8	31	< 10	80	25	347	< 10	300				
AH26666	MSE\FH5-223	10/25/99	13:00	10/26/99							840	294	340			< 10	80	25	< 300	< 10	300				
AH26667	MSE\FH4-224	10/25/99	19:00	10/26/99		6140	11																		
AH26668	MSE\FH5-225	10/25/99	19:00	10/26/99		6040	13															333	179	28	
AH26669	MSE\FH5-226	10/25/99	21:00	10/26/99																	347				
AH26670	MSE\FH5-227	10/26/99	5:00	10/26/99																	363				
AH26721	MSE\FH3-228	10/26/99	13:00	10/27/99												18			< 300		358				
AH26722	MSE\FH5-229	10/26/99	13:00	10/27/99												12			< 300		347				
AH26723	MSE\FH8-229	10/26/99	13:00	10/27/99												11			< 300		358				
AH26724	MSE\FH9-229	10/26/99	13:00	10/27/99												< 10			< 300		< 10				
AH26725	MSE\FH5-230	10/26/99	21:00	10/27/99																	342				
AH26726	MSE\FH5-231	10/27/99	5:00	10/27/99																	345				
AH26727	MSE\FH5-005	10/26/99	19:00	10/27/99																		340	223	24	
AH26837	MSE\FH1-232	10/27/99	13:00	10/28/99							116	48.6	365	4.2	261	15	35	16	< 300	82	1060				
AH26838	MSE\FH1-232	10/27/99	13:00	10/28/99							115	48.4	365			15	32	< 10	< 300	77	1023				
AH26839	MSE\FH2-233	10/27/99	13:00	10/28/99							115	46.8	355			< 10	94	210	134000 0	45	980				
AH26840	MSE\FH2-233	10/27/99	13:00	10/28/99							115	46.5	355			< 10	88	159	180000 0	44	949				
AH26841	MSE\FH3-234	10/27/99	13:00	10/28/99							1190	110	370			< 10	87	159	841000 0	38	833				
AH26842	MSE\FH3-234	10/27/99	13:00	10/28/99							1190	110	370			< 10	61	103	< 300	< 10	400				

**Table B-2. Selenium demonstration test—ferrihydrite process analytical data summary**

Lab #	Sample Description	Collection Date	Collection Time	Submission Date	Analyte CRDL Units	TDS 20 mg/L	TSS 3 mg/L	Iron 0.3 mg/L	Ferrous 0.5 mg/L	Ferric 0.5 mg/L	Calcium 1 mg/L	Magnesium 1 mg/L	Sodium 1 mg/L	Nitrate 0.2 mg/L	Sulfate 5 mg/L	Arsenic 10 ug/L	Barium 10 ug/L	Copper 10 ug/L	Iron 300 ug/L	Molybdenum 10 ug/L	Selenium 10 ug/L	Hydride 2 ug/L	Selenate 2 ug/L	Selenite 2 ug/L	
AH26843	MSE\FH4-235	10/27/99	13:00	10/28/99							1140	149	381			< 10	74	182	1630	< 10	300				
AH26844	MSE\FH4-235	10/27/99	13:00	10/28/99							1140	148	376			< 10	58	49	< 300	< 10	300				
AH26845	MSE\FH5-236	10/27/99	13:00	10/28/99							1140	150	381	4.2	31	< 10	80	65	1080	< 10	300				
AH26846	MSE\FH5-236	10/27/99	13:00	10/28/99							1120	148	379			< 10	80	40	< 300	< 10	300				
AH26847	MSE\FH5-237	10/27/99	19:00	10/28/99																		329	266	21	
AH26848	MSE\FH5-238	10/27/99	21:00	10/28/99																		264			
AH26849	MSE\FH5-239	10/28/99	5:00	10/28/99																		334			
AH26961	MSE\FH3-240	10/28/99	13:00	10/29/99												< 10			459		261				
AH26962	MSE\FH5-241	10/28/99	13:00	10/29/99												< 10			52		325				
AH26963	MSE\FH5-242	10/28/99	19:00	10/29/99							1230	115	335			< 10	69	27	40	11	350	278	156	22	
AH27037	MSE\FH1-243	10/29/99	13:00	11/1/99							117	48	342	4.2	267	17	68	14	< 300	145	1440				
AH27038	MSE\FH1-243	10/29/99	13:00	11/1/99							117	48	342			17	56	14	< 300	145	1440				
AH27039	MSE\FH2-244	10/29/99	13:00	11/1/99							115	46.1	327			< 10	92	130	166000 0	74	1200				
AH27040	MSE\FH3-245	10/29/99	13:00	11/1/99							1700	60	335			< 10	97	155	138000 0	64	1030				
AH27041	MSE\FH3-245	10/29/99	13:00	11/1/99							1700	60	335			< 10	60	102	582	< 10	232				
AH27042	MSE\FH4-246	10/29/99	13:00	11/1/99							1800	68.7	342			< 10	62	103	2470	< 10	227				
AH27043	MSE\FH4-246	10/29/99	13:00	11/1/99							1760	68.1	340			< 10	61	41	< 300	< 10	239				
AH27044	MSE\FH5-247	10/29/99	13:00	11/1/99							1790	69	344	4.1	10	< 10	64	69	1480	< 10	230				
AH27045	MSE\FH5-247	10/29/99	13:00	11/1/99							1790	68.9	344			< 10	60	31	< 300	< 10	230				
AH27046	MSE\FH5-248	10/29/99	19:00	11/1/99												< 10			< 300		240				
AH27047	MSE\FH3-249	10/30/99	13:00	11/1/99												< 10			583		222				
AH27048	MSE\FH5-250	10/30/99	13:00	11/1/99												< 10			< 300		256				
AH27049	MSE\FH5-251	10/30/99	19:00	11/1/99																	245				
AH27050	MSE\FH1-252	10/31/99	12:00	11/1/99							119	49.2	347	4.5	248	< 10	51	16	< 300	118	1450				
AH27051	MSE\FH1-252	10/31/99	12:00	11/1/99							118	48.7	347			10	47	< 10	< 300	114	1450				
AH27052	MSE\FH2-253	10/31/99	12:00	11/1/99							115	46.3	331			< 10	85	139	165000 0	66	1200				
AH27053	MSE\FH3-254	10/31/99	12:00	11/1/99							1200	230	340			< 10	86	135	985000	46	817				
AH27054	MSE\FH3-254	10/31/99	12:00	11/1/99							1200	230	340			< 10	61	97	654	< 10	239				
AH27055	MSE\FH5-256	10/31/99	12:00	11/1/99							1800	72.5	353	4	13	< 10	62	50	2800	< 10	240				
AH27056	MSE\FH5-256	10/31/99	12:00	11/1/99							1800	72.4	353			< 10	61	15	< 300	< 10	240				
AH27057	MSE\FH4-255	10/31/99	12:00	11/1/99							1750	74	350			< 10	61	86	4990	< 10	240				
AH27058	MSE\FH4-255	10/31/99	12:00	11/1/99							1750	74	350			< 10	59	20	< 300	< 10	240				
AH27059	MSE\FH5-257	10/31/99	20:15	11/1/99																	115				
AH27062	MSE\FH FILTRATE-221	10/31/99	17:45	11/1/99							1500	244	350	5.6	273	99	96	105	213000	< 10	1250				
AH27063	MSE\FH FILTRATE-221	10/31/99	17:45	11/1/99							1500	244	350			< 10	69	38	< 300	< 10	1190	1480	911	40	
AH27151	MSE\FH3-258	11/1/99	12:00	11/2/99												< 10			455		386				
AH27152	MSE\FH5-259	11/1/99	12:00	11/2/99												< 10			< 300		377				
<b>Medium Iron Test; Fe/Se mole ratio = 1945:1</b>																									
AH27153	MSE\FH4-260	11/1/99	18:00	11/2/99		7200	19																		
AH27154	MSE\FH5-261	11/1/99	18:00	11/2/99		6900	< 3																		
AH27155	MSE\FH5-261	11/1/99	18:00	11/2/99																	452	380	197	31	
AH27418	MSE\FH1-262	11/2/99	12:00	11/3/99							118	48.7	350	4	278	13	57	15	< 300	128	1550				
AH27419	MSE\FH1-262	11/2/99	12:00	11/3/99							118	48.6	350			< 10	50	< 10	< 300	115	1550				
AH27420	MSE\FH2-263	11/2/99	12:00	11/3/99							113	46.9	343			< 10	72	107	161000 0	52	1020				
AH27421	MSE\FH3-264	11/2/99	12:00	11/3/99							830	395	350			< 10	78	105	140000 0	38	886				
AH27422	MSE\FH3-264	11/2/99	12:00	11/3/99							830	395	350			< 10	63	50	828	< 10	380				
AH27423	MSE\FH4-265	11/2/99	12:00	11/3/99							982	346	354			< 10	65	27	1790	< 10	400				
AH27424	MSE\FH4-265	11/2/99	12:00	11/3/99							965	343	348			< 10	65	< 10	< 300	< 10	400				
AH27425	MSE\FH5-266	11/2/99	12:00	11/3/99							976	347	353	4	52	< 10	52	< 10	1210	< 10	343				
AH27426	MSE\FH5-266	11/2/99	12:00	11/3/99							964	342	343			< 10	49	< 10	< 300	< 10	339				
AH27427	MSE\FH8-266	11/2/99	12:00	11/3/99							966	340	346			< 10	64	< 10	< 300	< 10	439				

**Table B-2. Selenium demonstration test—ferrihydrite process analytical data summary**

Lab #	Sample Description	Collection Date	Collection Time	Submission Date	Analyte CRDL Units	TDS 20 mg/L	TSS 3 mg/L	Iron 0.3 mg/L	Ferrous 0.5 mg/L	Ferric 0.5 mg/L	Calcium 1 mg/L	Magnesium 1 mg/L	Sodium 1 mg/L	Nitrate 0.2 mg/L	Sulfate 5 mg/L	Arsenic 10 ug/L	Barium 10 ug/L	Copper 10 ug/L	Iron 300 ug/L	Molybdenum 10 ug/L	Selenium 10 ug/L	Hydride 2 ug/L	Selenate 2 ug/L	Selenite 2 ug/L
AH27428	MSE\FH9-266	11/2/99	12:00	11/3/99		< 1					< 1	< 1	< 1			< 10	< 10	< 10	< 300	14	< 10			
AH27429	MSE\FH5-267	11/2/99	18:00	11/3/99																	438			
AH27525	MSE\FH3-268	11/3/99	12:00	11/4/99												< 10			2560		42			
AH27526	MSE\FH5-269	11/3/99	12:00	11/4/99												< 10			< 300		415			
AH27527	MSE\FH5-270	11/3/99	20:00	11/4/99																	179			
AH27650	MSE\FH1-271	11/4/99	12:00	11/5/99							120	50	370	3.5	512	15	61	18	< 300	125	1580			
AH27651	MSE\FH1-271	11/4/99	12:00	11/5/99							120	50	370			10	51	12	< 300	125	1450			
AH27652	MSE\FH2-272	11/4/99	12:00	11/5/99							114	46.9	351			< 10	121	276	2270000	< 10	75			
AH27653	MSE\FH3-273	11/4/99	12:00	11/5/99							1600	750	360			< 10	112	191	1410000	< 10	650			
AH27654	MSE\FH3-273	11/4/99	12:00	11/5/99							1600	750	360			< 10	61	14	310	< 10	369			
AH27655	MSE\FH4-274	11/4/99	12:00	11/5/99							1710	786	362			< 10	74	56	6010	< 10	140			
AH27656	MSE\FH4-274	11/4/99	12:00	11/5/99							1710	777	362			< 10	71	< 10	< 300	< 10	140			
AH27657	MSE\FH5-275	11/4/99	12:00	11/5/99							1680	750	343	3.6	18	< 10	77	61	9270	< 10	144			
AH27658	MSE\FH5-275	11/4/99	12:00	11/5/99							1640	736	340			< 10	69	< 10	< 300	< 10	142			
AH27659	MSE\FH5-276	11/4/99	18:00	11/5/99																	154			
AH27735	MSE\FH3-277	11/5/99	12:00	11/8/99												< 10			345		150			
AH27736	MSE\FH5-278	11/5/99	12:00	11/8/99												< 10			< 300		174			
AH27737	MSE\FH5-279	11/5/99	18:00	11/8/99																	217			
AH27738	MSE\FH1-280	11/6/99	12:00	11/8/99							120	48.4	400	3.8	268	< 10	50	30	< 300	120	1440			
AH27739	MSE\FH1-280	11/6/99	12:00	11/8/99							111	45.8	392			< 10	38	< 10	< 300	91	1240			
AH27740	MSE\FH2-281	11/6/99	12:00	11/8/99							106	42	368			< 10	85	222	3300000	118	69			
AH27741	MSE\FH2-281	11/6/99	12:00	11/8/99							106	42	368			< 10	85	220	3300000	15	27			
AH27742	MSE\FH3-282	11/6/99	12:00	11/8/99							2760	170	380			< 10	160	255	1980000	64	652			
AH27743	MSE\FH3-282	11/6/99	12:00	11/8/99							2760	170	380			< 10	150	65	1630	< 10	160			
AH27744	MSE\FH4-283	11/6/99	12:00	11/8/99							2760	451	387			< 10	73	103	8730	< 10	188			
AH27745	MSE\FH4-283	11/6/99	12:00	11/8/99							2680	441	335			< 10	69	46	311	< 10	157			
AH27746	MSE\FH5-284	11/6/99	12:00	11/8/99							2680	453	338	3.7	27	< 10	74	101	2510	< 10	189			
AH27747	MSE\FH5-284	11/6/99	12:00	11/8/99							2680	452	337			< 10	62	45	< 300	< 10	163			
AH27748	MSE\FH5-285	11/6/99	18:00	11/8/99																	138			
AH27749	MSE\FH3-286	11/7/99	12:00	11/8/99												< 10			950		48			
AH27750	MSE\FH5-287	11/7/99	12:00	11/8/99												< 10			< 300		133			
AH27751	MSE\FH5-288	11/7/99	18:00	11/8/99																	130			
AH27863	MSE\FH1-289	11/8/99	12:00	11/9/99							122	48.5	348	4.2	256	13	52	39	< 300	121	1520			
AH27864	MSE\FH1-289	11/8/99	12:00	11/9/99							113	46.5	341			11	40	11	< 300	100	1430			
AH27865	MSE\FH2-290	11/8/99	12:00	11/9/99							107	43	330			< 10	115	296	2960000	118	77			
AH27866	MSE\FH2-290	11/8/99	12:00	11/9/99							107	43	330			< 10	115	279	2100	118	73			
AH27867	MSE\FH3-291	11/8/99	12:00	11/9/99							2500	59.3	348			< 10	140	330	2960000	119	1100			
AH27868	MSE\FH3-291	11/8/99	12:00	11/9/99							2500	58.2	344			< 10	67	150	2290	< 10	1050			
AH27869	MSE\FH4-292	11/8/99	12:00	11/9/99							2500	82	350			< 10	76	145	< 300	< 10	158			
AH27870	MSE\FH4-292	11/8/99	12:00	11/9/99							2500	82	350			< 10	66	110	< 300	< 10	152			
AH27871	MSE\FH5-293	11/8/99	12:00	11/9/99							2500	85	348	3.8	15	< 10	76	171	3770	< 10	159			
AH27872	MSE\FH5-293	11/8/99	12:00	11/9/99							2500	85	348			< 10	65	108	303	< 10	154			
AH27873	MSE\FH4-294	11/8/99	18:00	11/9/99		9400	6																	
AH27874	MSE\FH5-295	11/8/99	18:00	11/9/99		10200	6																	
AH27875	MSE\FH5-295	11/8/99	18:00	11/9/99																	173	128	30	18
<b>HighIron Test; Fe/Se mole ratio = 3186:1</b>																								
AH27940	MSE\FH3-296	11/9/99	12:00	11/10/99												< 10			973		54			
AH27941	MSE\FH5-297	11/9/99	12:00	11/10/99												< 10			313		124			
AH27942	MSE\FH8-297	11/9/99	12:00	11/10/99												< 10			338		125			

**Table B-2. Selenium demonstration test—ferrihydrite process analytical data summary**

Lab #	Sample Description	Collection Date	Collection Time	Submission Date	Analyte CRDL Units	TDS 20 mg/L	TSS 3 mg/L	Iron 0.3 mg/L	Ferrous 0.5 mg/L	Ferric 0.5 mg/L	Calcium 1 mg/L	Magnesium 1 mg/L	Sodium 1 mg/L	Nitrate 0.2 mg/L	Sulfate 5 mg/L	Arsenic 10 ug/L	Barium 10 ug/L	Copper 10 ug/L	Iron 300 ug/L	Molybdenum 10 ug/L	Selenium 10 ug/L	Hydride 2 ug/L	Selenate 2 ug/L	Selenite 2 ug/L
AH27943	MSE\FH9-297	11/9/99	12:00	11/10/99												< 10			< 300		< 10			
AH27944	MSE\FH5-298	11/9/99	18:00	11/10/99																	111			
AH28108	MSE\FH1-299	11/10/99	12:00	11/11/99							120	49.3	379	4	254	12	50	< 10	< 300	88	1100			
AH28109	MSE\FH1-299	11/10/99	12:00	11/11/99							120	47.9	340			12	50	< 10	< 300	88	1100			
AH28110	MSE\FH2-300	11/10/99	12:00	11/11/99							109	42.9	313			< 10	129	378	480000	193	49			
AH28111	MSE\FH2-300	11/10/99	12:00	11/11/99							109	42.6	311			< 10	98	197	480000	119	43			
AH28112	MSE\FH3-301	11/10/99	12:00	11/11/99							2920	62.6	335			< 10	137	398	423000	120	563			
AH28113	MSE\FH3-301	11/10/99	12:00	11/11/99							2920	62.1	335			< 10	86	191	4030	< 10	79			
AH28114	MSE\FH4-302	11/10/99	12:00	11/11/99							2920	66	335			< 10	66	198	19800	< 10	50			
AH28115	MSE\FH4-302	11/10/99	12:00	11/11/99							2920	66	335			< 10	59	187	632	< 10	30			
AH28116	MSE\FH5-303	11/10/99	12:00	11/11/99							2920	66	340	3.5	< 5	< 10	94	219	20000	< 10	60			
AH28117	MSE\FH5-303	11/10/99	12:00	11/11/99							2920	66	340			< 10	72	185	472	< 10	60	43		
AH28118	MSE\FH5-304	11/10/99	18:00	11/11/99																	64			
AH28241	MSE\FH3-305	11/11/99	12:00	11/12/99												< 10			4880		35			
AH28242	MSE\FH5-306	11/11/99	12:00	11/12/99												< 10			333		93			
AH28243	MSE\FH5-307	11/11/99	12:00	11/12/99																	147			
<b>Ferrous/Ferric Test</b>																								
AH28304	MSE\FH1-308	11/12/99	11:00	11/15/99							114	48.1	346	4	237	15	50	< 10	< 300	80	994			
AH28305	MSE\FH1-308	11/12/99	11:00	11/15/99							114	48.1	345			15	50	< 10	< 300	78	990			
AH28306	MSE\FH2-309	11/12/99	11:00	11/15/99			412				116	46.9	350			< 10	63	43		127	1460			
AH28307	MSE\FH2-309	11/12/99	11:00	11/15/99			364	116	248															
AH28308	MSE\FH3-310	11/12/99	11:00	11/15/99							740	50	350			< 10	88	110	135000	124	1330			
AH28309	MSE\FH3-310	11/12/99	11:00	11/15/99							740	50	350			< 10	68	100	500000	< 10	652			
AH28310	MSE\FH4-311	11/12/99	11:00	11/15/99							2780	56.4	356			< 10	72	182	52600	< 10	451			
AH28311	MSE\FH4-311	11/12/99	11:00	11/15/99							2780	56.2	353			< 10	72	181	47100	< 10	432			
AH28312	MSE\FH5-312	11/12/99	11:00	11/15/99							2780	58	355	3.5	126	< 10	73	222	36900	< 10	409			
AH28313	MSE\FH5-312	11/12/99	11:00	11/15/99							2780	58	355			< 10	72	191	18700	< 10	403			
AH28314	MSE\FH3-360	11/13/99	12:00	11/15/99												< 10			29900		664			
AH28315	MSE\FH5-361	11/13/99	12:00	11/15/99												< 10			15800		706			
AH28316	MSE\FH5-362	11/13/99	18:00	11/15/99																	758			
AH28317	MSE\FH5-314	11/14/99	12:00	11/15/99																	852			
AH28318	MSE\FH1-315	11/14/99	12:00	11/15/99			< 0.3				118	48	355	4	245	12	49	< 10		122	1500			
AH28319	MSE\FH1-315	11/14/99	12:00	11/15/99			< 0.3	< 0.5	< 0.5		117	48	355			< 10	49	< 10		115	1500	1440	1210	142
AH28320	MSE\FH2-316	11/14/99	12:00	11/15/99							115	45	337			< 10	110	15	295000	60	79			
AH28321	MSE\FH2-316	11/14/99	12:00	11/15/99							115	45	335			< 10	110	14	295000	< 10	77			
AH28322	MSE\FH3-317	11/14/99	12:00	11/15/99							1220	51	346			< 10	97	< 10	189000	74	1170			
AH28323	MSE\FH3-317	11/14/99	12:00	11/15/99							1220	51	346			< 10	84	< 10	754000	< 10	918			
AH28324	MSE\FH4-318	11/14/99	12:00	11/15/99		5920	128				1340	52.2	350			< 10	73	17	814000	< 10	800			
AH28325	MSE\FH4-318	11/14/99	12:00	11/15/99							1330	51.8	347			< 10	73	< 10	711000	< 10	800			
AH28326	MSE\FH8-319	11/14/99	12:00	11/15/99							1380	52.7	351			< 10	< 10	< 10	537000	< 10	825	961	265	202
AH28327	MSE\FH9-319	11/14/99	12:00	11/15/99							< 1	< 1	< 1			< 10	78	538	< 300	< 10	< 10	13	< 2	13
AH28328	MSE\FH5-319	11/14/99	12:00	11/15/99		5840	122				1360	53	347	2.6	1230	< 10	92	634	746000	< 10	800			
AH28329	MSE\FH5-319	11/14/99	12:00	11/15/99							1360	53	347			< 10	78	145	593000	< 10	800	935	364	126
AH28330	MSE\FH3-320	11/14/99	18:00	11/15/99																	822			
<b>Recycle Fe Sludge Test</b>																								
AH28425	MSE\FH3-321	11/15/99	12:00	11/16/99																	515			
AH28426	MSE\FH5-322	11/15/99	12:00	11/16/99																	879			
AH28427	MSE\FH3-323	11/15/99	18:00	11/16/99																	190			
AH28428	MSE\FH5-324	11/15/99	18:00	11/16/99																	747			
AH28429	MSE\FH3-325	11/15/99	0:00	11/16/99																	222			

**Table B-2. Selenium demonstration test—ferrihydrite process analytical data summary**

Lab #	Sample Description	Collection Date	Collection Time	Submission Date	Analyte CRDL Units	TDS 20 mg/L	TSS 3 mg/L	Iron 0.3 mg/L	Ferrous 0.5 mg/L	Ferric 0.5 mg/L	Calcium 1 mg/L	Magnesium 1 mg/L	Sodium 1 mg/L	Nitrate 0.2 mg/L	Sulfate 5 mg/L	Arsenic 10 ug/L	Barium 10 ug/L	Copper 10 ug/L	Iron 300 ug/L	Molybdenum 10 ug/L	Selenium 10 ug/L	Hydride 2 ug/L	Selenate 2 ug/L	Selenite 2 ug/L	
AH28430	MSE\FH5-326	11/15/99	0:00	11/16/99																	695				
AH28431	MSE\FH3-327	11/16/99	6:00	11/16/99																	195				
AH28432	MSE\FH5-328	11/16/99	6:00	11/16/99																	200				
AH28503	MSE\FH3-329	11/16/99	12:00	11/17/99																	231				
AH28504	MSE\FH5-330	11/16/99	12:00	11/17/99																	369				
AH28505	MSE\FH3-331	11/16/99	18:00	11/17/99																	214				
AH28506	MSE\FH5-332	11/16/99	18:00	11/17/99																	288				
AH28507	MSE\FH3-333	11/16/99	0:00	11/17/99																	220				
AH28508	MSE\FH5-334	11/16/99	0:00	11/17/99																	275				
AH28509	MSE\FH3-335	11/17/99	6:00	11/17/99																	225				
AH28510	MSE\FH5-336	11/17/99	6:00	11/17/99																	266				
AH28677	MSE\FH3-337	11/17/99	12:00	11/18/99																	283				
AH28678	MSE\FH5-338	11/17/99	12:00	11/18/99																	230				
AH28679	MSE\FH3-339	11/17/99	18:00	11/18/99																	185				
AH28680	MSE\FH5-340	11/17/99	18:00	11/18/99																	239				
AH28681	MSE\FH3-341	11/17/99	0:00	11/18/99																	103				
AH28682	MSE\FH5-342	11/17/99	0:00	11/18/99																	231				
AH28683	MSE\FH5-343	11/18/99	6:00	11/18/99																	231				
AH28684	MSE\FH1-344	11/18/99	6:00	11/18/99			< 0.3			127	50.2	350		3.9	248	12	53	< 10		136	1570				
AH28685	MSE\FH1-344	11/18/99	6:00	11/18/99			< 0.3	< 0.5	< 0.5	122	49.9	350				< 10	43	< 10		103	1280	1600	1400	134	
AH28686	MSE\FH2-345	11/18/99	6:00	11/18/99						541	45	320				< 10	201	686	8240000	< 10	89				
AH28687	MSE\FH2-345	11/18/99	6:00	11/18/99						540	45	320				< 10	90	581	6130000	< 10	< 10				
AH28688	MSE\FH3-346	11/18/99	6:00	11/18/99						2940	55	335				< 10	125	301	56400000	253	1770				
AH28689	MSE\FH3-346	11/18/99	6:00	11/18/99						2900	55	335				< 10	65	213	773	< 10	77				
AH28690	MSE\FH4-347	11/18/99	6:00	11/18/99		5770	16			1740	54.7	350				< 10	50	97	6730	< 10	200				
AH28691	MSE\FH4-347	11/18/99	6:00	11/18/99						1730	54.3	350				< 10	50	97	3450	< 10	200				
AH28692	MSE\FH5-348	11/18/99	6:00	11/18/99		5650	21			1710	55	350	3.6	62	< 10	50	90	6380	< 10	200					
AH28693	MSE\FH5-348	11/18/99	6:00	11/18/99						1710	55	350				< 10	50	82	1670	< 10	200	272	137	22	
AH28694	MSE\FH8-349	11/18/99	6:00	11/18/99						1670	54.5	344				< 10	54	95	1840	< 10	244	163	128	8	
AH28695	MSE\FH9-350	11/18/99	6:00	11/18/99						3.8	< 1	2				< 10	< 10	< 10	< 300	< 10	< 10	< 2	< 2	< 2	
AH29009	MSE\FH3-341	11/18/99		11/22/99																		100			
AH29010	MSE\FH2-345	11/18/99		11/22/99																		61			
AH29011	MSE\FH3-346	11/18/99		11/22/99																		79			
AH29012	MSE\FH9-350	11/18/99		11/22/99																		< 2			

**Table B-3. Summary total metals data**

Lab #	Sample Description	Collection Date	Submission Date	Analyte RL Units	Arsenic 0.5 mg/kg	Barium 5 mg/kg	Calcium 1 mg/kg	Cadmium 0.2 mg/kg	Chromium 1 mg/kg	Copper 1 mg/kg	Iron 1 mg/kg	Mercury 0.01 mg/kg	Lead 0.5 mg/kg	Selenium 0.5 mg/kg	Silver 1 mg/kg	Total Solid 1 %
AH27060	MSE\FH FILTER CAKE -221	10/31/99	11/1/99		21	< 0.5		0.8	72.6	23.6		0.7	< 0.5	< 0.5	< 1	29
AH27767	MSE\CC FILTER CAKE 221	11/6/99	11/8/99		22	< 0.5	1600	1	31.4	638	76400	0.6	< 0.5	< 0.5	< 1	24
AH28433	MSE\CCFILTERCAKE	11/15/99	11/16/99		13	< 0.5	2500	4.7	15.1	3300	11600	0.46	< 0.5	40	< 1	26
AH28671	FH Filter Cake-225	11/18/99	11/18/99		22.7	80	1130	< 0.2	10.5	63.1	29200	0.48	6.4	< 0.5	< 1	51

**Table B-4. Summary toxicity characteristic leachate procedure data**

Lab #	Sample Description	Collection Date	Submission Date	Analyte RL Units	AG-TCLP 0.1 mg/L	AS-TCLP 0.1 mg/L	BA-TCLP 0.1 mg/L	CD-TCLP 0.01 mg/L	CR-TCLP 0.1 mg/L	HG-TCLP 0.001 mg/L	PB-TCLP 0.1 mg/L	SE-TCLP 0.1 mg/L
AH27061	MSE\FH FILTER CAKE -221	10/31/99	11/1/99		0.1	< 0.1	0.1	< 0.1	< 0.1	0.001	< 0.1	1.6
AH27768	MSE\CC FILTER CAKE 221	11/6/99	11/8/99		< 0.1	< 0.1	0.1	< 0.1	< 0.1	0.001	< 0.1	0.3
AH28434	MSE\CC FILTER CAKE	11/15/99	11/16/99		< 0.1	< 0.1	0.1	0.02	< 0.1	0.002	< 0.1	< 0.1
AH28670	FH Filter Cake-225	11/18/99	11/18/99		< 0.1	< 0.1	0.1	0.01	< 0.1	< 0.001	< 0.1	1.1

**Table B-5. Catalyzed cementation process demonstration field data record**

BACKGROUND DAYS										
WEEK 1 10/26/99										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOURL -	CC3		0	6.01	OR	66	1.1	20:10	JB	ORP Over Range
HOURL-	CC5		0	N/A	N/A	N/A	N/A	N/A	MGL	
WEEK 1 (CONTINUOUS)										
DAY 1 INITIAL 10/27/99 Selenium Speciation										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOURL - 0	108	pH, ORP		3.59	-366	1400	2	0:00	JB	
HOURL - 0	109	pH, ORP		6.12	OR			0:00		ORP Over Range
HOURL - 0	FIT	Total Flow	145					0:00		Flow Meter not functioning properly
WEEK 1 (CONTINUOUS)										
DAY 1 INITIAL										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOURL - 4	108	pH, ORP		5.37	-423			5:35	RZ	
HOURL - 4	109	pH, ORP		6.16	OR	3300	58			
HOURL - 4	FIT	Total Flow								
HOURL - 8	108	pH, ORP		5.26	-396	330	2.4	8:30	KN	Cu=3.0 ppm in CC2
HOURL - 8	109	pH, ORP		6.03	-435					
HOURL - 8	FIT	Total Flow	346.81							
HOURL - 12	108	pH, ORP		5.38	-403	280	5.9	12:00	MGL	Cu=5.9 ppm in CCs
HOURL - 12	109	pH, ORP		6.01	-343					
HOURL - 12	FIT	Total Flow	423.38							
HOURL - 16	108	pH, ORP								
HOURL - 16	109	pH, ORP		5.67	-353	320	1.7	16:00	JB	
HOURL - 16	FIT	Total Flow	451.9	6.1	OR					
HOURL - 20	108	pH, ORP		5.01	-396	0.62	0.03	20:00	JB	All on PC5
HOURL - 20	109	pH, ORP		6	OR					
HOURL - 20	FIT	Total Flow	456.9							
HOURL - 24	108	pH, ORP		3.63	OR			0:00	RZ	
HOURL - 24	109	pH, ORP		6.16	OR					
HOURL - 24	FIT	Total Flow	457.61							
HOURL - 24	PC3	ORP			OR					
HOURL - 24	PC5	pH		6.31						
WEEK 1 (CONTINUOUS)										
DAY 2										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOURL - 4	108	pH, ORP		5.34	-406	230	1.2	4:00	RZ	CC3 Sample Port
HOURL - 4	109	pH, ORP		6.05	OR		4			CC2 Sample Port
HOURL - 4	FIT	Total Flow	55.65							
HOURL - 8	108	pH, ORP		4.72	-509	234		8:00	MGL	CC3 Sample Port
HOURL - 8	109	pH, ORP		6.2	-390		7.2			CC2 Sample Port
HOURL - 8	FIT	Total Flow	--							
HOURL - 12	108	pH, ORP		5.34	-512		6.5	13:45	MGL	CC2 Sample Port
HOURL - 12	109	pH, ORP		6.04	-405	157				CC5 Sample Port
HOURL - 12	FIT	Total Flow	--							
HOURL - 16	108	pH, ORP		4.98	-377		0.91	16:00	JB	CC2 Sample Port
HOURL - 16	109	pH, ORP		6.06	OR	180				CC3 Sample Port
HOURL - 16	FIT	Total Flow	--							

**Table B-5. Catalyzed cementation process demonstration field data record**

HOUR -20	108	pH, ORP		4.49	-370		1			CC2 Sample Port
HOUR -20	109	pH, ORP		6.18	OR	110				CC5 Sample Port
HOUR -20	FIT	Total Flow	--			360				CC3 Sample Port
<b>WEEK 1 (CONTINUOUS)</b>										
<b>DAY 2</b>										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR -24	108	pH, ORP		4.45	-344	220	1.4	0:00	RZ	CC3 Sample Port
HOUR -24	109	pH, ORP		6.19	OR	140				CC5 Sample Port
HOUR -24	FIT	Total Flow	--							
HOUR -24	PC3	ORP			--					
HOUR -24	PC5	pH		--						
<b>WEEK 1 (CONTINUOUS)</b>										
<b>DAY 3</b>										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR- 8	108	pH, ORP		5.33	-527		3.84	8:00	MGL	CC2 Sample Port
HOUR- 8	109	pH, ORP		6.06	--	194				CC5 Sample Port
HOUR- 8	FIT	Total Flow	--							
HOUR -16	108	pH, ORP		3.53	-324		1.19	16:00	JB	CC2 Unfiltered
HOUR -16	109	pH, ORP		6.08			1.16			CC3 Filtered
HOUR -16	FIT	Total Flow	--							
HOUR -24	108	pH, ORP		5.04	-368		6.1	0:00	RZ	CC2 Sample Port
HOUR -24	109	pH, ORP		6.2	-298		0.09			CC3 Sample Port
HOUR -24	FIT	Total Flow	--				1			CC5 Sample Port
HOUR -24	PC3	ORP								
HOUR -24	PC5	pH								
<b>WEEK 1 (CONTINUOUS)</b>										
<b>DAY 4</b>										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR- 8	108	pH, ORP		3.3	-319			8:00	MGL	
HOUR- 8	109	pH, ORP		6.07	-290					
HOUR- 8	FIT	Total Flow	--							
HOUR -16	108	pH, ORP		5.17	-330		8	16:00	JB	CC2 Sample Port
HOUR -16	109	pH, ORP		6.13			1.7			CC3 Sample Port
HOUR -16	FIT	Total Flow	--				1.34			CC5 Sample Port
18:30 Added 5 pounds of powdered Iron to reactor tank and increased copper sulfate flow to 90? As per Larry Twidwell										
HOUR -24	108	pH, ORP		4.54	-425		3.0/5.9	0:00	RZ	CC2 filtered/unfiltered
HOUR -24	109	pH, ORP		6.18	-210		1.2/1.5			CC3 filtered/unfiltered
HOUR -24	FIT	Total Flow	--				0.2/1.5			CC5 filtered/unfiltered
HOUR -24	PC3	ORP								
HOUR -24	PC5	pH		5.66	-89					

**Table B-5. Catalyzed cementation process demonstration field data record**

WEEK 1 (CONTINUOUS)										
DAY 5										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR- 8	108	pH, ORP		--	--		0.6	7:00	RZ	CC5 Sample Port
HOUR- 8	109	pH, ORP		--	--					
HOUR- 8	FIT	Total Flow	--							
HOUR -16	108	pH, ORP		3.93	-475			15:00	MGL	
HOUR -16	109	pH, ORP		6.08						
HOUR -16	FIT	Total Flow	--							
HOUR -24	108	pH, ORP		4.31	-336		13	23:00	RZ	CC2 Sample Port
HOUR -24	109	pH, ORP		6.06	-280		4.9			CC3 Sample Port
HOUR -24	FIT	Total Flow					2.6			CC5 Sample Port
HOUR -24	PC3	ORP	--		-280					
HOUR -24	PC5	pH		5.88	-78					
WEEK 1 (CONTINUOUS)										
DAY 6										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR - 6	108	pH, ORP		5.16	-543		18.2	5:00	RZ	CC2 Sample Port
HOUR - 6	109	pH, ORP		5.98	-280		3.2			CC3 Sample Port
HOUR - 6	FIT	Total Flow	--				1.3			CC5 Sample Port
HOUR -24	PC3	ORP			-217			23:00	JB	
HOUR -24	PC5	pH		5.5				23:00	RZ	
WEEK 1 (CONTINUOUS)										
DAY 7										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR -6	108	pH, ORP		5.29	-526		23.7	5:00	RZ	CC2 Sample Port
HOUR -6	109	pH, ORP		6.18	-254		3.8			CC3 Sample Port
HOUR -6	FIT	Total Flow	--				2.9			CC5 Sample Port
HOUR -24	PC3	ORP			OR		2.8	23:00	JB	CC3 Sample Port
HOUR -24	PC5	pH		5.82						
WEEK 2 (CONTINUOUS)										
DAY 1										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR -6	108	pH, ORP		3.68	-486		24.1	5:00	RZ	CC2 Sample Port
HOUR -6	109	pH, ORP		6.04	-505		2.5			CC3 Sample Port
HOUR -6	FIT	Total Flow	--							
HOUR -24	PC3	ORP			-264					
HOUR -24	PC5	pH		6.03						
WEEK 2 (CONTINUOUS)										
DAY 2										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR - 6	108	pH, ORP		5.15	-331		27.7	5:00	RZ	CC2 Sample Port
HOUR - 6	109	pH, ORP		6.3	-345		8.4			CC3 Sample Port
HOUR - 6	FIT	Total Flow	--							
HOUR -24	PC3	ORP					24.5	23:00	RZ	CC2 Sample Port
HOUR -24	PC5	pH		5.65						
WEEK 2 (CONTINUOUS)										
DAY 3										

**Table B-5. Catalyzed cementation process demonstration field data record**

Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR -6	108	pH, ORP		5.13	-530		23.1	5:00	RZ	
HOUR -6	109	pH, ORP		6.01	-284		13.7			
HOUR -6	FIT	Total Flow	0.264							
HOUR -24	PC3	ORP			-367	360	7.6			CC5 Sample Port
HOUR -24	PC5	pH		5.62						
<b>WEEK 2 (CONTINUOUS)</b>										
<b>DAY 4</b>										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR - 6	108	pH, ORP		3.77	-254		20.2	5:00	RZ	CC2 Sample Port
HOUR - 6	109	pH, ORP		6.04	235	650	8.1			CC3 Sample Port
HOUR - 6	FIT	Total Flow	--			520	8.2			CC4 Sample Port
HOUR -24	PC3	ORP			89		26.5	23:00	RZ	CC2 Sample Port
HOUR -24	PC5	pH		5.55		500	7.8			CC5 Sample Port
<b>WEEK 2 (CONTINUOUS)</b>										
<b>DAY 5</b>										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR -6	108	pH, ORP		4.54	-329		23.2	5:00	RZ	CC2 Sample Port
HOUR -6	109	pH, ORP			range -30 to +48		6.3			CC3 Sample Port
HOUR -6	FIT	Total Flow	--							
HOUR -24	PC3	ORP			-415		4.7			CC5 Sample Port
HOUR -24	PC5	pH		5.61						
<b>WEEK 2 (CONTINUOUS)</b>										
<b>DAY 6</b>										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR - 6	108	pH, ORP		4.74	-515		17.6	5:00	RZ	CC2 Sample Port
HOUR - 6	109	pH, ORP		6.07	120 to 280		7.3			CC3 Sample Port
HOUR - 6	FIT	Total Flow	--				1.5			CC5 Sample Port
HOUR -24	PC3	ORP			-350		24.2	23:00	RZ	CC2 Sample Port
HOUR -24	PC5	pH		5.67		70	1.5			CC5 Sample Port
<b>WEEK 2 (CONTINUOUS)</b>										
<b>DAY 7</b>										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR -6	108	pH, ORP		3.31	-221		24.1	5:00	RZ	CC2 Sample Port
HOUR -6	109	pH, ORP		5.96	78	510	8.7			CC3 Sample Port
HOUR -6	FIT	Total Flow	--			30	1.2			CC5 Sample Port
HOUR -24	PC3	ORP			220					
HOUR -24	PC5	pH		6.25						

**Table B-5. Catalyzed cementation process demonstration field data record**

WEEK 3 (CONTINUOUS)										
DAY 1										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR -6	108	pH, ORP		3.15	-440		38.6	5:00	RZ	CC2 Sample Port
HOUR -6	109	pH, ORP		--	--		21.8			CC3 Sample Port
HOUR -6	FIT	Total Flow	--				1.8			CC5 Sample Port
HOUR -24	PC3	ORP			-387		--	23:00	RZ	
HOUR -24	PC5	pH		--			--			
WEEK 3 (CONTINUOUS)										
DAY 2										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR - 6	108	pH, ORP		3.37	-330		20.7	5:00	RZ	CC2 Sample Port
HOUR - 6	109	pH, ORP		6.27	--		2.1			CC3 Sample Port
HOUR - 6	FIT	Total Flow	--							
HOUR -24	PC3	ORP			-360					
HOUR -24	PC5	pH		--						
WEEK 3 (CONTINUOUS)										
DAY 3										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR -6	108	pH, ORP		3.26	-344		16.6			CC2 Sample Port
HOUR -6	109	pH, ORP		6.49	--		43.4			CC3 Sample Port
HOUR -6	FIT	Total Flow	--				9.2			CC5 Sample Port
HOUR -24	PC3	ORP			-293		17.7	23:00	RZ	CC2 Sample Port
HOUR -24	PC5	pH		5.46		570	17.3			CC5 Sample Port
WEEK 3 (CONTINUOUS)										
DAY 4										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR - 6	108	pH, ORP		2.85	-326		22.5	6:15	RZ	CC2 Sample Port
HOUR - 6	109	pH, ORP		6.38	130		28			CC3 Sample Port
HOUR - 6	FIT	Total Flow	--				21.6			CC5 Sample Port
HOUR -24	PC3	ORP			121		27.9			CC5 Sample Port
HOUR -24	PC5	pH		4.52						
WEEK 3 (CONTINUOUS)										
DAY 5										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analy	Sampled Time	Initials	Comments
HOUR -6	108	pH, ORP		3.1	-349		21.2	5:00	RZ	CC2 Sample Port
HOUR -6	109	pH, ORP		6.2	118		27.2			CC3 Sample Port
HOUR -6	FIT	Total Flow	--				23.2			CC5 Sample Port
HOUR -24	PC3	ORP				Not Collected				
HOUR -24	PC5	pH				Not Collected				

**Table B-5. Catalyzed cementation process demonstration field data record**

WEEK 3 (CONTINUOUS)										
DAY 6										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analys	Sampled Time	Initials	Comments
HOUR - 6	108	pH, ORP				Not Collected				
HOUR - 6	109	pH, ORP				Not Collected				
HOUR - 6	FIT	Total Flow	--			Not Collected				
HOUR -24	PC3	ORP				Not Collected				
HOUR -24	PC5	pH				Not Collected				
WEEK 3 (CONTINUOUS)										
DAY 7 FINAL										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analys	Sampled Time	Initials	Comments
HOUR -6	108	pH, ORP				Not Collected				
HOUR -6	109	pH, ORP				Not Collected				
HOUR -6	FIT	Total Flow	--			Not Collected				
HOUR -24	108	pH, ORP		3.15	-288			7:30	RZ	
HOUR -24	109	pH, ORP		6.2	--					
HOUR -24	FIT	Total Flow	--							
HOUR -24	PC3	ORP			121					
WEEK 3 (CONTINUOUS)										
DAY 7 FINAL										
Sample Time	Sample Port	Sample Analysis	Totalizer Flow	pH Value	ORP Value	Iron Field Analy	Copper Field Analys	Sampled Time	Initials	Comments
HOUR -24	PC5	pH	--					7:30	RZ	

**Table B-6. Selenium demonstration project—summary data for catalyzed cementation process**

Lab #	Sample Description	Collection Date	Collection Time	Submission Date	Analyte CRDL Units	TDS 20 mg/L	TSS 3 mg/L	Iron 0.3 mg/L	Ferrous 0.5 mg/L	Ferric 0.5 mg/L	Calcium 1 mg/L	Magnesium 1 mg/L	Sodium 1 mg/L	Nitrate 0.2 mg/L	Sulfate 5 mg/L	Arsenic 10 ug/L	Barium 10 ug/L	Copper 10 ug/L	Iron 300 ug/L	Molybdenum 10 ug/L	Selenium 10 ug/L	Selenium Hydride 2 ug/L	Selenate 2 ug/L	Selenite 2 ug/L	
AH26728	MSE\CC3-001	10/26/99	20:00	10/27/99												93			6340		3700				
AH26729	MSE\CC1-101	10/27/99	0:00	10/27/99				< 0.3			120	50	350	4.1	256	13	54	13		124	1550				
AH26730	MSE\CC1-101	10/27/99	0:00	10/27/99				< 0.3	< 0.5	< 0.5	120	50	350			12	45	< 10		110	1530	1510	917	147	
AH26731	MSE\CC2-102	10/27/99	0:00	10/27/99							119	48	342			< 10	32	6080	6700	59	870				
AH26732	MSE\CC2-102	10/27/99	0:00	10/27/99							117	47	342			< 10	30	1250	6700	54	990				
AH26733	MSE\CC3-103	10/27/99	0:00	10/27/99							120	48	360			< 10	58	1700	52400	79	1470				
AH26734	MSE\CC3-103	10/27/99	0:00	10/27/99							119	48	360			< 10	43	298	10200	< 10	672				
AH26735	MSE\CC4-106	10/27/99	5:35	10/27/99																	482				
AH26736	MSE\CC4-107	10/27/99	8:00	10/27/99																	536				
AH26850	MSE\CC4-108	10/27/99	12:00	10/28/99																	680				
AH26851	MSE\CC4-109	10/27/99	16:00	10/28/99																	828				
AH26852	MSE\CC5-110	10/27/99	20:00	10/28/99																	193				
AH26853	MSE\CC3-111	10/28/99	0:00	10/28/99												< 10			247000		785				
AH26854	MSE\CC5-112	10/28/99	0:00	10/28/99												< 10			13250		493				
AH26855	MSE\CC5-113	10/28/99	4:00	10/28/99																	490				
AH26856	MSE\CC5-114	10/28/99	6:00	10/28/99																		755	446	34	
AH26857	MSE\CC5-115	10/28/99	8:00	10/28/99																	624				
AH26964	MSE\CC5-125	10/29/99	6:00	10/29/99		2020	52																		
AH26965	MSE\CC5-125	10/29/99	6:00	10/29/99																		1090	452	81	
AH26966	MSE\CC5-126	10/29/99	8:00	10/29/99																		1060			
AH26967	MSE\CC5-116	10/28/99	12:00	10/29/99																		948			
AH26968	MSE\CC5-117	10/28/99	16:00	10/29/99																		1030			
AH26969	MSE\CC5-118	10/28/99	20:00	10/29/99																		977			
AH26970	MSE\CC1-119	10/29/99	0:00	10/29/99							119	46.8	336	4.2	255	11	47	15	830	111	1340				
AH26971	MSE\CC1-119	10/29/99	0:00	10/29/99							116	46.8	335			15	56	10	208	123	1690				
AH26972	MSE\CC2-120	10/29/99	0:00	10/29/99							118	46.4	331			< 10	56	4020	54100	85	1240				
AH26973	MSE\CC2-120	10/29/99	0:00	10/29/99							111	45.4	327			< 10	49	733	16200	48	1400				
AH26974	MSE\CC3-121	10/29/99	0:00	10/29/99							114	47	340			< 10	58	365	184000	< 10	996				
AH26975	MSE\CC3-121	10/29/99	0:00	10/29/99							114	47	340			< 10	59	106	176000	< 10	1190				
AH26976	MSE\CC4-122	10/29/99	0:00	10/29/99							203	52.2	365			< 10	55	592	172000	< 10	1150				
AH26977	MSE\CC4-122	10/29/99	0:00	10/29/99							195	48.4	343			< 10	51	38	113000	< 10	1060				
AH26978	MSE\CC5-123	10/29/99	0:00	10/29/99							197	52.1	356	2.5	992	< 10	47	25	125000	< 10	1010				
AH26979	MSE\CC5-123	10/29/99	0:00	10/29/99							184	50.1	338			< 10	95	14	129000	< 10	890				
AH26980	MSE\CC4-124	10/29/99	6:00	10/29/99		1940	147																		
AH27064	MSE\CC5-127	10/29/99	16:00	11/1/99																		1040			
AH27065	MSE\CC3-128	10/30/99	0:00	11/1/99												< 10			213000		1120				
AH27066	MSE\CC5-129	10/30/99	0:00	11/1/99												< 10			134000		1050				
AH27067	MSE\CC8-129	10/30/99	0:00	11/1/99												< 10			131000		1020				
AH27068	MSE\CC9-129	10/30/99	0:00	11/1/99												< 10			< 300		< 10				
AH27069	MSE\CC5-002	10/30/99	6:00	11/1/99				112	96	16												1240	586	32	
AH27070	MSE\CC5-130	10/30/99	8:00	11/1/99																		1030			
AH27071	MSE\CC5-131	10/30/99	16:00	11/1/99																		1060			
AH27072	MSE\CC1-132	10/31/99	0:00	11/1/99							119	49.4	355	4.2	258	14	139	38	534	137	1540				
AH27073	MSE\CC1-132	10/31/99	0:00	11/1/99							119	49.1	355			< 10	50	10	< 300	116	1530				
AH27074	MSE\CC2-133	10/31/99	0:00	11/1/99							119	48.2	355			< 10	52	10000	78700	73	1200				
AH27075	MSE\CC2-133	10/31/99	0:00	11/1/99							119	47.8	351			< 10	49	3290	23700	17	1200				
AH27076	MSE\CC3-134	10/31/99	0:00	11/1/99							118	47.6	350			< 10	60	982	244000	< 10	832				
AH27077	MSE\CC3-134	10/31/99	0:00	11/1/99							118	47.6	350			< 10	58	157	232000	< 10	830				
AH27078	MSE\CC4-135	10/31/99	0:00	11/1/99							189	49.4	359			< 10	48	94	93300	< 10	1000				
AH27079	MSE\CC4-135	10/31/99	0:00	11/1/99							189	49.4	359			< 10	47	43	77200	< 10	1000				
AH27080	MSE\CC5-136	10/31/99	0:00	11/1/99							190	49	354	3.5	826	< 10	45	53	81900	< 10	1000				





**Table B-6. Selenium demonstration project—summary data for catalyzed cementation process**

AH28347 MSE\CC9-219	11/14/99	7:30	11/15/99													15	< 2	12	
AH28348 MSE\CC5-219	11/14/99	7:30	11/15/99	6970	54			596	47.8	965	0.6	3190	< 10	78	715	1100000	< 10	105	
AH28349 MSE\CC5-219	11/14/99	7:30	11/15/99					574	47.8	915			< 10	72	466	1090000	< 10	105	81
AH28350 MSE\CC8-219	11/14/99	7:30	11/15/99					703	47.8	1220			< 10	< 10	< 10	775000	< 10	29	
AH28351 MSE\CC9-219	11/14/99	7:30	11/15/99					< 1	< 1	< 1			< 10	142	420	< 300	172	< 10	

**Table B-7. BSeR™ Series 1, 12-hr retention time, total selenium**

Biological Selenium Removal, Series 1			Total Selenium, ug/L						
RT	Day	Date	Influent	Reactor 1 (Carbon)	Reactor 2 (Carbon)	Reactor 3 (Biosolids)	Reactor 4 (Biosolids)	Reactor 5 (Biosolids)	Final Effluent
Startup	1	9/27/99	1470.00	624.00	620.00	69.50	139.00	5.00	6.00
Startup	2	9/28/99	1600.00	749.00	230.00	7.00	8.00	5.00	6.00
Startup	3	9/29/99	1470.00	817.00	336.00	9.00	8.00	5.00	7.00
Startup	4	9/30/99	1680.00	276.00	323.00	6.00	3.00	0.00	0.00
Startup	5	10/1/99	860.00	227.00	260.00	0.00	0.00	0.00	0.00
Startup	6	10/2/99	1580.00	711.00	339.00	0.00	0.00	0.00	0.00
Startup	7	10/3/99	1440.00	792.00	300.00	8.00	5.00	0.00	3.00
12 hr	8	10/4/99	1010.00	736.00	321.00	12.00	9.00	5.00	6.00
12 hr	9	10/5/99	1120.00	1100.00	270.00	13.00	11.00	6.00	8.00
12 hr	10	10/6/99	1520.00	693.00	220.00	36.00	37.00	25.00	26.00
12 hr	11	10/7/99	1470.00	910.00	236.00	29.00	30.00	22.00	20.00
12 hr	12	10/8/99	1410.00	524.00	148.00	0.00	0.00	0.00	0.00
12 hr	13	10/9/99	1540.00	581.00	321.00	0.00	0.00	0.00	0.00
12 hr	14	10/10/99	1580.00	261.00	66.00	0.00	0.00	0.00	0.00
12 hr	15	10/11/99	1440.00	276.00	68.00	24.00	13.00	9.00	8.00
12 hr	16	10/12/99	1480.00	22.00	160.00	16.00	11.00	6.00	6.00
12 hr	17	10/13/99	1520.00	15.00	18.00	16.00	12.00	2.00	
12 hr	18	10/14/99	1120.00	0.00	0.00	0.00	5.00	3.00	4.00
12 hr	19	10/15/99	1580.00	0.00	0.00	0.00	0.00	0.00	0.00
12 hr	20	10/16/99	1880.00	0.00	0.00	0.00	0.00	0.00	0.00
12 hr	21	10/17/99	1540.00	22.00	3.00	3.00	0.00	0.00	0.00
12 hr	22	10/18/99	1610.00	47.00	2.00	2.00	0.00		
12 hr	23	10/19/99	1650.00	73.00	2.00	2.00	2.00	0.00	0.00
12 hr	24	10/20/99	1920.00	81.00	18.00	28.00	22.00	21.00	35.00
12 hr	25	10/21/99	1780.00	99.00	19.00	22.00	22.00	19.00	14.00
12 hr	26	10/22/99							
12 hr	27	10/25/99	1950.00		47.00		44.00	45.00	
12 hr	28	10/26/99	1570.00	17.00	0.00	19.00	18.00	0.00	
12 hr	29	10/28/99	1680.00	16.00	12.00	15.00	15.00	15.00	16.00

**Table B-8. BSeR™ Series 1, 12-hr retention time, dissolved oxygen**

Biological Selenium Removal, Series 1			Dissolved Oxygen, Percent Saturation						
RT	Day	Date	Influent	Reactor 1 (Carbon)	Reactor 2 (Carbon)	Reactor 3 (Biosolids)	Reactor 4 (Biosolids)	Reactor 5 (Biosolids)	Final Effluent
Startup	1	9/27/99	77.00	60.00	49.00	48.00	45.00	54.00	65.00
Startup	2	9/28/99	61.00	53.00	51.00	39.00	35.00	48.00	63.00
Startup	3	9/29/99	57.00	45.00	82.00	38.00	49.00	52.00	60.00
Startup	4	9/30/99	77.00	67.00	55.00	46.00	52.00	63.00	80.00
Startup	5	10/1/99	70.00	62.00	64.00	61.00	55.00	62.00	61.00
Startup	6	10/2/99	73.00	64.00	60.00	58.00	60.00	61.00	63.00
Startup	7	10/3/99	76.00	73.00	49.00	45.00	43.00	43.00	59.00
12 hr	8	10/4/99	63.00	61.00	48.00	40.00	44.00	49.00	84.00
12 hr	9	10/5/99	70.00	65.00	61.00	55.00	55.00	54.00	74.00
12 hr	10	10/6/99	62.00	49.00	49.00	37.00	36.00	43.00	55.00
12 hr	11	10/7/99	69.00	76.00	51.00	48.00	45.00	47.00	57.00
12 hr	12	10/8/99	72.00	66.00	57.00	57.00	52.00	61.00	63.00
12 hr	13	10/9/99	70.00	59.00	51.00	48.00	48.00	52.00	58.00
12 hr	14	10/10/99	72.00	68.00	62.00	51.00	43.00	48.00	69.00

**Table B-9. BSeR™ Series 1, 12-hr retention time, oxidation-reduction potential**

Biological Selenium Removal, Series 1			Oxidation/Reduction Potential, mV						
RT	Day	Date	Influent	Reactor 1 (Carbon)	Reactor 2 (Carbon)	Reactor 3 (Biosolids)	Reactor 4 (Biosolids)	Reactor 5 (Biosolids)	Final Effluent
Startup	1	9/27/99	248.00	215.00	210.00	23.30	(3.00)	205.00	226.00
Startup	2	9/28/99	172.00	129.00	135.00	(3.00)	(40.00)	125.00	152.00
Startup	3	9/29/99	281.00	209.00	167.00	44.70	(27.00)	122.00	144.00
Startup	4	9/30/99	193.00	134.00	138.70	17.30	(33.00)	126.30	167.70
Startup	5	10/1/99	155.30	147.50	149.50	49.00	(29.50)	74.50	120.00
Startup	6	10/2/99	147.10	140.00	137.20	48.00	(31.00)	84.10	136.10
Startup	7	10/3/99	100.00	151.70	159.20	26.00	(13.30)	64.90	128.30
12 hr	8	10/4/99	136.00	112.00	132.00	14.50	11.30	110.00	165.30
12 hr	9	10/5/99	146.50	159.30	163.50	83.00	56.50	125.30	154.30
12 hr	10	10/6/99	97.00	125.30	140.00	23.00	(3.00)	98.00	135.00
12 hr	11	10/7/99	248.00	152.00	154.00	37.30	(3.00)	128.50	174.00
12 hr	12	10/8/99	178.00	116.00	81.30	(2.70)	(15.00)	153.00	194.00
12 hr	13	10/9/99	94.30	145.00	176.00	27.00	(32.50)	140.70	149.70
12 hr	14	10/10/99	142.30	116.00	93.20	46.80	(36.30)	116.00	144.20
12 hr	15	10/11/99	172.30	153.00	198.70	205.00	182.00	201.00	198.00
12 hr	16	10/12/99	146.70	125.30	150.00	(17.50)	(47.30)	105.30	196.30
12 hr	17	10/13/99	95.70	166.50	91.50	(42.70)	(29.50)	100.90	130.30
12 hr	18	10/14/99	98.50	65.00	89.30	(21.70)	(10.30)	93.00	164.00
12 hr	19	10/15/99	120.00	93.30	107.50	(30.70)	(22.50)	123.50	157.00
12 hr	20	10/16/99	121.30	100.70	85.50	(5.70)	(52.30)	95.70	135.50
12 hr	21	10/17/99	131.70	115.70	116.50	(30.10)	(51.00)	90.20	118.00
12 hr	22	10/18/99	210.00	116.30	100.50	59.00	7.50		
12 hr	23	10/19/99	248.00	116.30	131.30	(3.30)	(19.70)	126.00	152.70
12 hr	24	10/20/99	208.00	115.60	104.50	(10.50)	(16.70)	60.30	99.30
12 hr	25	10/21/99	226.00	126.50	96.30	(15.70)	(33.00)	68.00	108.00
12 hr	26	10/22/99	136.00	102.50	112.30	24.70	11.30	105.50	139.00
12 hr	27	10/25/99	323.00		(0.30)		81.30	82.50	
12 hr	28	10/26/99	314.00	67.00	90.30	(14.00)	69.70	143.00	
12 hr	29	10/28/99	325.00	113.00	72.30	42.00	9.70	27.50	

**Table B-10. BSeR™ Series 1, 12-hr retention time, temperature**

Biological Selenium Removal, Series 1			Temperature, °C						
RT	Day	Date	Influent	Reactor 1 (Carbon)	Reactor 2 (Carbon)	Reactor 3 (Biosolids)	Reactor 4 (Biosolids)	Reactor 5 (Biosolids)	Final Effluent
Startup	1	9/27/99	17.00	14.40	16.60	15.10	16.80	16.80	15.20
Startup	2	9/28/99	16.50	13.10	14.20	12.20	14.10	13.10	13.10
Startup	3	9/29/99	16.20	13.30	14.10	13.80	14.10	14.10	13.70
Startup	4	9/30/99	16.20	16.30	17.30	15.50	16.70	15.40	14.10
Startup	5	10/1/99	16.20	14.20	17.10	14.50	16.40	14.30	13.80
Startup	6	10/2/99	18.10	18.00	18.00	18.40	18.30	19.00	16.40
Startup	7	10/3/99	18.20	19.00	18.00	18.00	18.50	19.50	18.70
12 hr	8	10/4/99	17.10	17.10	17.60	15.00	16.00	15.00	16.40
12 hr	9	10/5/99	16.60	16.50	19.00	17.00	18.60	18.00	15.60
12 hr	10	10/6/99	17.10	19.40	21.00	20.50	20.30	20.20	16.70
12 hr	11	10/7/99	16.40	14.10	16.90	16.30	17.20	16.60	14.90
12 hr	12	10/8/99	16.20	14.30	16.90	15.10	16.70	15.80	14.40
12 hr	13	10/9/99	17.20	18.00	18.40	18.10	17.80	19.40	16.30
12 hr	14	10/10/99	17.60	20.10	20.10	21.10	19.60	21.40	17.90
12 hr	15	10/11/99	15.50	14.20	18.40	15.90	18.20	16.30	14.30
12 hr	16	10/12/99	16.50	18.30	19.50	20.20	20.10	21.40	16.00
12 hr	17	10/13/99	16.00	16.00	18.20	18.40	19.60	19.90	15.10
12 hr	18	10/14/99	17.10	16.50	18.00	18.00	19.20	19.00	15.30
12 hr	19	10/15/99	16.20	15.60	16.90	17.10	19.20	17.50	14.90
12 hr	20	10/16/99	15.70	14.10	13.80	13.60	13.80	14.30	12.70
12 hr	21	10/17/99	15.50	14.20	13.90	13.90	13.70	14.00	12.90
12 hr	22	10/18/99	15.20	14.00	13.80	13.80	13.60		
12 hr	23	10/19/99	15.70	12.40	13.20	13.10	12.70	11.90	11.30
12 hr	24	10/20/99	15.80	12.60	13.20	13.20	12.90	12.00	11.50
12 hr	25	10/21/99	16.50	14.00	14.50	13.00	14.50	12.00	12.00
12 hr	26	10/22/99	16.30	13.00	14.10	13.00	14.50	13.20	14.80
12 hr	27	10/25/99	16.10		14.60		15.30	15.00	
12 hr	28	10/26/99	17.10	19.20	19.40	21.60	19.70	20.90	
12 hr	29	10/28/99	16.80	19.10	16.80	19.80	16.70	19.10	

**Table B-11. BSeR™ Series 1, 12-hr retention time, pH**

Biological Selenium Removal, Series 1				pH					
RT	Day	Date	Influent	Reactor 1 (Carbon)	Reactor 2 (Carbon)	Reactor 3 (Biosolids)	Reactor 4 (Biosolids)	Reactor 5 (Biosolids)	Final Effluent
Startup	1	9/27/99	7.22	7.55	7.53	7.54	7.25	7.00	7.10
Startup	2	9/28/99	7.20	7.60	7.50	7.52	7.10	7.12	7.06
Startup	3	9/29/99	7.40	7.86	7.74	7.43	7.34	7.03	7.31
Startup	4	9/30/99	7.12	8.01	7.88	7.66	7.56	7.15	7.66
Startup	5	10/1/99	7.02	7.75	7.57	7.53	7.40	7.09	7.49
Startup	6	10/2/99	7.66	7.85	7.80	7.53	7.44	7.18	7.48
Startup	7	10/3/99	7.33	7.90	7.82	7.87	7.34	7.09	7.25
12 hr	8	10/4/99	7.49	8.02	7.82	7.65	7.50	7.17	7.40
12 hr	9	10/5/99	7.27	7.86	7.75	7.55	7.42	7.12	7.39
12 hr	10	10/6/99	7.22	8.12	7.98	7.77	7.64	7.37	7.52
12 hr	11	10/7/99	7.36	7.70	7.69	7.43	7.33	7.12	7.39
12 hr	12	10/8/99	7.40	7.80	7.86	7.63	7.55	7.26	7.57
12 hr	13	10/9/99	7.48	8.08	7.90	7.68	7.63	7.31	7.60
12 hr	14	10/10/99	7.20	8.10	7.95	7.69	7.54	7.20	7.26
12 hr	15	10/11/99	7.38	7.98	7.76	7.49	7.37	7.03	7.35
12 hr	16	10/12/99	7.64	8.06	7.93	7.66	7.61	7.29	7.55
12 hr	17	10/13/99	7.31	7.78	7.86	7.60	7.55	7.29	7.47
12 hr	18	10/14/99	7.68	7.35	7.80	7.64	7.56	7.30	7.44
12 hr	19	10/15/99	7.55	7.24	7.85	7.68	7.59	7.40	7.82
12 hr	20	10/16/99	7.40	7.31	7.64	7.48	7.40	7.14	7.52
12 hr	21	10/17/99	7.42	7.35	7.60	7.51	7.42	7.23	7.52
12 hr	22	10/18/99	7.56	6.85	7.62	7.52	7.48		
12 hr	23	10/19/99	7.32	6.94	7.51	7.56	7.59	7.45	7.72
12 hr	24	10/20/99	7.53	7.10	7.31	7.52	7.50	7.45	7.60
12 hr	25	10/21/99	7.56	7.08	7.20	7.60	7.56	7.58	7.72
12 hr	26	10/22/99	7.23	7.04	7.00	7.26	7.35	7.38	7.47
12 hr	27	10/25/99	7.43		7.09		7.32	7.98	
12 hr	28	10/26/99	7.37	7.17	6.99	7.30	7.34	8.06	
12 hr	29	10/28/99	7.45	7.10	7.03	7.25	7.30	7.96	

**Table B-12. BSeR™ Series 2, 11- and 5.5-hr retention time, total selenium**

Biological Selenium Removal, Series 2			Total Selenium, Fg/L			
RT	Day	Date	Influent	Reactor 1	Reactor 2	Reactor 3
Startup	1	1/10/99	1700		139	241
Startup	2	1/11/00	1520	1350	881	187
Startup	4	1/13/00	1500	1150	551	113
Startup	5	1/14/00	1750	1110	324	65
Startup	6	1/15/00	1650	1260	213	34
Startup	7	1/16/00	1600	690	199	16
Startup	8	1/17/00	1570	1140	235	9
11 hr	9	1/18/00	1880	940	317	8
11 hr	10	1/19/00	1670	328	347	19
11 hr	11	1/20/00	1540	184	154	8
11 hr	12	1/21/00	1810	139	7	5
11 hr	13	1/22/00	1670	92	9	11
11 hr	14	1/23/00	1800	77	10	0
11 hr	15	1/25/00	1640	42	9	4
11 hr	16	1/26/00	1590	15	8	0
11 hr	17	1/27/00	1740	44	13	7
11 hr	18	1/28/00	2230	9	5	2
11 hr	19	1/29/00	1830	12	8	3
11 hr	20	1/30/00	1860	12	8	2
11 hr	22	2/1/00	1400	5	2	0
11 hr	23	2/3/00	1650	11	3	0
11 hr	24	2/4/00	1210	36	3	3
11 hr	25	2/5/00	1590	16	2	2
5.5 hr	1	2/6/00	1626	40	3	2
5.5 hr	2	2/7/00	1510	24	3	2
5.5 hr	3	2/8/00	1480	26	0	0
5.5 hr	4	2/9/00	1451	22	2	10
5.5 hr	5	2/10/00	1585	30	3	0
5.5 hr	6	2/11/00	1590	15	0	0
5.5 hr	7	2/12/00	1540	15	0	0
5.5 hr	8	2/13/00	1530	9	0	0
5.5 hr	9	2/14/00	1560	21	0	0
5.5 hr	10	2/16/00	1580	8	0	0
5.5 hr	11	2/17/00	1780	10	0	0
5.5 hr	12	2/18/00	1400	14	0	0

**Table B-13. BSeR™ Series 2, 11- and 5.5-hr retention time, dissolved oxygen**

Biological Selenium Removal, Series 2			Dissolved Oxygen, Percent Saturation			
RT	Day	Date	Influent	Reactor 1	Reactor 2	Reactor 3
Startup	1	1/10/99	53	40.2	66.7	68
Startup	2	1/11/00	58.3	48.7	12.7	46.9
Startup	4	1/13/00	57.4	45.4	12.3	33
Startup	5	1/14/00	47.4	41.2	11.7	36
Startup	6	1/15/00	47.6	42	16.6	39.2
Startup	7	1/16/00	55.4	38	12.4	47.6
Startup	8	1/17/00	55.7	46	12.2	44.7
11 hr	9	1/18/00	51.6	34.1	12.3	42.2
11 hr	10	1/19/00	51.6	34.1	12.3	42.2
11 hr	11	1/20/00	49.3	40.1	19.1	39.2
11 hr	12	1/21/00	52.1	35.6	11.6	40.5
11 hr	13	1/22/00	51.2	39.8	17	38.7
11 hr	14	1/23/00	54.6	42.7	20.2	39.7
11 hr	15	1/25/00	44.1	39.9	14.5	43.1
11 hr	16	1/26/00	49.8	36.1	13.2	35.1
11 hr	17	1/27/00	53.8	39.8	17.2	37.9
11 hr	18	1/28/00	44	24.8	27.6	39.2
11 hr	19	1/29/00	52.4	37.7	17.1	40
11 hr	20	1/30/00	55.9	44.6	18.4	41.5
11 hr	22	2/1/00	52.6	24.2	17.3	43.7
11 hr	23	2/3/00	52.9	42.5	21	46.6
11 hr	24	2/4/00	47.6	39.5	20.1	41.2
11 hr	25	2/5/00	50.2	35.7	17.2	46.5
5.5 hr	1	2/6/00	56.6	37.7	20	39.9
5.5 hr	2	2/7/00	52	28.5	15.9	30
5.5 hr	3	2/8/00	47.4	33.2	14.8	31.4
5.5 hr	4	2/9/00	48.3	30.5	15.6	30.3
5.5 hr	5	2/10/00	48.2	24.5	10.8	24.6
5.5 hr	6	2/11/00	46.9	25.2	16	30.1
5.5 hr	7	2/12/00	47.7	23.1	11.6	26.7
5.5 hr	8	2/13/00	44.1	26.4	12	27.6
5.5 hr	9	2/14/00	44.5	30	15.7	29.5
5.5 hr	10	2/16/00	46.1	23.7	17	33.5
5.5 hr	11	2/17/00				
5.5 hr	12	2/18/00				

**Table B-14. BSeR™ Series 2, 11- and 5.5-hr retention time, oxidation-reduction potential**

Biological Selenium Removal, Series 2			Oxidation/Reduction Potential, mV			
RT	Day	Date	Influent	Reactor 1	Reactor 2	Reactor 3
Startup	1	1/10/99				
Startup	2	1/11/00	272	226	182	150
Startup	4	1/13/00	147	310	314	355
Startup	5	1/14/00	290	361	347	394
Startup	6	1/15/00	251	265	248	266
Startup	7	1/16/00	282	303	327	311
Startup	8	1/17/00	313	296	286	289
11 hr	9	1/18/00	336	312	313	283
11 hr	10	1/19/00	332	315	313	148
11 hr	11	1/20/00	333	224	234	108
11 hr	12	1/21/00	335	155	217	8037
11 hr	13	1/22/00	332	141	198	65
11 hr	14	1/23/00	328	136	187	62
11 hr	15	1/25/00	145.7	143.7	206	71
11 hr	16	1/26/00	304	114	207	52
11 hr	17	1/27/00	334	98	159	33
11 hr	18	1/28/00	342	91	144	37
11 hr	19	1/29/00	327	75	133	42
11 hr	20	1/30/00	330	67	117	24
11 hr	22	2/1/00	223	3	4.5	-14.3
11 hr	23	2/3/00	272	13.5	92.7	-49
11 hr	24	2/4/00	312	5	99.7	-41.5
11 hr	25	2/5/00	311	-2.5	110.3	-51.3
5.5 hr	1	2/6/00	318	19.5	132	-23
5.5 hr	2	2/7/00	315	-72	98.7	-43.7
5.5 hr	3	2/8/00	308	-105.7	62	-40.6
5.5 hr	4	2/9/00	306	-129	44.5	-11.3
5.5 hr	5	2/10/00	293	-130.5	44.5	2
5.5 hr	6	2/11/00	313	-102	75.7	12.7
5.5 hr	7	2/12/00	310	-124.7	40.5	18.3
5.5 hr	8	2/13/00	309	-83.5	65.5	37.3
5.5 hr	9	2/14/00	319	-60.7	73	45.5
5.5 hr	10	2/16/00	308	-41.7	112.7	56
5.5 hr	11	2/17/00				
5.5 hr	12	2/18/00				

**Table B-15. BSeR™ Series 2, 11- and 5.5-hr retention time, temperature**

Biological Selenium Removal, Series 2			Temperature, °C			
RT	Day	Date	Influent	Reactor 1	Reactor 2	Reactor 3
Startup	1	1/10/99				
Startup	2	1/11/00	14.5	14.7	13	12.2
Startup	4	1/13/00	14.7	14.2	14	11.3
Startup	5	1/14/00	14.8	14	13.8	10.9
Startup	6	1/15/00	14	13.5	13.1	11.3
Startup	7	1/16/00	15.3	14.7	14.5	13
Startup	8	1/17/00	14.8	14	14	12.9
11 hr	9	1/18/00	15	14.2	13.2	12.7
11 hr	10	1/19/00	14.9	14.1	14.4	12.2
11 hr	11	1/20/00	14.8	14.3	13.5	12.6
11 hr	12	1/21/00	15	15.1	14.6	12.7
11 hr	13	1/22/00	15.3	15.1	14.1	12.6
11 hr	14	1/23/00	15.5	16.1	14.3	12.7
11 hr	15	1/25/00	15	14.7	14.2	12.5
11 hr	16	1/26/00	14.9	16	14.5	12.9
11 hr	17	1/27/00	16.1	15.8	14.8	14
11 hr	18	1/28/00	14.5	14.2	13.1	11.3
11 hr	19	1/29/00	16.3	16.3	16.8	14.7
11 hr	20	1/30/00	16.6	17.4	18.6	16.4
11 hr	22	2/1/00	14.7	19.2	20	15.9
11 hr	23	2/3/00	15.2	17.7	20.3	16.8
11 hr	24	2/4/00	14.7	14.3	14.6	15.8
11 hr	25	2/5/00	15.6	17.8	19.2	16.3
5.5 hr	1	2/6/00	16.3	16.8	16.6	18.2
5.5 hr	2	2/7/00	16.1	18.5	19.7	17.4
5.5 hr	3	2/8/00	14.4	14.7	13.9	14.9
5.5 hr	4	2/9/00	15.1	15.3	14.6	14
5.5 hr	5	2/10/00	14.2	14.3	14	14
5.5 hr	6	2/11/00	15.3	15	14.3	13.9
5.5 hr	7	2/12/00	15.4	14.9	14.1	14.1
5.5 hr	8	2/13/00	14.6	15.5	14.1	14.6
5.5 hr	9	2/14/00	14.8	15.4	14.2	13.3
5.5 hr	10	2/16/00	14.9	17.9	16.1	16
5.5 hr	11	2/17/00				
5.5 hr	12	2/18/00				

**Table B-16. BSeR™ Series 2, 11- and 5.5-hr retention time, pH**

Biological Selenium Removal, Series 2			pH			
RT	Day	Date	Influent	Reactor 1	Reactor 2	Reactor 3
Startup	1	1/10/99	7.7	8.48	7.67	7.38
Startup	2	1/11/00	7.46	7.6	8.27	7.94
Startup	4	1/13/00	7.29	7.41	8.07	7.22
Startup	5	1/14/00	7.39	7.43	7.89	7.19
Startup	6	1/15/00	7.39	7.48	7.37	7.24
Startup	7	1/16/00	7.37	7.51	7.61	7.2
Startup	8	1/17/00	7.42	7.62	7.58	7.22
11 hr	9	1/18/00	7.45	7.6	7.54	7.24
11 hr	10	1/19/00	7.42	7.57	7.45	7.3
11 hr	11	1/20/00	7.39	7.22	7.44	7.21
11 hr	12	1/21/00	7.45	6.9	7.35	7.31
11 hr	13	1/22/00	7.38	6.64	7.38	7.23
11 hr	14	1/23/00	7.37	6.55	7.42	7.31
11 hr	15	1/25/00	7.35	6.46	7.16	7.36
11 hr	16	1/26/00	7.37	6.42	7.11	7.45
11 hr	17	1/27/00	7.37	6.36	7.02	7.43
11 hr	18	1/28/00	7.4	6.31	7.05	7.51
11 hr	19	1/29/00	7.38	6.36	6.74	7.44
11 hr	20	1/30/00	7.4	6.45	6.65	7.46
11 hr	22	2/1/00	7.33	6.59	6.58	7.35
11 hr	23	2/3/00	7.44	6.65	6.65	7.21
11 hr	24	2/4/00	7.41	6.8	6.8	7.18
11 hr	25	2/5/00	7.4	6.84	6.7	7.1
5.5 hr	1	2/6/00	7.43	6.86	6.78	7.05
5.5 hr	2	2/7/00	7.42	6.72	6.66	6.95
5.5 hr	3	2/8/00	7.38	6.86	6.73	6.94
5.5 hr	4	2/9/00	7.4	6.97	6.58	6.86
5.5 hr	5	2/10/00	7.43	7.02	6.6	6.91
5.5 hr	6	2/11/00	7.48	7.09	6.75	6.8
5.5 hr	7	2/12/00	7.43	7.18	6.82	6.81
5.5 hr	8	2/13/00	7.42	7.41	7.09	6.99
5.5 hr	9	2/14/00	7.43	7.54	7.12	7.1
5.5 hr	10	2/16/00	7.45	7.48	7.15	7.15
5.5 hr	11	2/17/00				
5.5 hr	12	2/18/00				

**Table B-17. BSeR™ Series 3, 8-hr retention time, total selenium**

Biological Selenium Removal, Series 3			Total Selenium, ug/L			
RT	Day	Date	Influent	Reactor 1	Reactor 2	Reactor 3
Startup	1	1/16/00	1600	152	67	0
Startup	2	1/17/00	1570	294		0
Startup	3	1/18/00	1880	80	0	0
8 hr	4	1/19/00	1670	0	0	0
8 hr	5	1/20/00	1540	0	0	0
8 hr	6	1/21/00	1810	3	0	0
8 hr	7	1/22/00	1670	40	11	11
8 hr	8	1/23/00	1800	30	0	0
8 hr	9	1/25/00	1640	4	4	3
8 hr	10	1/26/00	1590	4	2	0
8 hr	11	1/27/00	1740	10	7	4
8 hr	12	1/28/00	2230			
8 hr	13	1/29/00	1830			
8 hr	14	1/30/00	1860	7	4	0
8 hr	16	2/1/00	1400	12	8	0
8 hr	18	2/3/00	1650	11	8	0
8 hr	19	2/4/00	1210	16	7	0
8 hr	20	2/5/00	1590	16	7	0
8 hr	21	2/6/00	1626	9	8	0
8 hr	22	2/7/00	1510	5	9	0
8 hr	23	2/8/00	1480	8	9	0
8 hr	24	2/9/00	1451	5	7	0
8 hr	25	2/10/00	1585	5	5	0
8 hr	26	2/11/00	1590	2	5	0
8 hr	27	2/12/00	1540	3	4	0
8 hr	28	2/13/00	1530	3	3	0
8 hr	29	2/14/00	1560	4	4	0
8 hr	31	2/16/00	1580	5	4	3
8 hr	32	2/17/00	1780	2	2	0
8 hr	33	2/18/00	1400	2	2	0

**Table B-18. BSeR™ Series 3, 8-hr retention time, dissolved oxygen**

Biological Selenium Removal, Series 3			Dissolved Oxygen			
RT	Day	Date	Influent	Reactor 1	Reactor 2	Reactor 3
Startup	1	1/16/00	55.4	12.2		26.2
Startup	2	1/17/00	55.7	17.9		39.7
Startup	3	1/18/00	51.6	12.6	17.6	30.6
8 hr	4	1/19/00	51.6	12.6	17.6	30.6
8 hr	5	1/20/00	49.3	10.4	18.4	29.8
8 hr	6	1/21/00	52.1	18.3	17.7	23.6
8 hr	7	1/22/00	51.2	14.7	22.1	27.3
8 hr	8	1/23/00	54.6	21.4	22.1	38.1
8 hr	9	1/25/00	44.1	18.2	16.9	36.8
8 hr	10	1/26/00	49.8	15.7	21.7	26.7
8 hr	11	1/27/00	53.8	17.8	16.5	35.5
8 hr	12	1/28/00				
8 hr	13	1/29/00				
8 hr	14	1/30/00	55.9	17.5	23	32
8 hr	16	2/1/00	52.6	18.6	26.1	28.2
8 hr	18	2/3/00	52.9	21.4	19	31.8
8 hr	19	2/4/00	47.6	18.7	16.9	32.7
8 hr	20	2/5/00	50.2	17.7	22.2	32.2
8 hr	21	2/6/00	56.6	20.1	20	47
8 hr	22	2/7/00	52	18.4	21.3	37
8 hr	23	2/8/00	47.4	20.6	18.2	15.9
8 hr	24	2/9/00	48.3	21.2	16.4	29.2
8 hr	25	2/10/00	48.2	18.7	17	29.3
8 hr	26	2/11/00	46.9	20	17.5	35.5
8 hr	27	2/12/00	47.7	18.5	13.4	31
8 hr	31	2/16/00	44.1	18.9	15.9	35.7
8 hr	32	2/17/00	44.5	14.1	13.6	39.3
8 hr	33	2/18/00	46.1	18.9	18	34.1

**Table B-19. BSeR™ Series 3, 8-hr retention time, oxidation-reduction potential**

Biological Selenium Removal, Series 3			Oxidation/Reduction Potential, mV			
RT	Day	Date	Influent	Reactor 1	Reactor 2	Reactor 3
Startup	1	1/16/00	282	28.3		220
Startup	2	1/17/00	313	287		272
Startup	3	1/18/00	336	1.5	119	188
8 hr	4	1/19/00	332	-29.7	60.7	150
8 hr	5	1/20/00	333	-20	8.3	115
8 hr	6	1/21/00	335	-15.3	30.7	18.5
8 hr	7	1/22/00	332	-1.5	33.5	-24.3
8 hr	8	1/23/00	328	-5	26	-16
8 hr	9	1/25/00	145.7	-25.5	6.7	17.3
8 hr	10	1/26/00	304	-39	-16	20
8 hr	11	1/27/00	334	-29	-27	23
8 hr	12	1/28/00	342			
8 hr	13	1/29/00	327			
8 hr	14	1/30/00	330	-41	-38	14
8 hr	16	2/1/00	223	-49.3	-96.3	36
8 hr	18	2/3/00	272	-51.7	-98.5	-39.5
8 hr	19	2/4/00	312	-51	-99.5	-26
8 hr	20	2/5/00	311	-59.5	-100.7	-46.3
8 hr	21	2/6/00	318	-55.3	-91.7	-39.5
8 hr	22	2/7/00	315	-84.5	-90.3	-42
8 hr	23	2/8/00	308	-74	-117.3	-50
8 hr	24	2/9/00	306	-78.3	-117.5	-67.3
8 hr	25	2/10/00	293	-76.5	-103	-58.7
8 hr	26	2/11/00	313	-50.5	-90.5	-51.5
8 hr	27	2/12/00	310	-82.3	-65.5	-45
8 hr	31	2/16/00	309	-105.7	-51	-43.3
8 hr	32	2/17/00	319	-93.7	-49.3	-47.3
8 hr	33	2/18/00	308	-74.5	-27	-45.7

**Table B-20. BSeR™ Series 3, 8-hr retention time, temperature**

Biological Selenium Removal, Series 3			Temperature, °C			
RT	Day	Date	Influent	Reactor 1	Reactor 2	Reactor 3
Startup	1	1/16/00	15.3	14.5		10.1
Startup	2	1/17/00	14.8	14.2		13.6
Startup	3	1/18/00	15	14.5	13.8	13
8 hr	4	1/19/00	14.9	13.8	13.6	12.7
8 hr	5	1/20/00	14.8	14.5	12.9	12.5
8 hr	6	1/21/00	15	14	13.9	13
8 hr	7	1/22/00	15.3	14.4	13.6	12.6
8 hr	8	1/23/00	15.5	14.1	13.9	12.6
8 hr	9	1/25/00	15	14.7	13.6	12.9
8 hr	10	1/26/00	14.9	14.8	14.8	13.3
8 hr	11	1/27/00	16.1	15.4	14.2	13.7
8 hr	12	1/28/00	14.5			
8 hr	13	1/29/00	16.3			
8 hr	14	1/30/00	16.6	16.4	16.7	16.6
8 hr	16	2/1/00	14.7	16	16.7	15.7
8 hr	18	2/3/00	15.2	16.4	17.1	17.3
8 hr	19	2/4/00	14.7	15.9	16	16.4
8 hr	20	2/5/00	15.6	15.7	15	14.6
8 hr	21	2/6/00	16.3	16.7	16.8	16.5
8 hr	22	2/7/00	16.1	16.3	15.9	15.3
8 hr	23	2/8/00	14.4	14.8	13.6	13.3
8 hr	24	2/9/00	15.1	14.8	14.4	13.7
8 hr	25	2/10/00	14.2	14.5	14.8	13.9
8 hr	26	2/11/00	15.3	15.1	15.9	15.6
8 hr	27	2/12/00	15.4	15.2	15.7	15.2
8 hr	31	2/16/00	14.6	15.4	14.6	15.6
8 hr	32	2/17/00	14.8	15	14.1	13.6
8 hr	33	2/18/00	14.9	15.9	15.2	14.6

**Table B-21. BSeR™ Series 3, 8-hr retention time, pH**

Biological Selenium Removal, Series 3			pH			
RT	Day	Date	Influent	Reactor 1	Reactor 2	Reactor 3
Startup	1	1/16/00	7.37	7.15		8.2
Startup	2	1/17/00	7.42	7.24		7.82
Startup	3	1/18/00	7.45	6.95	6.93	7.49
8 hr	4	1/19/00	7.42	6.78	6.94	7.49
8 hr	5	1/20/00	7.39	6.64	6.85	7.33
8 hr	6	1/21/00	7.45	6.67	6.63	7.15
8 hr	7	1/22/00	7.38	6.68	6.47	6.98
8 hr	8	1/23/00	7.37	6.8	6.57	6.89
8 hr	9	1/25/00	7.35	6.72	6.63	6.83
8 hr	10	1/26/00	7.37	6.64	6.55	6.8
8 hr	11	1/27/00	7.37	6.66	6.53	6.91
8 hr	12	1/28/00	7.4			
8 hr	13	1/29/00	7.38			
8 hr	14	1/30/00	7.4	6.72	6.57	6.84
8 hr	16	2/1/00	7.33	6.83	6.71	6.73
8 hr	18	2/3/00	7.44	7.01	6.83	6.74
8 hr	19	2/4/00	7.41	7.08	6.9	6.88
8 hr	20	2/5/00	7.4	7.15	6.94	6.97
8 hr	21	2/6/00	7.43	7.22	6.97	7.1
8 hr	22	2/7/00	7.42	7.1	7	7.04
8 hr	23	2/8/00	7.38	7.06	6.98	7.09
8 hr	24	2/9/00	7.4	6.98	7.05	7.05
8 hr	25	2/10/00	7.43	6.97	7.08	6.95
8 hr	26	2/11/00	7.48	6.96	7.08	6.86
8 hr	27	2/12/00	7.43	6.96	7.09	6.9
8 hr	31	2/16/00	7.42	7.05	7.11	7.14
8 hr	32	2/17/00	7.43	7.12	7.11	7.26
8 hr	33	2/18/00	7.45	7.15	7.13	7.31

**Table B-22. Catalyzed Cementation Process Demonstration Test Data Record Follow on Testing**

<b>BACKGROUND DAYS 3/28/00</b>								
<b>WEEK 1</b>								
Sample Time	Sample Number	Sample Port	Sample Analysis	pH Value	ORP Value	Sampled Time	Initials	Comments
HOUR -	CC 1 -050	CC1	pH, ORP	7.89	284.9	11:50	RS	
HOUR -		Metal Reactor	pH	3.02		10:40	RS	
HOUR -		Floc Tank	pH	6.78		10:45	RS	
HOUR -	CC1-051	CC1	pH, ORP	8.26	337	10:35	RS	
HOUR -	CC2-052	CC2	pH, ORP	3.48	416	10:30	RS	
HOUR -	CC 2-053	CC2	pH, ORP	3.52	372	15:30	RS	
<b>WEEK 1 (CONTINUOUS)</b>								
<b>DAY 1 INITIAL 3/30/00</b>								
Sample Time	Sample Number	Sample Port	Sample Analysis	pH Value	ORP Value	Sampled Time	Initials	Comments
HOUR - 0		Metal Reactor 2	pH	3.8		7:20	RS	
HOUR - 0		Floc Tank	pH	6.3		7:20	RS	
HOUR - 0		CC1	pH, ORP	8.3	267	7:35	RS	
HOUR - 0		CC2	pH, ORP	3.5	389	7:50	RS	
HOUR - 0		CC3	pH, ORP	2.93	277	8:25	RS	
HOUR - 0		CC4	pH, ORP	6.48	-108	8:50	RS	
HOUR - 0		CC5	pH, ORP	3.93	2.34	9:10	RS	
<b>Comments</b>								
Time Zero Begins when system has been filled, and residual tap water has been flushed out								
<b>WEEK 1 (CONTINUOUS)</b>								
<b>DAY 1 3/30/00</b>								
Sample Time	Sample Number	Sample Port	Sample Analysis	pH Value	ORP Value	Sampled Time	Initials	Comments
HOUR - 4		Metal Reactor 2	pH	2.7		11:45	RS	
HOUR - 4		Floc Tank	pH	7		11:45	RS	
HOUR - 6		CC1	pH, ORP	8.27	275	13:20	RS	
HOUR - 6		CC2	pH, ORP	3.5	390	13:35	RS	
HOUR - 6		CC3	pH, ORP	3.22	278	1350	RS	
HOUR - 6		CC4	pH, ORP	7.01	-218	14:20	RS	
HOUR - 6		CC5	pH, ORP	NR	269	14:45	RS	
HOUR - 8		Metal Reactor 2	pH	3.3		15:30	RS	
HOUR - 8		Floc Tank	pH	6.9		15:30	RS	
<b>Comment</b>								
<b>WEEK 1</b>								
<b>DAY 2 3/31/00</b>								
Sample Time	Sample Number	Sample Port	Sample Analysis	pH Value	ORP Value	Sampled Time	Initials	Comments
HOUR - 0		Metal Reactor 2	pH	2.8		7:45	DL	
HOUR - 0		Floc Tank	pH	7		7:45		
HOUR - 4		Metal Reactor 2	pH	2.5		11:25		
HOUR - 4		Floc Tank	pH	6.4		11:25		
HOUR - 6		CC1	pH, ORP	7.49	241.9	14:16		
HOUR - 6		CC2	pH, ORP	3.35	459	14:00	DL	
HOUR - 6		CC3	pH, ORP	3.22	310	13:39	DL	
HOUR - 6		CC4	pH, ORP	6.87	-157	13:20	DL	
HOUR - 6		CC5	pH, ORP	4.06	312	13:11	DL	

**Table B-22. Catalyzed Cementation Process Demonstration Test Data Record Follow on Testing**

HOUR - 8		Metal Reactor 2	pH	2.2		15:40	DL	
HOUR - 8		Floc Tank	pH	6.9		15:40	DL	
<b>Comments</b>								
<b>WEEK 1</b>								
<b>DAY 3 (WEEKLY) 4/3/00</b>								
Sample Time	Sample Number	Sample Port	Sample Analysis	pH Value	ORP Value	Sampled Time	Initials	Comments
HOUR - 0		Metal Reactor 2	pH	2.6		8:10	DL	
HOUR - 0		Floc Tank	pH	11.1		8:10	DL	
HOUR - 0		oxidation tank	pH, ORP	5.9	-5.1	8:10	DL	
HOUR - 4		Metal Reactor 2	pH	2.7		12:00	DL	
HOUR - 4		Floc Tank	pH	7		12:00	DL	
HOUR - 6		CC1	pH, ORP	7.64	136	14:50	DL	
HOUR - 6		CC2	pH, ORP	3.3	300	14:40	DL	
HOUR - 6		CC3	pH, ORP	2.5	300	14:18	DL	
HOUR - 6		CC4	pH, ORP	6.62	-202	13:50	DL	
HOUR - 6		CC5	pH, ORP	3.83	320	13:30	DL	
HOUR - 8		Metal Reactor 2	pH	3.1		15:35	DL	
HOUR - 8		Floc Tank	pH	7		15:35	DL	
<b>Comments 8:55--noticed batch tank valve not open. Opened valve and now have process flow,</b>								
<b>WEEK 1</b>								
<b>DAY 4 4/4/00</b>								
Sample Time	Sample Number	Sample Port	Sample Analysis	pH Value	ORP Value	Sampled Time	Initials	Comments
HOUR - 0		Metal Reactor 2	pH	2.5		8:10	BL	
HOUR - 0		Floc Tank	pH	11.5		8:10	BL	
HOUR - 4		Metal Reactor 2	pH	2.3		11:50	DL	
HOUR - 4		Floc Tank	pH	7.8		11:50	DL	
HOUR - 6		CC1	pH, ORP	7.85	61.8	14:45		
HOUR - 6		CC2	pH, ORP	3.22	204	14:32		
HOUR - 6		CC3	pH, ORP	2.75	-30.8	14:20		
HOUR - 6		CC4	pH, ORP	6.7	-223	14:00		
HOUR - 6		CC5	pH, ORP	2.59	458	13:46		
HOUR - 8		Metal Reactor 2	pH	2.8		15:40		
HOUR - 8		Floc Tank	pH	7		15:40		
<b>Comments</b>								
<b>WEEK 1</b>								
<b>DAY 5 4/5/00</b>								
Sample Time	Sample Number	Sample Port	Sample Analysis	pH Value	ORP Value	Sampled Time	Initials	Comments
HOUR - 0		Metal Reactor 2	pH	2.3		8:00	BL	
HOUR - 0		Floc Tank	pH	11.8		8:00	BL	
HOUR - 2	CC5-530	CC5	Dissolved Metals (Fe, As, Cu, Se)					
HOUR - 4		Metal Reactor 2	pH	2.3		11:50	DL	
HOUR - 4		Floc Tank	pH	6.9		11:50	DL	
HOUR - 6		CC1	pH, ORP					
HOUR - 6		CC2	pH, ORP	3.31	366	14:10	DL	
HOUR - 6		CC3	pH, ORP	2.64	-260	14:00	DL	
HOUR - 6		CC4	pH, ORP	7.11	860	13:50	DL	

**Table B-22. Catalyzed Cementation Process Demonstration Test Data Record Follow on Testing**

HOUR - 6		CC5	pH, ORP	2.34	547	13:45	DL	
HOUR - 8		Metal Reactor 2	pH	3.7		15:50	DL	
HOUR - 8		Floc Tank	pH	7.4		15:50	DL	
<b>Comments</b>								
<b>WEEK 2</b>								
<b>DAY 1 4/6/00</b>								
Sample Time	Sample Number	Sample Port	Sample Analysis	pH Value	ORP Value	Sampled Time	Initials	Comments
HOUR - 0		Metal Reactor 2	pH	2.3		7:45	DL	
HOUR - 0		Floc Tank	pH	7.1		7:45	DL	
HOUR - 4		Metal Reactor 2	pH	2.6			DL	
HOUR - 4		Floc Tank	pH	7.2			DL	
HOUR - 6		CC1	pH, ORP	NR	NR		DL	
HOUR - 6		CC2	pH, ORP	NR	NR		DL	
HOUR - 6		CC3	pH, ORP	2.62	-328	10:03	DL	
HOUR - 6		CC4	pH, ORP	7.56	849	9:49	DL	
HOUR - 6		CC5	pH, ORP	2.26	553	9:40	DL	
HOUR - 8		Metal Reactor 2	pH	NR			DL	
HOUR - 8		Floc Tank	pH	NR			DL	
<b>Comments</b>								
CC-Eff3				2.34	514	13:20	DL	
<b>WEEK 2</b>								
<b>DAY 2 4/10/00</b>								
Sample Time	Sample Number	Sample Port	Sample Analysis	pH Value	ORP Value	Sampled Time	Initials	Comments
HOUR - 0		Metal Reactor 2	pH	2.1		8:50	DL	
HOUR - 0		Floc Tank	pH	7.2		8:50	DL	
HOUR - 4		Metal Reactor 2	pH	2.9		12:50	DL	
HOUR - 4		Floc Tank	pH	6.9		12:50	DL	
HOUR - 6		CC1	pH, ORP	NR	NR		DL	
HOUR - 6		CC2	pH, ORP	3.49	22.4	15:50	DL	
HOUR - 6		CC3	pH, ORP	2.66	-336	15:30	DL	
HOUR - 6		CC4	pH, ORP	6.68	-547	15:05	DL	
HOUR - 6		CC5	pH, ORP	3.33	148	14:44	DL	
HOUR - 8		Metal Reactor 2	pH	3.2		15:58	DL	
HOUR - 8		Floc Tank	pH	7		15:58	DL	
<b>Comments</b>								
<b>WEEK 2 (WEEKLY)</b>								
<b>DAY 3 4/11/00</b>								
Sample Time	Sample Number	Sample Port	Sample Analysis	pH Value	ORP Value	Sampled Time	Initials	Comments
HOUR - 0		Metal Reactor 2	pH	3.9		7:59	DL	
HOUR - 0		Floc Tank	pH	7.9		7:59	DL	
HOUR - 4		Metal Reactor 2	pH	3		11:59	DL	
HOUR - 4		Floc Tank	pH	8.7		11:59	DL	
HOUR - 6		CC1	pH, ORP	NR	NR		DL	
HOUR - 6		CC2	pH, ORP	3.53	302	14:21	DL	
HOUR - 6		CC3	pH, ORP	2.93	-130	14:10	DL	
HOUR - 6		CC4	pH, ORP	8.33	-686	14:00	DL	
HOUR - 6		CC5	pH, ORP	2.89	431	13:34	DL	
HOUR - 8		Metal Reactor 2	pH	2.3		15:50	DL	

**Table B-22. Catalyzed Cementation Process Demonstration Test Data Record Follow on Testing**

HOUR - 8		Floc Tank	pH	6.8		15:50	DL	
<b>Comments</b>								
<b>WEEK 2</b>								
<b>DAY 4 4/12/00</b>								
Sample Time	Sample Number	Sample Port	Sample Analysis	pH Value	ORP Value	Sampled Time	Initials	Comments
HOUR - 0		Metal Reactor 2	pH	2.4		7:30	RS	
HOUR - 0		Floc Tank	pH	8.8		7:30	RS	
HOUR - 4		Metal Reactor 2	pH	2.5		11:30	DL	
HOUR - 4		Floc Tank	pH	11.1		11:30	DL	
HOUR - 6		CC1	pH, ORP				DL	
HOUR - 6		CC2	pH, ORP	NR	NR		DL	
HOUR - 6		CC3	pH, ORP	2.85	416	14:25	DL	
HOUR - 6		CC4	pH, ORP	2.66	-350	14:05	DL	
HOUR - 6		CC5	pH, ORP	4.49	127.2	13:40	DL	
HOUR - 8		Metal Reactor 2	pH	2.9		15:50	DL	
HOUR - 8		Floc Tank	pH	11.1		15:50	DL	
<b>Comments</b>								
<b>WEEK 2 (FINAL)</b>								
<b>DAY 5 4/13/00</b>								
Sample Time	Sample Number	Sample Port	Sample Analysis	pH Value	ORP Value	Sampled Time	Initials	Comments
HOUR - 0		Metal Reactor 2	pH	2.3		7:50	DL	
HOUR - 0		Floc Tank	pH	6.5		7:50	DL	
HOUR - 4		Metal Reactor 2	pH	2.6		11:50	DL	
HOUR - 4		Floc Tank	pH	7.3		11:50	DL	
HOUR - 6		CC1	pH, ORP	NR	NR		DL	
HOUR - 6		CC2	pH, ORP	2.97	414	13:35	DL	
HOUR - 6		CC3	pH, ORP	2.91	170	13:20	DL	
HOUR - 6		CC4	pH, ORP	6.93	-504	13:09	DL	
HOUR - 6		CC5	pH, ORP	4.38	1560	13:00	DL	

**Table B-23. Summary data for additional catalyzed cementation tests (aqueous)**

Lab #	Sample Description	Collect Date	Collect Time	Analyte CRDL Units	Nitrate 0.2 mg/L	Sulfate 5 mg/L	Total Arsenic 10 ug/L	Total Copper 10 ug/L	Total Iron 300 ug/L	Total Selenium 40 ug/L	Total by AA Selenium 1 ug/L	Dissolved Arsenic 10 ug/L	Dissolved Copper 10 ug/L	Dissolved Iron 300 ug/L	Dissolved Selenium 40 ug/L	Dissolved by AA Selenium 1 ug/L
3291017	CC1-050	3/28/00	11:45				40	30	320	1880	N/A					
3291019	CC2-053	3/28/00	15:15				40	4760	320	1910	N/A					
000330Q001	CC1-501	3/30/00	N/T		4.7	255	<29	29	<15	1600	N/A	40	12	33	1800	
000330Q002	CC2-502	3/30/00	N/T				<29	490	630000	1600		47	460	500000	1700	
000330Q003	CC3-503	3/30/00	N/T				<29	0.088	670000	210		<29	7	579000	570	
000330Q004	CC4-504	3/30/00	N/T				<29	37	561000	220		11	<1.8	264000	490	
000330Q005	CC5-505	3/30/00	N/T		0.08	2090	<29	42	581000	44		<29	29	536000	410	
000330Q006	CC5-506	3/30/00	N/T									<29	27	389000	410	
000330Q007	CC6-506	3/30/00	N/T									<29	26	382	440	DUPLICATE
000330Q008	CC7-506	3/30/00	N/T									<29	<1.8	<15	<40	BLANK
000330Q009	CC1-507	3/30/00	N/T				<29	21	28	1600		<29	10	29	1800	
000330Q010	CC2-508	3/30/00	N/T				<29	4900	730	1600		<29	4600	600	1700	
000330Q011	CC3-509	3/30/00	N/T				<29	120	584000	360		<29	29	527000	690	
000330Q012	CC4-510	3/30/00	N/T				<29	55	550000	270		<29	<1.8	68000	420	
000330Q013	CC5-511	3/30/00	N/T				<29	48	355000	230		<29	33	328000	520	
000331Q001	CC5-512	3/31/00	9:00				30	380	500000	650		<29	31	444000	980	

**Table B-23. Summary data for additional catalyzed cementation tests (aqueous)**

Lab #	Sample Description	Collect Date	Collect Time	Analyte CRDL Units	Nitrate 0.2 mg/L	Sulfate 5 mg/L	Total Arsenic 10 ug/L	Total Copper 10 ug/L	Total Iron 300 ug/L	Total Selenium 40 ug/L	Total by AA Selenium 1 ug/L	Dissolved Arsenic 10 ug/L	Dissolved Copper 10 ug/L	Dissolved Iron 300 ug/L	Dissolved Selenium 40 ug/L	DissolvedbyAA Selenium 1 ug/L
000331Q002	CC3-512	3/31/00	9:00				<29	18	75500	790		<29	11	82800	890	
000331Q003	CC1-513	3/31/00	13:00				<29	24	22	1700		60	20	25	2000	
000331Q004	CC2-514	3/31/00	13:00				36	3200	340	1600		<29	3200	310	1800	
000331Q005	CC3-515	3/31/00	13:00				45	88	405000	720		<29	24	419000	1000	
000331Q006	CC4-516	3/31/00	13:00				<29	57	386000	720		<29	<1.8	81100	970	
000331Q007	CC5-517	3/31/00	13:00				<29	18	53900	850		<29	10	60900	890	
000331Q008	CC-EFF1	3/31/00	13:00				<29	23	768000	120		<29	11	1000000	660	
000404L007	CC5-518	4/4/00	N/T									<29	15	26000	880	
000404L008	CC1-519	4/4/00	N/T				<29	23	<15	1500		41	19	21	1800	
000404L009	CC2-520	4/4/00	N/T				<29	6400	710	1500		48	6100	700	1600	
000404L010	CC3-521	4/4/00	N/T				<29	58	675000	350		<29	20	607000	140	
000404L011	CC4-522	4/4/00	N/T				<29	16	270000	320		<29	3	176000	280	
000404L012	CC5-523	4/4/00	N/T				<29	13	46900	740		<29	10	45200	800	
000405J001	CC5-524	4/4/00	N/T									<29	260	201000	470	
000405J002	CC6-524	4/4/00	N/T									<29	260	204000	460	DUPLICATE
000405J003	CC7-524	4/4/00	N/T									<29	<2	25	<40	BLANK
000405J004	CC1-525	4/4/00	N/T				<29	22	210	1500		38	15	17	1700	
000405J005	CC2-526	4/4/00	N/T				<29	6500	1700	1500		<29	6200	2000	1600	
000405J006	CC3-527	4/4/00	N/T				75	58	3690000	<40	48	<29	17	3600000	<40	28
000405J007	CC4-528	4/4/00	N/T				42	120	2030000	670		<29	<18	228000	540	
000405J008	CC5-529	4/4/00	N/T				<29	96	110000	730		<29	91	113000	790	
000405P001	CC5-530	4/5/00	9:50									<29	140	404000	460	
000405P002	CC-EFF2	4/5/00	10:40				<29	39	67800	640		<29	38	71800	710	
000405P003	CC2-532	4/5/00	9:50				<29	6200	630	1500		34	6000	810	1700	
000405P004	CC3-533	4/5/00	9:50				<29	71	504000	840		<29	22	520000	650	
000405P005	CC4-534	4/5/00	9:50				<29	61	420000	770		<29	4	21	840	
000405P006	CC5-535	4/5/00	9:50				<29	72	189000	730		<29	70	209000	720	
000406K001	CC3-539	4/6/00	N/T				<29	120	473000	870		<29	75	495000	700	
000406K002	CC4-540	4/6/00	N/T				<29	94	660000	770		<29	<1.8	<15	920	
000406K003	CC5-541	4/6/00	N/T				<29	20	29100	750		<29	18	30800	820	
4070927	CC-EFF3	4/6/00	13:30				<29	27	48000	810		<29	27	48000	810	
000411J003	CC2-544	4/10/00	N/T				<58	910	746000	370		<58	10300	846000	400	
000411J004	CC3-545	4/10/00	N/T				63	170	3100000	<80	9	60	28	3580000	<58	4
000411J005	CC4-546	4/10/00	N/T				120	310	4110000	<48	42	<58	<1	305000	<48	12
000411J006	CC5-547	4/10/00	N/T				<58	1.8	24500	520		<29	1900	24600	490	
000411P001	CC5-548	4/11/00	N/T									60	310	495000	<48	<1
000411P002	CC2-550	4/11/00	N/T				31	9700	882000	360		<150	10000	947000	<190	
000411P003	CC3-551	4/11/00	N/T				62	560	2710000	<48	18	<58	66	139000	<48	<1
000411P004	CC4-552	4/11/00	N/T				100	410	3140000	<48	13	<58	15	67500	<48	<1
000411P005	CC5-553	4/11/00	N/T		<50	1950	<58	64	127000	<48	13	<58	360	2940000	<48	<1
000413K004	CC2-556	4/12/00	9:30				<29	9500	341000	490		32	9600	315000	530	
000413K005	CC3-557	4/12/00	9:30				86	1500	3460000	<48	14	<150	400	3560000	<48	<1
000413K006	CC4-558	4/12/00	9:30				<58	810	1880000	<48	44	<58	520	1780000	<48	13
000413K007	CC5-559	4/12/00	9:30				<58	800	1520000	<48	35	<58	810	1630000	<48	11
000414I001	CC5-560	4/13/00	N/T									<58	<14	3130000	<48	<1
000414I002	CC6-560	4/13/00	N/T									<290	<18	3240000	<400	
000414I003	CC7-560	4/13/00	N/T									<29	<1.8	<15	<40	
000414I004	CC2-567	4/13/00	N/T				<29	9400	345000	480		<150	9700	337000	630	
000414I005	CC3-568	4/13/00	N/T				100	3900	5520000	<48	14	<150	1000	5910000	<48	1
000414I006	CC4-569	4/13/00	N/T				130	3600	4370000	<48	8	<58	4	2990000	<48	<1
000414I007	CC5-570	4/13/00	N/T		1.3	6000	<58	<1.6	2330000	<48	13	<150	<4	2580000	<48	2.4
000519P005	CC-Eff-3-0517	5/17/00	14:15							730						
000519P006	CCEff-4-0517	5/17/00	14:15							150						
000519P007	CCEff-5-0517	5/17/00	14:15							140						

**Table B-24. Summary data for additional catalyzed cementation tests (solid)**

Lab #	Sample Description	Collect Date	Collect Time	TCLP Arsenic mg/L	TCLP Barium mg/L	TCLP Cadmium mg/L	TCLP Selenium mg/L	Total Arsenic mg/kg	Total Barium mg/kg	Total Cadmium mg/kg	Total Calcium mg/kg	Total Chromium mg/kg	Total Copper mg/L	Total Iron mg/kg	Total Mercury mg/kg	Total Selenium mg/kg	Total Zinc mg/kg	Lead mg/kg
4111040	CC-Filtercake	4/7/00	10:30	<0.029	0.057	0.07	<0.04	10.9	11.7	13.2	37300	57.1	256	5E+05	0.054	19.2	2610	<6

## **APPENDIX C**

### Sampling Schedule and Analytical Protocols

## **C.0 INTRODUCTION**

The following sections describe the analytical protocols, the field measurement protocols, and the sampling schedules for each technology.

### **C.1 TOTAL SELENIUM, SELENITE, AND SELENATE**

Selenium and selenite were determined using a hydride generation inductively coupled plasma-mass spectrometry (ICP-MS) procedure at KEL according to SW-846 Method 7742 (Modified Cutter Method) as outlined in *Test Methods for Evaluation of Solid Waste-Physical/Chemical Methods (SW-846)* (Ref. 1). Selenite was determined directly by hydride generation. Total selenium was determined by oxidizing all selenium in the sample to selenate in a potassium persulfate-nitric acid digestion followed by reduction to selenite with hydrochloric acid (HCl). Selenate was calculated as the difference between total selenium and selenite.

### **C.2 DISSOLVED, TOTAL RECOVERABLE, AND TOXICITY CHARACTERISTIC LEACHING PROCEDURE METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA SPECTROMETER**

Dissolved and total recoverable metals will be determined using SW-846 Method 6010B using an inductively coupled plasma atomic emission spectrometer (ICP-AES) or SW-846 Method 6020 using ICP-MS. The samples were prepared for ICP analysis as outlined in SW-846 Method 3005A.

The ICP-AES was calibrated according to the procedures outlined in SW-846 Method 6010B and the equipment manufacturer's instructions. The ICP-MS was calibrated according to the procedures outlined in SW-846 Method 6020 and the manufacturer's instructions.

### **C.3 pH**

Although process pH measurements were made through installed probes, some pH measurements were done manually using a hand-held probe. A pH meter with automatic temperature compensation capable of measuring pH at the demonstration site to  $\pm 0.1$  pH units was used for this project. The pH probe was calibrated daily using two fresh buffer solutions that bracket the expected pH. Temperature values were also be recorded from the readout during pH measurements.

### **C.4 ORP**

An ORP meter with a silver/silver chloride reference electrode was used to determine the ORP at the demonstration site. The electrode was calibrated using a solution of known ORP. The calibration procedures were conducted for every measurement set, and measurements for the biological process were performed under anerobic and anaerobic conditions.

## **C.5 DISSOLVED OXYGEN**

Dissolved oxygen was measured using a dissolved oxygen meter at the demonstration site. The meter was calibrated using a sodium sulfite with a trace of cobalt chloride solution to represent 0% dissolved oxygen and atmospheric air to represent 100% dissolved oxygen. Adjustments for barometric pressure and salinity were made following calibration, as indicated in the manufacturer's instructions.

## **C.6 SULFATE**

Sulfate analyses were performed according to SW-846 Method 9036. The auto-analyzer was calibrated using at least five calibration standards of appropriate concentrations.

## **C.7 TOTAL SUSPENDED SOLIDS/TOTAL DISSOLVED SOLIDS**

To determine how the filtering system was functioning, total suspended solids (TSS) and total dissolved solids (TDS) were determined at KEL according to EPA Method 160.2 and EPA Method 160.1, respectively. These methods are contained in EPA's Methods for Chemical Analyses of Water and Wastes (Ref. 2).

## **C.8 IRON SPECIATION**

The concentration of dissolved iron will be determined by ICP-AES at KEL. The concentration of ferrous iron will be determined using the colorimetric Standard Methods for the Examination of Water and Wastewater (Ref. 3) Method 3500-Fe B and phenanthroline as the color developer.

## **C.9 TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)**

Solid materials from the ferrihydrite adsorption and catalyzed cementation processes were subjected to the TCLP procedure outlined in SW-846 Method 1311 at KEL. If sufficient sample was not available from filter-cake samples, the TCLP procedure was modified according to the weight of the solids submitted for analysis. The amount of extraction fluid added to the sample was determined by the weight of the sample and was adjusted according to the sample weight. All reagent additions will be adjusted accordingly. The resulting extraction fluids from the TCLP were digested according to procedures outlined in SW-846 Method 3005A for total recoverable metals. Digested samples were analyzed by ICP-AES according to SW-846 Method 6010B. Splits of TCLP extracts were prepared/analyzed for mercury by cold vapor atomic absorption (CVAA) according to procedures outlined in SW-846 Method 7470A.

## **C.10 TOTAL METALS**

The solid samples were characterized for total metals by ICP SW-846 Method 6010B at KEL. Samples were digested according to SW-846 Method 3050A. The ICP-AES was calibrated according to SW-846 Method 6010B. Mercury in solid samples was determined according to procedures outlined in SW-846 Method 7471A.

## **C.11 PERCENT MOISTURE**

The percent moisture of each solid sample was determined at KEL using the method outlined in Exhibit D, Part F of the USEPA Contract Laboratory Program Statement of Work to Inorganics Analysis, Document Number Ilm03.0 (Ref. 4). The percent moisture data will be used to report the metals on a dry weight basis. Although the method specifies percent solids, percent moisture was reported by the laboratory.

## **C.12 MICROBIAL ISOLATION AND CHARACTERIZATION**

All samples were stored at -4 EC to inhibit microbial growth until analysis. Before samples were tested they were allowed to warm to ambient temperature and vortexed to ensure a representative sample for plating. Plate counts were obtained using the standard laboratory procedure using 0.1 mL of sample or sample dilution.

All plate counts, including plating to isolate individual colony types, were done at room temperature on trypticase soy agar (TSA) plates and TSA plates containing 25-mg/L selenium. Plates were incubated 24 to 48 hr in a constant temperature incubator at 28 °C, at ambient temperature, and in a COY anaerobic chamber.

The baseline microbial characterization portion of the testing included microbial isolations and plate counts. Microbial isolations were performed on trypticase soy agar (TSA) using the streak-plate method. All culturing was performed in a Class II Laminar Flow hood. Isolates were initially characterized by colony morphology and gram stain, and isolates were slanted on appropriate media for future testing. Microbial counts were performed on the provided waters using the standard plate count method (Ref. 3). Samples with low numbers of organisms present in the sample were concentrated 1:50 using centrifugation to achieve a representative plate count. Plate counts are reported in colony forming units (CFU)/mL. Selected site isolates capable of selenate to elemental selenium reduction were further characterized using the BIOLOG™ metabolic profiling system and by MIDI Labs fatty acid analysis. The following characterizations were completed on all samples collected:

- total heterotrophs—nonselenium reducers (CFU);
- total aerobes (CFU);
- total anaerobes (CFU);
- total selenium reducers—aerobic (CFU); and
- total selenium reducers—anaerobic (CFU).

The following analyses were completed on selected samples:

- BIOLOG™;
- MIDI profiles of predominant heterotrophs (nonselenium reducers); and
- MIDI profiles of selenium reducers.

### **C.12.1 Total Heterotrophs/Total Selenium Reducers**

Plate counts of total heterotrophs and total selenium reducers were made under aerobic and anaerobic conditions to profile the site microorganisms and to determine potentially interfering nonselenium reducing microbes. This profile was later used to judge the general reactor conditions with respect to the desired microbial population. Total heterotroph plate counts were conducted using standard log dilutions and plating techniques that used 0.1 mL per TSA plate. Colonies forming on the plates were enumerated within 24 to 48 hr under aerobic conditions and up to two week for anaerobes. Selenium reducers were enumerated using the same techniques with the exceptions of using TSA plates with 25-mg/L sodium selenate added.

### **C.12.2 BIOLOG™ and MIDI Fatty Acid Analysis**

Where appropriate, BIOLOG™ plates were used to provide tentative microbial identification, and to help characterize the metabolic profiles of microbes important in the selenium reduction process. BIOLOG™ plates provide a profile of 96 carbon sources or selected carbon sources to profile the metabolic character or individual microorganisms.

MIDI fatty acid profiles were used where appropriate to fingerprint the microbial population for bioreactor tests. Selected heterotrophic and selenium reducing isolates were obtained by plating an isolate for purity a minimum of three times on TSA plates. The isolate was streaked through four quadrants and incubated at 28 °C for 24 hr, harvesting approximately 50 to 75 mg of microbial cells from the third and fourth quadrants. These microbial cells were used to prepare a hexane fatty acid extract. The fatty acid extracts were injected into a micro-bore gas chromatograph column designed to separate fatty acids and analyzed using MIDI microbial identification software and databases.

## **C.13 SAMPLING LOCATIONS/SCHEDULE**

The sampling locations for each process as well as the sampling schedule for each process are defined in the following tables. The sampling schedules were originally developed in the project-specific QAPP. Table C-1 describes the sampling locations for the ferrihydrite adsorption process, and Table C-2 is the sampling schedule for the ferrihydrite adsorption process. Table C-3 describes the sampling locations for the catalyzed cementation process, and Table C-4 is the sampling schedule for the catalyzed cementation process. Table C-5 describes the sampling locations for the BSeR™ process, and Table C-6 is the sampling schedule for the BSeR™ process. The preservative, holding times, and analytical protocols for each sample type are summarized in Table C-7. The frequency of field QC sampling is summarized in Table C-8.

**Table C-1. Sample port/location descriptions and sample matrix at each location for the ferrihydrite process.**

Sample Port/Sample Location	Description	Matrix
FH1	Process influent	Aqueous
FH2	Process influent after FeCl <sub>2</sub> addition	Aqueous
FH3	Process influent with HCl and CaO addition	Aqueous
FH4	Treated water discharge	Aqueous
FH5	Unfiltered discharge	Aqueous
FH Filter cake	Sludge product	Solid
FE/FT	Flow Totalizer	Aqueous
ORP	Tank 101	Aqueous
pH	Tanks 201, 203, and 204 pH monitors	Aqueous

**Table C-2 Noncritical and critical measurements for the ferrihydrite adsorption tests.**

Measurement	Matrix	Classification	Sample Frequency	Sample Location	Total Number of Samples
pH	Aqueous	Noncritical	Initially, every 4 hr for 2 days, every 8 hr for 3 days, daily	pH probes in tank 101, tank 102, and tank 103	114
pH	Aqueous	Noncritical	Initial, every 24 hr	FH5	22
ORP	Aqueous	Noncritical	Initially, every 4 hr for 2 days, every 8 hr for 3 days, daily	ORP probes in tank 102	38
Total Flow	Aqueous	Noncritical	Initially, every 4 hr for 2 days, every 8 hr for 3 days, daily	FE/FT (Total flow indicator)	38
Selenium Speciation	Aqueous	Noncritical	Initial, every tanker truck delivery, final	FH1	4
Selenium Speciation	Aqueous	Noncritical	Initial, daily for 5 days, weekly	FH5	8
Iron Speciation	Aqueous	Noncritical	Initial, every tanker truck delivery, final	FH1	4
Sulfate	Aqueous	Noncritical	Initial, every 48 hr of operation, final	FH1, FH5	24
Nitrate-Nitrite as N	Aqueous	Noncritical	Initial, every 48 hr of operation, final	FH1, FH5	24
Total Suspended Solids	Aqueous	Noncritical	Initial, weekly, final	FH4 and FH5	10
Total Dissolved Solids	Aqueous	Noncritical	Initial, weekly, final	FH4 and FH5	10
Total Recoverable Metals (Ca, Fe, Mg, Na, As, Ba, Cu, Mo, Se)	Aqueous	Noncritical	Initial, every 48 hr of operation, final	FH1, FH2, FH3, FH4, FH5	60

**Table C-2 Noncritical and critical measurements for the ferrihydrite adsorption tests.**

Measurement	Matrix	Classification	Sample Frequency	Sample Location	Total Number of Samples
Dissolved Metals (Ca, Mg, Na, Ba, Cu, Mo, Se)	Aqueous	Noncritical	Initial, every 48 hr of operation, final	FH1, FH2, FH3, FH4, FH5	60
Dissolved Metals (As, Fe)	Aqueous	Noncritical	Initially, every 24 hr of operation	FH3, FH5	44
Total Metals (As, Ba, Cd, Cr, Cu, Fe, Pb, Se, Ag, Zn, Ca)	Solid	Noncritical	Each sludge sample	FH Filter cake	3
% Moisture	Solid	Noncritical	Each sludge sample	FH Filter cake	3
TCLP (As, Ba, Cd, Cr, Pb, Hg, Se, Ag)	Solid	Noncritical	Each sludge sample	FH Filter cake	3
Dissolved Metals (Se)	Aqueous	Critical	Initially, every 4 hr for 2 days, every 8 hr for 3 days, daily	FH5	38

Note: Sample collection will begin after the one system volume has been processed.

**Table C-3. Sample port/location descriptions and sample matrix at each location for the catalyzed cementation process.**

Sample Port/Sample Location	Description	Matrix
CC1	Process influent	Aqueous
CC2	Process influent after reagent addition	Aqueous
CC3	Process influent with additional reagents	Aqueous
CC4	Unfiltered discharge	Aqueous
CC5	Treated water discharge	Aqueous
CC Filter cake	Sludge product	Solid
FE/FT	Flow totalizer	Aqueous
ORP	Tanks 108 and 109	Aqueous
pH	Tanks 201, 203, and 204 pH monitors	Aqueous

**Table C-4. Noncritical and critical measurements for catalyzed cementation process demonstration (3-week test).**

Measurement	Matrix	Classification	Sample Frequency	Sample Location	Total Number of Samples
pH	Aqueous	Noncritical	Initially, every 4 hr for 2 days, every 8 hr for 3 days, daily	pH probes in tanks 108 and 109	76
pH	Aqueous	Noncritical	initial, every 24 hr	PC5	22
ORP	Aqueous	Noncritical	Initially, every 4 hr for 2 days, every 8 hr for 3 days, daily	ORP probes in tanks 108 and 109	76
ORP	Aqueous	Noncritical	Initial, every 24 hr	PC3	22
Total Flow	Aqueous	Noncritical	Initially, every 4 hr for 2 days, every 8 hr for 3 days, daily	FIT (Total flow indicator)	38
Selenium Speciation	Aqueous	Noncritical	Initial, every tanker truck delivery	PC1	4
Selenium Speciation	Aqueous	Noncritical	Initial, daily for 5 days, weekly	PC5	8
Iron Speciation	Aqueous	Noncritical	Initial, every tanker truck delivery	PC1	4
Sulfate	Aqueous	Noncritical	Initial, every 48 hr of operation, final	PC1, PC5	24
Nitrate-Nitrite as N	Aqueous	Noncritical	Initial, every 48 hr of operation, final	PC1, PC5	24
Total Suspended Solids	Aqueous	Noncritical	Initial, weekly, final	PC4 and PC5	10
Total Dissolved Solids	Aqueous	Noncritical	Initial, weekly, final	PC4 and PC5	10
Total Recoverable Metals (Ca, Fe, Mg, Na, As, Ba, Cu, Mo, Se)	Aqueous	Noncritical	Initial, every 48 hr of operation, final	PC1, PC2, PC3, PC4, PC5	60
Dissolved Metals (Ca, Mg, Na, Ba, Cu, Mo, Se)	Aqueous	Noncritical	Initial, every 48 hr of operation, final	PC1, PC2, PC3, PC4, PC5	60
Dissolved Metals (As, Fe)	Aqueous	Noncritical	Initially, every 24 hr of operation	PC3, PC5	44
Total Metals (As, Ba, Cd, Cr, Cu, Fe, Pb, Se, Ag, Zn, Ca)	Solid	Noncritical	Each Sludge Sample	PC Filter cake	3
% Moisture	Solid	Noncritical	Each Sludge Sample	PC Filter cake	3
TCLP (As, Ba, Cd, Cr, Pb, Hg, Se, Ag)	Solid	Noncritical	Each Sludge Sample	PC Filter cake	3
Dissolved Metals (Se)	Aqueous	Critical	Initially, every 4 hr for 2 days, every 8 hr for 3 days, daily	PC5	38

Note: Sample collection will begin after the one system volume has been processed.

**Table C-5. Sample port/location descriptions and sample matrix at each location for the biological selenium reduction process.**

Sample Port/ Sample Location	Description	Matrix
BR01	Process influent	Aqueous
BR02	Process influent after nutrient addition and first reactor	Aqueous
BR03	Process water exiting second reactor	Aqueous
BR04	Process water exiting third reactor	Aqueous
BR05	Process water after exiting fourth reactor	Aqueous
BR06	Process water after exiting fifth reactor	Aqueous
BR07	Process water after exiting sixth reactor	Aqueous
BR08	Final process effluent after slow sand filter	Aqueous
Bioreactor	Selenium precipitate product	Solid
Flowmeter/Totalizer	Flowmeter/totalizer for biological reduction system	Aqueous

**Table C-6. Noncritical and critical measurements for demonstration of the biological selenium reduction ( 1-week test at residence times of approximately 24 hr, 12 hr, 6 hr, 3 hr, and repeat of optimum).**

Measurement	Matrix	Classification	Sample Frequency	Sample Location	Total Number of Samples
pH	Aqueous	Noncritical	Daily	BR01 through BR08	up to 1,224
Temperature	Aqueous	Noncritical	Daily	BR01 through BR08	up to 1,224
Dissolved Oxygen	Aqueous	Noncritical	Daily	BR01 through BR08	up to 1,224
ORP	Aqueous	Noncritical	Daily	BR01 through BR08	up to 1,224
Flow Rate/Total Flow	Aqueous	Noncritical	Daily	Flowmeter/Totalizer	up to 153
Total Recoverable Metals (Ca, K, P, Mg, Na, As, Ba, Cu, Mo, Se)	Aqueous	Noncritical	Weekly	BR01 through BR08	up to 176
Dissolved Metals (Ca, K, P, Mg, Na, As, Ba, Cu, Mo, Se)	Aqueous	Noncritical	Weekly	BR01 through BR08	up to 176
Nitrate-Nitrite as N	Aqueous	Noncritical	Weekly	BR01 through BR08	up to 176
Cell Count	Aqueous/Solid	Noncritical	Initial, weekly, final	Bioreactors	up to 144
MIDI Fatty Acid Analysis	Aqueous/Solid	Noncritical	Initial and final	Bioreactors	up to 12

**Table C-6. Noncritical and critical measurements for demonstration of the biological selenium reduction ( 1-week test at residence times of approximately 24 hr, 12 hr, 6 hr, 3 hr, and repeat of optimum).**

Measurement	Matrix	Classification	Sample Frequency	Sample Location	Total Number of Samples
Selenium Speciation	Aqueous	Noncritical	Initially, daily during residence times tests, then weekly	BR01 through BR08	up to 424
Total Metals (As, Ba, Cd, Cr, Cu, Fe, Pb, Se, Ag, Zn)	Solid	Noncritical	Each product sample	Bioreactor	up to 5
% Moisture	Solid	Noncritical	Each product sample	Bioreactor	up to 5
Dissolved Metals (Se)	Aqueous	Critical	Initially, daily during residence times tests, then weekly	BR08	53

Note: Sample collection will begin after the one system volume has been processed.

**Table C-7. Preservatives, holding times, containers, method types, and references.**

Parameter	Matrix	Preservative	Holding Time	Sample Size & Container	Method Type	Reference
Selenium Speciation	Aqueous	#4EC, Filter, pH#2 HCl	Analyze immediately	500-mL HDPE	AA hydride generation	See Section 5.1, Modified SW-846 Method 7742
Iron Speciation	Aqueous	#4EC, Filter, pH#2 HCl	Analyze immediately	500-mL HDPE	Colorimetric	Standard Methods 3500-Fe B, Appendix C
pH	Aqueous	None	Analyze immediately	100-mL HDPE	pH meter	EPA (SW-846) Method 9040
Dissolved Oxygen	Aqueous	None	Analyze immediately	100-mL HDPE	DO meter	EPA (SW-846) Method 9040
Temperature	Aqueous	None	Analyze immediately	100-mL HDPE	Thermometer	EPA (SW-846) Method 9040
ORP	Aqueous	None	Analyze immediately	100-mL HDPE	ORP meter	Equip. Manufacturer instructions
Flow Rate/Total Flow	N/A	None	Analyze immediately	N/A	Flowmeter/Totalizer	Manufacturer's Instructions
Sulfate	Aqueous	#4°C	28 days	500-mL HDPE	Colorimetric	EPA Method 375.2
Nitrate-Nitrite asN	Aqueous	#4EC, pH#2 H <sub>2</sub> SO <sub>4</sub>	28 days	500-mL HDPE	Colorimetric	EPA Method 353.3
TDS	Aqueous	#4°C	7 days	500-mL HDPE	Filter/Weigh	EPA Method 160.2
TSS	Aqueous	#4°C	7 days	500-mL HDPE	Filter/Weigh	EPA Method 160.1
Total Recoverable Metals (Al, As, Cd, Cu, Fe, Pb, P, Zn by ICP)	Aqueous	#4EC, pH#2 HNO <sub>3</sub>	6 Months	500-mL HDPE	ICP	EPA SW-846 Preparation Method 3005A/ICP Method 6010B
Dissolved Metals (Se by ICP-MS)	Aqueous	#4EC, Filter, pH#2 HNO <sub>3</sub>	6 Months	500-mL HDPE	ICP-MS	EPA SW-846 Preparation Method 3005A/ICP-MS Method 6020
Dissolved Metals by ICP-AES or ICP-MS	Aqueous	#4EC, Filter, pH#2 HNO <sub>3</sub>	6 Months	500-mL HDPE	ICP	EPA SW-846 Preparation Method 3005A/ICP Method 6010B or 6020
Total Metals by ICP-AES (Hg by CVAA)	Solid	None	6 Months	8 oz CWM	ICP	EPA SW-846 Preparation Method 3050A/ICP Method 6010B (Hg Method 7471A)
MIDI Fatty Acid Analysis	Aqueous/Solid	None	48 hr after colony isolation	15mL HDPE	Gas Chromatograph (GC)	See Section 5.12
Cell Counts	Aqueous/Solid	None	48 hr	15 mL HDPE	Plate Count	See Section 5.12
% Solids	Solid	None	6 Months	Taken from solid sample	Drying/Weighing	CLP SOW 3/90 Exhibit D, Part F and Appendix C
TCCLP Metals (Hg by CVAA)	Solid	None	7 days to extraction, 40 days after, 28 days until extraction, 28 days until analysis of extract	at least 100 g 16 oz CWM	ICP	EPA SW-846 Extraction Method 1311/Preparation Method 3005 <sup>3</sup> /ICP Method 6010B (Hg Method 7470A)

**Table C-8. Field QC sampling for each process demonstration.**

Process	Field Duplicates Frequency	Field Cross Contamination Blanks Frequency	Total Number of Field QC Samples
ABC Biological Process	weekly	weekly	23 field duplicates and 23 field blanks
MSE Catalyzed Cementation	weekly	weekly	4 field duplicates and 4 blanks
MSE Ferrihydrite Adsorption	weekly	weekly	4 field duplicates and 4 blanks
<sup>1</sup> Field QC samples are to be taken at the initial sampling event and then weekly for each technology demonstration. The field duplicate samples will be taken from the effluent location of each process.			

## REFERENCES

1. U.S. Environmental Protection Agency, "Test Methods for Evaluating Solid Waste—Physical/Chemical Methods," U.S. EPA, Washington D.C., 1990 through Update IIB, January 1995.
2. U.S. Environmental Protection Agency, "Methods for Chemical Analyses of Water and Wastes."
3. American Public Health Association (APHA), "Standard Methods for the Examination of Water and Wastewater," 16th Edition, 1985.
4. U.S. Environmental Protection Agency, "USEPA Contract Laboratory Program Statement of Work to Inorganics Analysis," Document number ILM03.0, Washington D.C., June 1992.

## **APPENDIX D**

### Microbial Screening and Laboratory Testing

## **D.1 MICROBIAL SELENIUM REDUCTION SCREENING**

Endpoint, qualitative, and quantitative selenium reduction assays were utilized as screening tools to assess selected microbial strains and microbial support materials for selenium reduction. The selenium test water used for the screening series consisted of Garfield Wetlands-Kessler Springs water collected by KUCC and used unspiked (2-mg/L selenium) and spiked (25-mg/L selenium). Screening tests used log-phase microbial cultures prepared in trypticase soy broth (TSB), washed and resuspended in sterile saline, and inoculated into 15-mL culture tubes containing selenium test water at a concentration of  $2 \times 10^9$  cells per mL. Sterile saline served as the abiotic control. Tubes were incubated in both an aerobic environment and a COY anaerobic chamber at room °C for 24 to 48 hr and then evaluated for selenium reduction.

## **D.2 NUTRIENT SCREENING**

Endpoint, qualitative, and quantitative selenium reduction assays were utilized as screening tools to assess selected microbial strains and microbial support materials for selenium reduction using various supplementary nutrients. The selenium test water used for the screening series consisted of Garfield Wetlands-Kessler Springs water collected by KUCC and used unspiked and spiked to a final selenate concentration of 25 mg/L. Screening tests used log-phase microbial cultures prepared in TSB, washed and resuspended in sterile saline, and inoculated into 15-mL culture tubes containing selenium test water and or selected nutrient(s) at a concentration of  $2 \times 10^9$  cells per mL. Nutrients screened for selenium reduction included acetate, an acetate nutrient mix-1, methanol, several proprietary molasses-based nutrient mixes, ammonium phosphate, ammonium phosphate nutrient mix-1, and a peptone-based nutrient. Nutrient mixes are proprietary and significantly effect selenium reduction over extended periods. Nutrient-selenium containing media without microorganisms and selenium containing media without nutrients were used as controls. Tubes were incubated in both aerobic environments and a COY anaerobic chamber at room °C for 24 to 48 hr and then evaluated for selenium reduction.

## **D.3 MICROBIAL SUPPORT MATERIALS**

Microbial support materials were evaluated for selenium-reduction at the Garfield Wetlands-Kessler Springs site because of KUCC's desire to test alternative biofilm support materials. Materials tested included slag and biosolids obtained from KUCC that were screened to +8 mesh, Darco charcoal +8 mesh, Celite, and continuous-release microbe-containing alginate beads. Continuous release beads were prepared for sustained reactor inoculation with desired microbes. Controls were used to determine possible sorption of dissolved selenium to materials used in the proposed testing. Sorption tests were conducted with biofilm support materials (50% by volume to approximate reactor conditions) under static conditions at ambient temperature for 2, 4, and 8 hr. Tests used prewetted biofilm support materials, 25–100 mL of actual process water, and were conducted as shown in Table D-1 below.

**Table D-1. Support material test matrix.**

Test Condition	Process Water	Process Water with Nutrients	Process Water with Se (25 mg/L)
Test Material			
Carbon Support	X	X	X
Biosolids Support	X	X	X
Live Cells	X	X	X
Heat Inactivated Cells	X	X	X

#### **D.4 LABORATORY BIOREACTOR/BIOPROCESS TESTS**

Staggered sets of anaerobic up-flow bioreactors were used to evaluate preliminary BSeR™ operating parameters, economics, retention time, flow rate, system kinetics, nutrients and overall system performance. All tests were conducted in one-inch diameter columns operated in single-pass, up-flow mode with retention times ranging from 3 to 24 hr, at ambient temperature (-24 EC). The bioreactors used a defined microbial cocktail of *Pseudomonas* and other site bacteria to provide scale-up estimates for pilot-scale application. All tests used provided KUCC waters and live microbial biofilms. Bioreactors used an agricultural-grade molasses based media. Kinetic determinations were made over a 2-week period by varying retention time and measuring selenium in the effluent. Controls used granular activated carbon, slag, Celite and/or biosolids without microorganisms.

#### **D.5 RESULTS**

Results of the microbial screening and laboratory testing are discussed in the following sections.

##### **D.5.1 Microbial Isolation and Characterization**

Microbes were characterized through plating samples, noting colony morphology, gram stains, BIOLOG™ plates, and MIDI fatty acid profiles when appropriate. Multiple site and previously collected microbial isolates were tested for the ability to remove selenium in unspiked and spiked KUCC waters and synthetic waters (to 25 mg/L selenium as sodium selenate). Each isolate was plated on TSA containing 25 mg/L selenium. These plates were incubated in both aerobic and anaerobic environments and screened for selenium reduction. Microbial characterization results are shown in Table D-2.

A number of site isolates were also nonselenium reducers. Table D-3 below lists some of the nonselenium reducers of concern in developing a selenium reducing microbial cocktail biofilm that would resist replacement by indigenous nonselenium reducing microbes.

**Table D-2. Garfield Wetlands-Kessler Springs microbial characterization.**

Sample Name	Total Plate Count	Selenium Reducers	Non-Selenium Reducers
KS001	2.05E+04	7.50E+03	1.30E+03
Stake 1		Four different colonies	Three different colonies
KS002	1.60E+06	1.53E+05	6.00E+03
E. Seep Black		Three different colonies	One colony
KS003	1.71E+05	1.50E+05	2.10E+04
White N of Stake		Three different colonies	One colony (rapid growth)
KS004	1.40E+06	7.00E+05	7.00E+05
E. Seep		Three different colonies	Four different colonies
KS005	9.00E+04	7.90E+04	1.10E+04
Stake 2		Four different colonies	One colony
KS006	1.90E+05	1.80E+05	1.00E+04
Sample from pool with stake		Four different colonies	One colony
KS007	1.63E+04	1.48E+04	1.50E+03
Sample from pool with stake		Three different colonies	Two different colonies
KS008	3.60E+05	3.00E+05	6.00E+04
Sample from pool with stake		Three different colonies	Three different colonies
KS009	4.00E+05	1.95E+05	2.05E+05
Sample from pool with stake – Channel		Three different colonies	Two different colonies

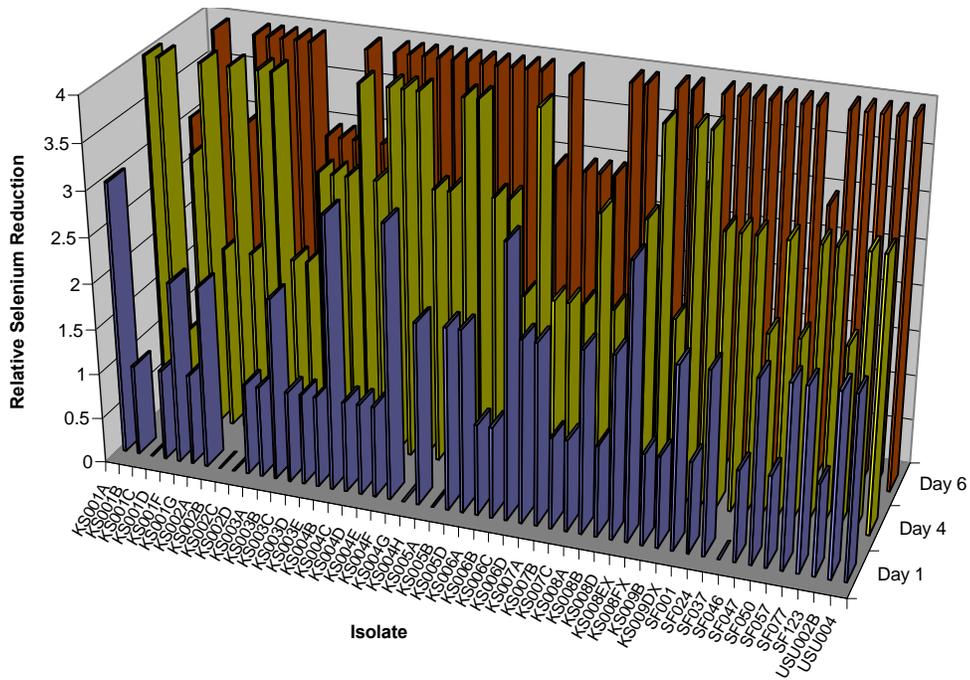
**Table D-3. Nonselenium reducing site isolates.**

Nonselenium Reducers	Aerobic Growth	Anaerobic Growth	Gram-Stain	Biolog ID
KS003AX	+	-	-	<i>Pseudomonas fluorescens type c</i>
KS001CX	+	-	-	No Identification
KS009E	+	-	-	No Identification
KS001E	+	-	-	<i>Pseudomonas putida</i>
KS007D	+	-	-	<i>Pseudomonas corrugata</i>
KS003F	+	-	+	<i>Bacillus sp.</i>
KS007E	+	-	-	<i>Pseudomonas fragi</i>
KS004A	+	-	-	<i>Pseudomonas fluorescens type G</i>
KS005CX	+	-	-	<i>Pseudomonas mendocina</i>

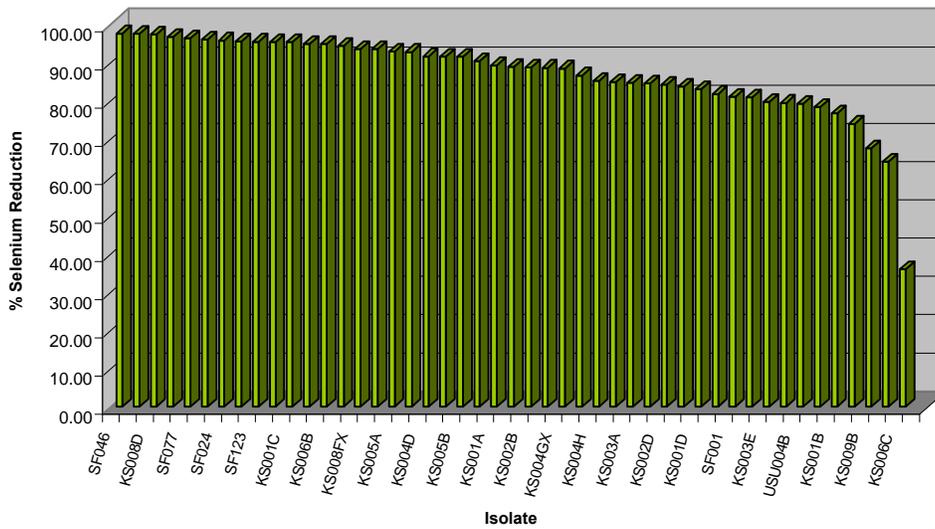
### D.5.2 Microbial Selenium Reduction Screening

Selected microbes were tested for their ability to reduce selenium in an economical proprietary molasses-based nutrient mix. Results of this screening are presented in Figure D-1. Top performing microbes from this screening were selected for bioreactor testing. Previously collected selenium-reducing strains were selected based on their original source of isolation (high selenium containing mining and industrial process waters) and their ability to perform a reduction on selenium and other oxyanionic contaminants. Figure D-2 shows endpoint screening results of microbial strains tested for selenium reduction in synthetic laboratory water. Four laboratory isolates demonstrating the best selenium reduction in KUCC waters were selected for further testing—*Pseudomonas putida*, *P. pseudoalcaligenes*, *P. stutzeri*, *Cellulomonas flavis*. Isolates tested are naturally occurring, nonpathogenic facultative anaerobes. Isolates that reduced selenium by 95% in this screening were selected for a further study to determine relative selenium reduction rates.

The microorganisms reducing selenium in the above screenings were subsequently tested in KUCC waters containing -14.7 and -2.0 mg/L selenium for their ability to reduce selenium. Tests were conducted in 15-mL tubes under static conditions at ambient temperature for 7 days. Results of this screening are in Figures D-3 and D-4 and demonstrate the effect that site waters have on selenium reduction at this site. Different microbes were shown to have different levels of effectiveness in the two KUCC provided waters. This information was processed with additional information obtained from the testing described herein to formulate a microbial cocktail that would effectively remove selenium from both waters.



**Figure D-1.** Isolate screen for selenium reduction (spiked laboratory waters containing 50-mg/L selenium).



**Figure D-2.** Isolate screen for relative selenium reduction (spiked laboratory waters containing 50-mg/L selenium).

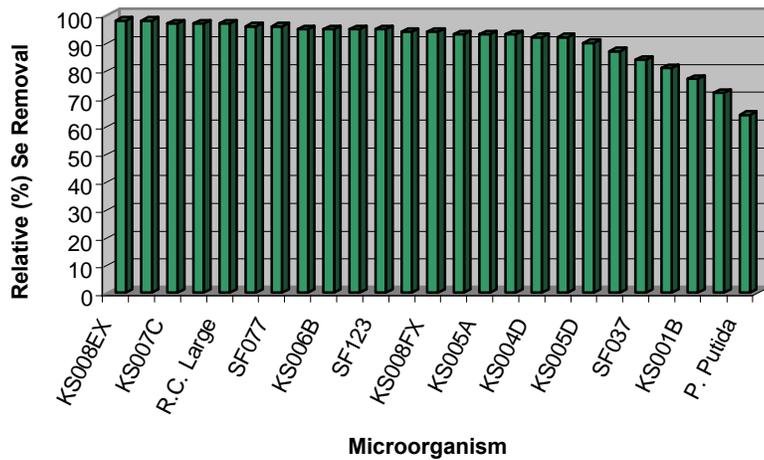


Figure D-3. Relative selenium reduction in KUCC water (-14.7 mg/L).

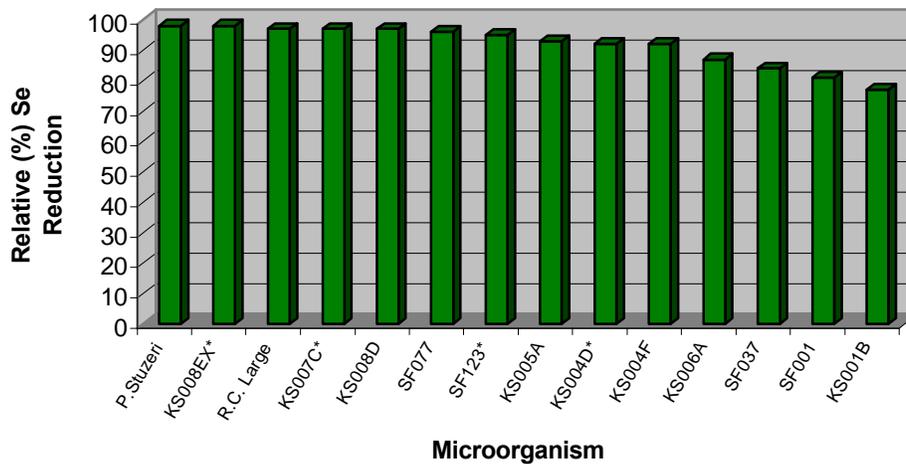


Figure D-4. Relative selenium reduction in KUCC water (-2 mg/L).

### D.5.3 Nutrient Screening

Microbes demonstrated to be effective in KUCC waters containing -2.0 mg/L selenium were grown for 24 hr in 50-mL volumes containing TSB at ambient temperature. Each sample was subsequently diluted or concentrated to a cell density of  $-2.0 \times 10^9$ /mL. The cells were washed with saline, resuspended in site waters with selected nutrients and incubated at ambient temperature for 6 days. Figures D-5, D-6 and D-7 show the effectiveness of selected nutrients for selenium reduction in site water. As can be seen in these figures, different nutrient mixes affect selenium reduction by different microbes differently. Molasses-based nutrient mixes were shown to be most effective for selenium reduction by site and other selected microbes using KUCC waters.

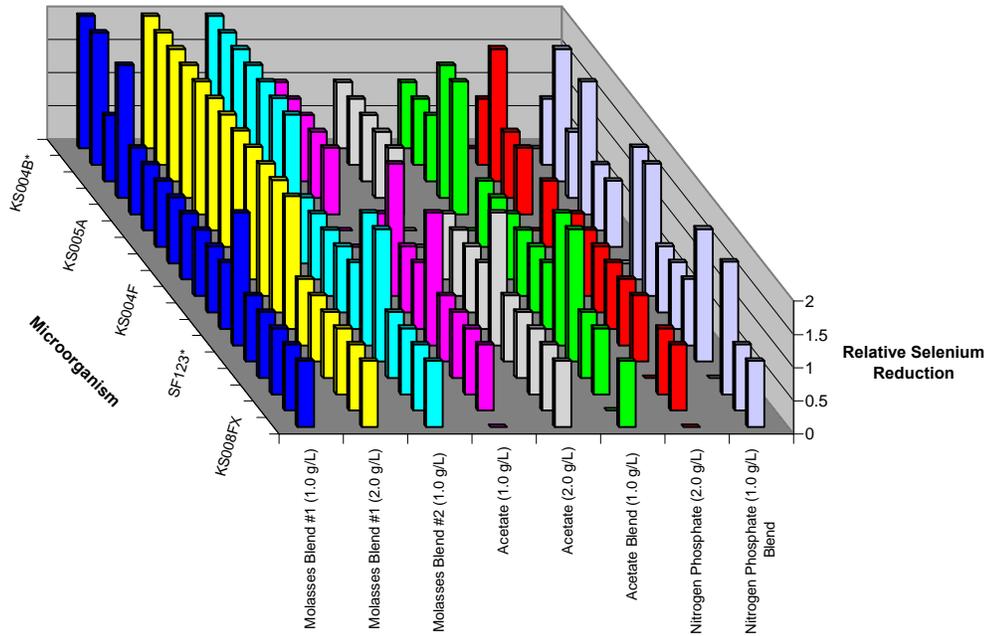


Figure D-5. Nutrient screening for selenium reduction in KUCC waters (-2 mg/L).

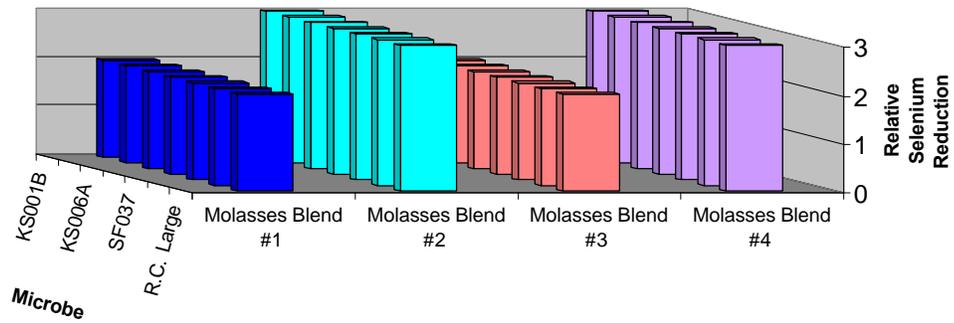
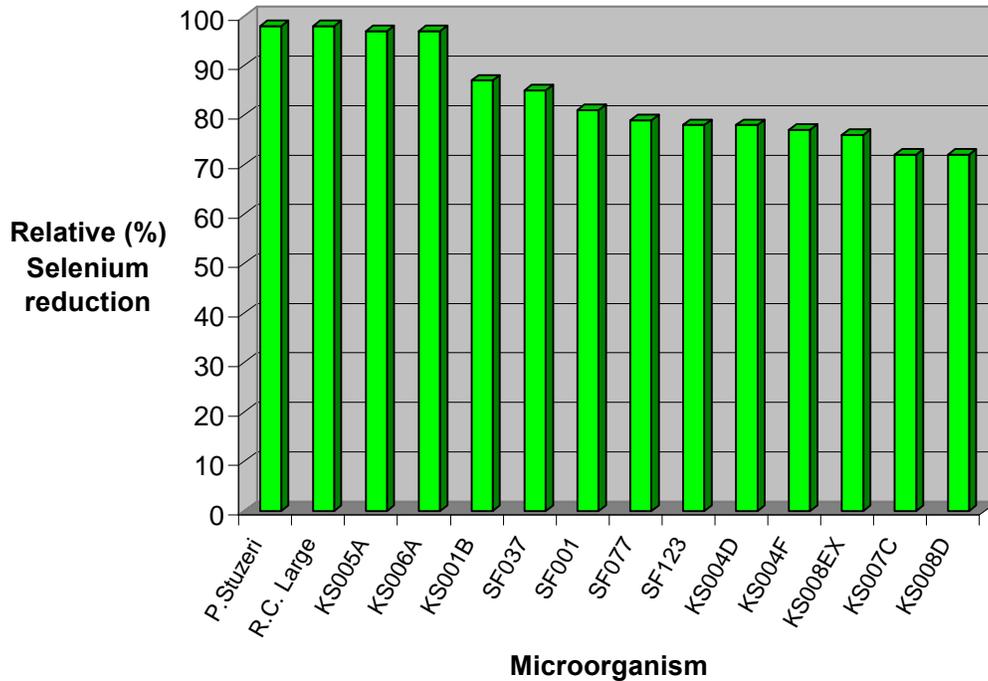


Figure D-6. Nutrient screening for selenium reduction in KUCC waters (-2 mg/L).



*Figure D-7. Selenium reduction with proprietary molasses-based nutrient blend. Test series used KUCC water at -2.0 mg/mL spiked to final selenium concentration of 25 mg/L.*

#### D.5.4 BIOLOG™ and MIDI Fatty Acid Analysis

BIOLOG™ plates were used to help determine metabolic profiles of potential key microbial cocktail microorganisms and potentially interfering nonselenium-reducing microbes. The results of these tests are presented here instead of with the rest of the microbial characterization results to represent the relative sequence in which the testing was conducted. Metabolic and site profiles were compared to develop a microbial cocktail that resembled the existing microbial population but that reduced selenium under site conditions using an economical nutrient source. Example BIOLOG™ screening plates are presented in Figure D-8.

MIDI analysis was conducted to develop profiles of important selenium reducers and nonselenium reducers to monitor biofilm development and performance throughout the pilot-scale tests. Examples of the monitored profiles are shown in Figure D-9. The MIDI profiles were also used to monitor microbial establishment and persistence in the bioreactors.

# Nutrient Utilization Profiles KS001EX

A1 water	A2 α-cyclodextrin	A3 dextrin	A4 glycogen	A5 tween 40 +	A6 tween 80 +	A7 N-acetyl-D-galactosamine	A8 N-acetyl-D-glucosamine	A9 adonitol	A10 L-arabinose	A11 D-arabitol	A12 cellobiose
B1 D-erythritol	B2 D-fructose +	B3 L-fucose	B4 D-galactose	B5 gentobiose	B6 α-D-glucose +	B7 D-inositol	B8 α-D-lactose	B9 lactulose	B10 maltose	B11 D-mannitol +	B12 D-mannose +
C1 D-melibiose	C2 β-methyl-D-glucoside	C3 D-psicose	C4 D-raffinose	C5 L-rhamnose	C6 D-sorbitol	C7 sucrose	C8 D-trehalose	C9 sulanose	C10 xylitol	C11 methyl pyruvate	C12 mono-methyl succinate +
D1 acetic acid +	D2 cis-aconitic acid +	D3 citric acid +	D4 formic acid +	D5 D-galactonic acid lactone	D6 D-galacturonic acid	D7 D-gluconic acid +	D8 D-glucosaminic acid	D9 D-glucuronic acid	D10 α-hydroxybutyric acid	D11 β-hydroxybutyric acid +	D12 γ-hydroxybutyric acid
E1 p-hydroxy-phenylacetic acid +	E2 itaconic acid	E3 α-keto butyric acid	E4 α-keto glutaric acid +	E5 α-keto valeric acid	E6 D,L-lactic acid +	E7 malonic acid	E8 propionic acid +	E9 quinic acid +	E10 D-saccharic acid	E11 sebacic acid	E12 succinic acid +
F1 bromo succinic acid +	F2 succinamic acid +	F3 glucuronamide	F4 alaninamide +	F5 D-alanine	F6 L-alanine +	F7 L-alanyl-glycine +	F8 L-asparagine +	F9 L-aspartic acid +	F10 L-glutamic acid +	F11 glycyl-L-aspartic acid +	F12 glycyl-L-glutamic acid +
G1 L-histidine +	G2 hydroxy-L-proline +	G3 L-leucine +	G4 L-ornithine +	G5 L-phenylalanine	G6 L-proline +	G7 L-pyroglutamic acid +	G8 D-serine	G9 L-serine +	G10 L-threonine	G11 D,L-carnitine +	G12 γ-amino butyric acid +
H1 urocanic acid +	H2 inosine +	H3 uridine	H4 thymidine +	H5 phenyl ethylamine +	H6 putrescine +	H7 2-amino ethanol	H8 2,3-butanediol +	H9 glycerol	H10 D,L-α-glycerol phosphate	H11 glucose-1-phosphate	H12 glucose-6-phosphate

# KS003AX

A1 water	A2 α-cyclodextrin	A3 dextrin	A4 glycogen	A5 tween 40 +	A6 tween 80 +	A7 N-acetyl-D-galactosamine	A8 N-acetyl-D-glucosamine	A9 adonitol	A10 L-arabinose	A11 D-arabitol	A12 cellobiose
B1 D-erythritol	B2 D-fructose +	B3 L-fucose	B4 D-galactose	B5 gentobiose	B6 α-D-glucose +	B7 D-inositol +	B8 α-D-lactose	B9 lactulose	B10 maltose	B11 D-mannitol +	B12 D-mannose +
C1 D-melibiose	C2 β-methyl-D-glucoside	C3 D-psicose +	C4 D-raffinose	C5 L-rhamnose	C6 D-sorbitol	C7 sucrose +	C8 D-trehalose +	C9 sulanose	C10 xylitol	C11 methyl pyruvate	C12 mono-methyl succinate
D1 acetic acid +	D2 cis-aconitic acid +	D3 citric acid +	D4 formic acid +	D5 D-galactonic acid lactone	D6 D-galacturonic acid	D7 D-gluconic acid +	D8 D-glucosaminic acid	D9 D-glucuronic acid	D10 α-hydroxybutyric acid	D11 β-hydroxybutyric acid +	D12 γ-hydroxybutyric acid
E1 p-hydroxy-phenylacetic acid +	E2 itaconic acid	E3 α-keto butyric acid	E4 α-keto glutaric acid +	E5 α-keto valeric acid	E6 D,L-lactic acid +	E7 malonic acid +	E8 propionic acid +	E9 quinic acid +	E10 D-saccharic acid	E11 sebacic acid +	E12 succinic acid +
F1 bromo succinic acid +	F2 succinamic acid	F3 glucuronamide	F4 alaninamide +	F5 D-alanine	F6 L-alanine +	F7 L-alanyl-glycine +	F8 L-asparagine +	F9 L-aspartic acid +	F10 L-glutamic acid +	F11 glycyl-L-aspartic acid +	F12 glycyl-L-glutamic acid +
G1 L-histidine +	G2 hydroxy-L-proline +	G3 L-leucine +	G4 L-ornithine +	G5 L-phenylalanine	G6 L-proline +	G7 L-pyroglutamic acid +	G8 D-serine	G9 L-serine +	G10 L-threonine	G11 D,L-carnitine +	G12 γ-amino butyric acid +
H1 urocanic acid	H2 inosine +	H3 uridine	H4 thymidine	H5 phenyl ethylamine	H6 putrescine +	H7 2-amino ethanol +	H8 2,3-butanediol	H9 glycerol	H10 D,L-α-glycerol phosphate	H11 glucose-1-phosphate	H12 glucose-6-phosphate

Figure D-8. *BIOLOG* metabolic screening plates.

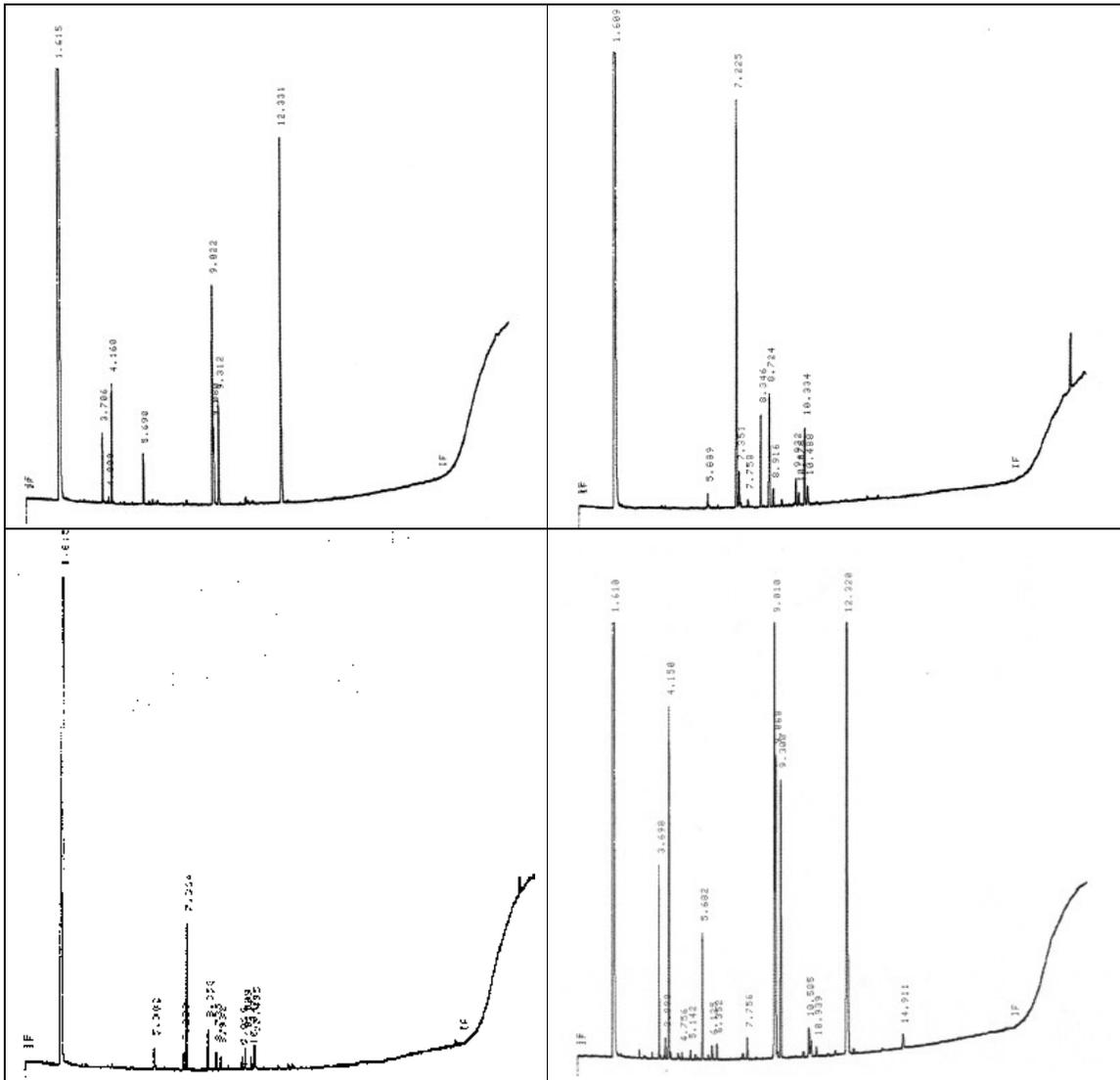


Figure D-9. MIDI profiles of bioreactor microbes.

### D.5.5 Microbial Support Materials

The 20 best selenium reducers from previous screening tests were screened for their ability to growth to a cell density of  $5 \times 10^9$ /mL on different reactor materials (see Table D-4). One milliliter of  $2 \times 10^9$ /mL cells was added to 9 mL of reactor materials and TSB under static conditions for 4 days. Celite is not listed because it was determined that it required pretreatment to obtain high microbial growth and the tests conducted were not designed to take this into account and were, therefore, biased in this respect.

Dissolved selenium sorption controls were run on the alginate, biosolids, and carbon. Using KUCC water at  $-2.0$  mg/L selenium, the alginate, as expected, sorbed considerably more than the carbon or biosolids, as shown below in Table D-5.

*Table D-4. Reactor matrix/biofilm testing.*

Microbe Name	Crushed Rock	Carbon	Alginate	Biosolids
SF046	+	+	+	+
KS008EX	+	+	+	+
KS008D	+	+	+	+
KS007C	+	+	+	+
SF077	+	+	+	+
SF047	+	+	+	+
SF024	+	+	+	+
SF050	+	+	+	+
SF123	+	+	+	+
KS008A	+	+	+	+
KS001C	+	+	+	+
KS009DX	+	+	+	+
KS006B	+	+	+	+
KS004C	+	+	+	+
KS008FX	+	+	+	+
SF057	+	+	+	+
KS005A	+	+	+	+
KS004B	+	+	+	+
KS004D	+	+	+	+
KS004F	+	+	+	+
KS005B	+	+	+	+

*Table D-5. Matrix sorption controls.*

Sample	[Se] mg/L	%
Kestler Spring Water	1790	N/A
Biosolids 1g/10mL	1660	7
Carbon 1g/10mL	1600	11
Alginate 1g/10mL	1182	34

### D.5.6 Laboratory Bioreactor/Bioprocess Tests

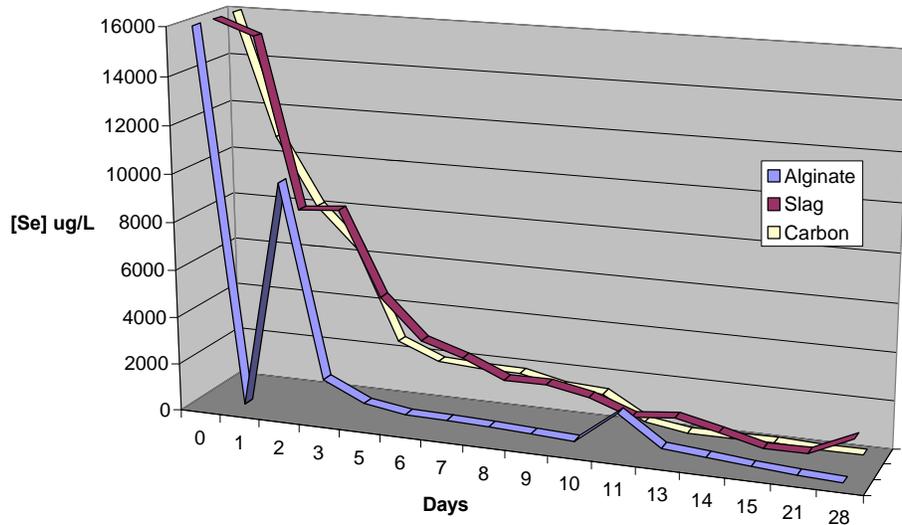
The first series of reactors tested used calcium alginate beads configured to evaluate microbial cocktail compositions in the following process ( $\text{SeO}_4^{2-}$ )  $\rightarrow$  ( $\text{Se}^0$ ) and slag and activated carbon sized to +8. Celite was not included in these tests. Slag and carbon reactors were treated to enhance biofilm establishment and then inoculated with the top performing microorganisms as shown in Figure D-10 (*Pseudomonas stutzeri*, RC-large, KS005A, KS006A, KS001B, SF037, and SF001). Reactors were inoculated in a manner to ensure establishment of this microbial cocktail as the predominant microorganisms in the carbon and biosolids reactors. With a 24-hr retention time and KUCC waters containing -14.7 mg/L selenium, the carbon and alginate reactors were removing -96% of the selenium. At day 10, the reactors were switched to KUCC waters containing -2.0 mg/L selenium and a 12-hr retention time. The microbes took a couple of days to adjust to the new water but then continued to remove 90% to 97% of the selenium for about 2 weeks. The slag reactor did not perform as well, removing up to 74% of the selenium in 12 hr. At this point, the first series of reactors was discontinued and a second series of reactors containing alginate beads, carbon, and a carbon-biosolids mixture was started using KUCC water containing -2.0 mg/L selenium.

The second reactor series was operated with a 12-hr retention time. Alginate beads were again used to evaluate different microbial cocktail compositions. As can be seen in Figure D-11, the microbial compositions tested in alginate did not perform as well as the first alginate test series and were discontinued at day 25. The microbial cocktails tested in series one were optimized for the KUCC water containing -2.0 mg/L selenium. In these second series tests, the carbon bioreactor again performed slightly better than the carbon biosolids reactors. However, both the carbon and carbon biosolids reactors were removing selenium to well below target levels; reaching low microgram to nondetectable levels. The low-level microgram selenium spikes are probably due to elemental selenium that was observed to migrate through the reactors in both test series.

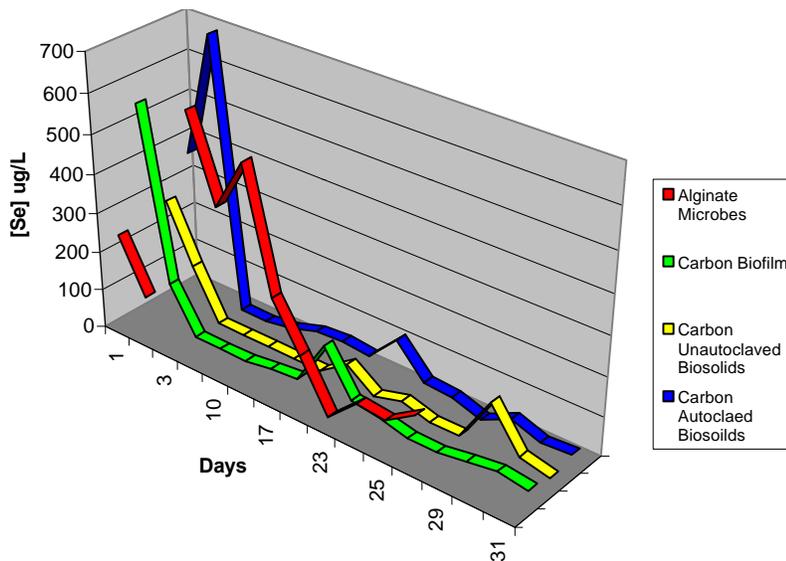
Control reactors consisted of alginate, slag, carbon, and carbon-biosolids without microorganisms. Slight initial dissolved selenium sorption was observed in all control columns except for the slag column. This sorption leveled out within a few days and control selenium levels were near reactor feed values. Reactor configurations tested are shown in Figure D-12 (initial laboratory reactors) and Figure D-13 (second series of laboratory reactors).

### D.5.7 Nutrient Feed Testing

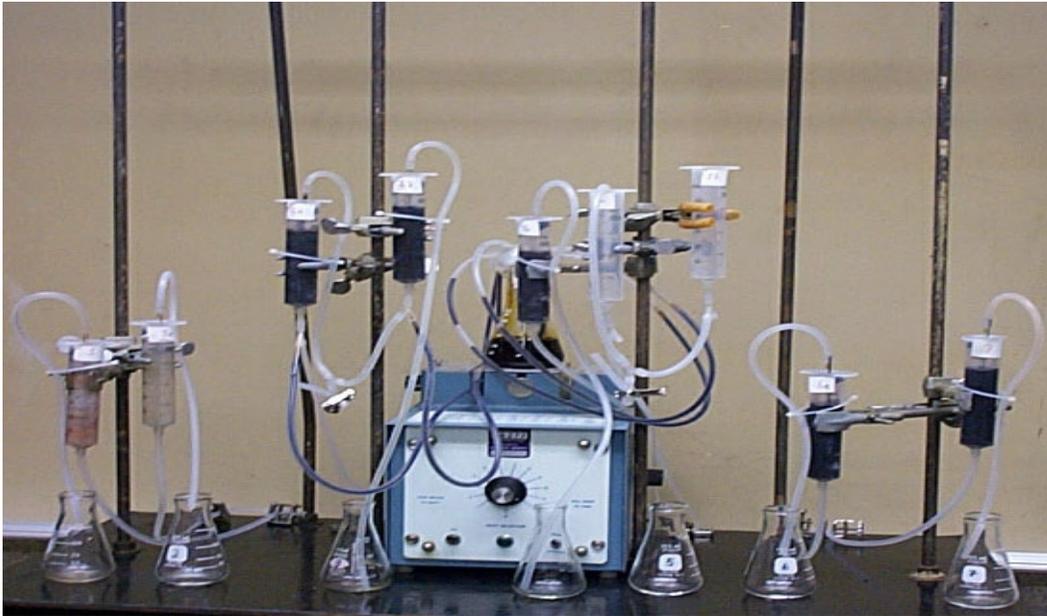
A pulse versus continuous reactor feed was tested in the bench-scale reactors. Both systems delivered the same amount of nutrient over the test period. Both feed delivery systems sufficiently supported selenium reduction in the reactors. However, in the continuous feed reactor, excess biomass formation was noted at the nutrient delivery site, resulting in poor fluid transfer through the reactor matrix. Based on these observations, a pulsed nutrient feed was implemented in the field reactors.



**Figure D-10. Laboratory Bioreactor Series 1.** The initial carbon, slag, and alginate columns that were used to measure selenium reduction using KUCC waters (~14.7 mg/L). Columns used a 24-hr retention time until day 10 when they were switched to a 12-hr retention time. Columns were run at ambient temperature under anaerobic conditions. The alginate column was used to measure relative effectiveness of various different selenium reducing microbes.



**Figure D-11. Laboratory Bioreactor Series 2.** The second series of carbon, carbon-biosolids, and alginate columns used to measure selenium reduction using KUCC waters (~2 mg/L). Columns used a 12-hr retention time and were run at ambient temperature under anaerobic conditions. The alginate column was used to measure relative effectiveness of various different selenium reducing microbes.



*Figure D-12. Laboratory bioreactor test configuration.*



*Figure D-13. Second series of BSeR™ laboratory reactors.*

## **APPENDIX E**

Enzymatic Selenium Reduction Laboratory Project

## **ENZYMATIC SELENIUM REDUCTION LABORATORY PROJECT**

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## **Executive Summary**

This project was focused on furthering the development of enzymatic selenium removal for demonstration in pilot-scale tests. Applied Biosciences has demonstrated, in bench scale tests, enzymatic selenium reduction from economical extracts of microbial cells. This document describes testing conducted toward development of an prototype enzymatic treatment system for demonstration at pilot scale. Enzymatic systems have the potential for greater kinetics, do not appear to be affected by contaminant levels that would kill live microbial cells and do not require nutrients. Furthermore, enzyme preparations have been demonstrated to reduce selenium in environments inhibitory to live microorganisms. Selenium was reduced in the presence of >100 mg/L cyanide, a cyanide concentration inhibitory to or toxic to all selenium reducing microbes tested to date.

Methods to economically prepare stable enzyme preparations and enzyme preparations from different microorganisms were investigated. Several immobilization polymers were evaluated to increase enzyme operational longevity. Of the polymers tested, Calcium alginate performed the best in regards to ease of handling, toxicity, cost, and performance. Problems with stability or possibly loss of an electron donor system were problematic throughout the testing, and are thought to be responsible for the variation in stability or performance observed between various tests. Even though enzymatic selenium reduction was demonstrated for periods ranging from 2-6 months, the stability or electron donor systems of the preparations tested was not sufficiently reproducible to warrant pilot scale tests at this time. In summary, although successful in furthering preparation of economical selenium reducing enzyme extracts, more research is required to enhance the stability and/or electron donor systems for pilot-scale tests.

# ENZYMATIC SELENIUM REDUCTION LABORATORY PROJECT

## Introduction

This document is a report for the Applied Biosciences Corp. (ABC) Enzymatic Selenium Reduction Laboratory Project. The Enzymatic Selenium Reduction Laboratory Project is a project within the Mine Waste Technology Program (MWTP). The MWTP is funded by the U.S. Environmental Protection Agency (EPA) and is jointly administered by the U.S. Department of Energy (DOE) and the EPA. This project tested selenium reducing enzyme preparations for stability and operational functionality. The project approach used an optimized mixture of naturally occurring bacterial enzymes from heterotrophic bacteria previously isolated from selenium-contaminated mining waters and soils, to reduce selenate and selenite to elemental selenium in mining wastewaters. Enzymatic selenium reduction was evaluated to make a decision for scale up and pilot testing. Project goals are to:

- Test enzyme extracts from microbes with the best demonstrated selenium reduction capabilities and from mixtures of these microbes to examine selenium-reduction kinetics
- Optimize selenium enzyme extraction/purification protocols
- Examine select, immobilization/encapsulation formulations to increase the stability and extend the functional time of the selenium-reducing enzyme(s) preparation
- Evaluate the immobilized/encapsulated enzyme preparation's durability, enzyme function (kinetics and stability).
- Determine initial bench-scale process operational parameters, estimated costs, and any pretreatment recommendations

## Background

Selenium is a common water contaminant throughout the world and represents a major environmental problem in the U.S., being a problem contaminant in at least nine western states. This contamination, originating from mining operations, mineral processing, abandoned mining sites, petroleum processing and agricultural run-off. Microbes have been identified and cultured with very high selenium tolerance and accelerated selenium reduction capabilities. These live microorganisms assembled in the Applied Biosciences' BSeR™ selenium bioprocess serve as a baseline for selenium reduction and removal. The high selenium tolerance and selenium reducing capabilities of these microorganisms were the basis for initial testing of the enzymatic selenium reduction process.

Enzyme technologies are revolutionizing all biotechnology disciplines. Enzyme technologies are commonplace in the pharmaceutical industry, medical and environmental diagnostics, and are found in household products such as laundry detergent and degreasing products. In the area of pollution control, various enzyme technologies have been demonstrated. In water treatment, enzymatic contaminant removal is considered an emerging technology, potentially applicable to waste and drinking water treatment. For removal of selenium from waters, Applied Biosciences has demonstrated that cell free extracts have been able to reduce and remove selenium from various mining waters at the bench scale.

Proprietary enzyme technologies for contaminant removal have been demonstrated, by Applied Biosciences at bench scale. The prototype enzymatic selenium reduction system functioned equally well in both synthetic and actual mining wastewaters. The potential of enzymatic selenium reduction is based on proprietary enzyme extraction/purification methods combined with unique immobilization/encapsulation techniques that keep the selenium reducing enzyme(s) in a functional arrangement within an immobilization matrix. Enzyme extraction methods and immobilization matrices require improvement to make a pilot-scale evaluation of enzymatic selenium reduction system practical.

## **Materials and Methods**

### General

Enzyme preparations were produced from selenium-reducing microorganisms by lysing bacterial cells in a bead-mill type cell homogenizer, extracting/purifying specific cellular fractions and subsequently immobilizing the preparation in several different immobilization/encapsulation matrices. Preparations immobilized in a standard calcium alginate polymer were formed into beads for base line tests and comparisons.

### Microbes

Microbes were screened to select microorganisms with the greatest potential for selenium reduction and would therefore be good candidates for enzyme sources. Microbial strains were collected from sites with a long history of selenium contamination. Select *Pseudomonas* and *Alcaligenes sp.* were used for the selenium-reducing immobilized enzyme preparations. These strains have unique selenium-reducing characteristics and have been utilized in selenium removal systems at bench, pilot, and full scale in the **BSeR™** process.

### Controls

Comparative tests with biofilms, immobilized live cells, and immobilized enzymes used controls consisting of support materials without biofilms and immobilized heat-inactivated cells or enzymes. Immobilized live cells and enzyme preparations used the same starting live microbial cell concentrations.

### Endpoint selenium reduction assays

Endpoint selenium reduction assays were utilized as a screening tool to assess selected microbial strains, enzyme preparations, and immobilization supports for selenium reduction capabilities. The test water used for the screening series consisted of collected Kennecott Utah Copper Corporation (KUCC) water, unspiked, and spiked to a concentration of 50 or 100 mg/L Se. For the microbial screening, log-phase cultures were prepared in Trypticase Soy Broth (TSB). Cultures were washed and re-suspended in sterile saline. 15-ml culture tubes containing test water were inoculated with log phase suspended cultures, at a concentration of  $2 \times 10^8$  cells per ml. Sterile saline served as the abiotic control.

The tubes were incubated at 22° C for 24 hours, and periodically assayed for selenium reduction. Relative selenium reduction was determined by the formation of a red amorphous selenium precipitate. For enzyme extract testing, cell free extracts were immobilized in calcium alginate beads, or other polymers listed in Table 2. Beads containing one ml of enzyme extract

were tested as described above. Control tubes containing blank beads were prepared using sterile saline. All testing was done with actual site waters.

#### Cell-free Extract Preparation

In general, cell free extracts were prepared using a bead mill containing 0.2 mm beads in disruption buffer (HEPES buffered saline, pH 7.5). Non-disrupted cells and cell debris were removed using low speed centrifugation (1000xg, 20 min). Controls on all enzyme test materials included two tests: (1) direct microscopic examination of the enzyme preparation for live cells and (2) plating 1.0 ml of enzyme preparation on trypticase soy agar (TSA). Initially, an additional control sample was plated, 1.0 ml of a 10-fold concentration of the enzyme preparation, with no observable live cells on TSA. Data from the enzyme preparations were not used if any live microorganisms were present.

#### Immobilization Testing

Various immobilization schemes were screened, tested, and compared in the laboratory, including: Alginate acid, high viscosity (Sigma #A7003); Alginate acid, low viscosity (Sigma #A2158); Bulk Sodium Alginate (WEGO Chemical Corp.); Agarose (BBL #11849); Carrageen Type I (Sigma C1013; Carrageen, Type II (Sigma C1138); Polyacrylamide; polysulfone; nitrocellulose membrane (SpectraPor #132680); and granular activated carbon.

For testing a prototype enzyme system, alginate acid as calcium alginate was selected as the best initial encapsulation polymer. Low viscosity calcium alginate was selected because it stabilized the enzyme preparation more than other matrix materials, ease of handling during matrix preparation, negligible toxicity, cost, and observed stability in KUCC test water.

Strains demonstrating the highest selenium reduction capabilities from the microbial screening were selected for preparation of enzyme extracts and additional screening. Extracts immobilized in calcium alginate were tested individually, and then as a mixture using the described endpoint selenium reduction assay. A control using empty immobilized matrix material was also tested. Heat inactivated (denatured) extracts (80°C for 15 minutes), were utilized as a negative (dead enzyme) control.

## **Results**

#### Microbial Screening

Multiple microbial isolates, including the microbes used in the **BSeR**<sup>TM</sup> process, were tested for their ability to reduce selenium in spiked (to 50 mg/L Se) and un-spiked synthetic and actual KUCC waters, Figure 1. Strains were selected based on their original source of isolation (high selenium containing mining and industrial process waters) and their ability to perform a reduction on other oxyanionic contaminants such as selenate. All isolates tested are naturally occurring, non-pathogenic facultative anaerobes. Some of the isolates tested for selenium reduction are shown in Table 1.

Microbe	Microbial Se Red. In synthetic water	Microbial Se Red. in KUCC test water	Enzymatic Se Red.(cell-free prep)
C1-1a	+++	+++	+++
(53)9-26	+++	+++	++
C1-1b	+++	+++	+++
A-27	+++	+++	++
P. stutzeri 1	+++	++	+
R.C. Large	+++	++	+
SF123	+++	+	ND
SF077	+++	+++	0
KS005a	+++	++	0
KS004d	+++	+++	*
C. flavis	+++	+	ND
P. putida	+++	+	*
KS006A	+++	++	0

**Table 1. Microbial Screening.** Selenium reducing strains were initially screened for selenium reduction in synthetic waters, and then actual KUCC test water. Cell free preps for the strains that scored ++ or higher were prepared and evaluated. The top 4 cell free preps (scoring +++) were selected for use in additional evaluations.

#### Enzyme preparation testing

Top performing microbial cultures (C1-1a, (53)9-26, C1-1b, and A-27) from the microbial screening were utilized as a source material for enzyme preparation, and as cultures for the live microbial biofilm reactors. Cell-free extracts were screened in an immobilized form in calcium alginate beads. Controls included denatured enzyme preparations, immobilized live microbial cells and immobilization polymers. The live microbial controls contained the same number of cells used to prepare the enzyme extracts so that a direct comparison could be made. Results of the screening are detailed in Figures 2 and 3. The tests were evaluated for and screened for the formation of elemental selenium over a 2 month period. With the optimized preparations, the enzymatic preparations exceeded the initial selenium reducing activity of the live cell beads.

However, a loss in stability was observed in the cell-free preparations that was not observed in the living system. This loss in stability contributed to variation between cell free preps of the same origin and unpredictable operational longevity of the system, Figure 4.

#### Immobilization Support Testing

Granular activated carbon support material performed the best for a live microbial system, and was utilized for reactor testing. Based on the testing and evaluation of various other supports, bulk sodium alginate was selected as the best immobilization material for the enzyme system.

Sodium alginate was cross-linked with  $\text{Ca}^{3+}$  to form the calcium alginate matrix. Calcium alginate was selected as an encapsulation polymer for due to function of the immobilized

reduction system, ease of handling during matrix preparation, low toxicity, cost, and observed stability. As a microbial support, the calcium alginate beads have been demonstrated to remain intact for periods greater than two years, without loss of microbial function or support structural breakdown. The support materials are ranked in Table 2.

Table 2. Immobilization Materials

Support Material	Poor 1 < ----- > 5 Good			Relative Performance (Cell Free)	Relative Performance (Microbes)	Overall Rating
	Ease of Handling	Toxicity	Cost			
Alginate Acid, low viscosity	4	Low	High	4	4	3
Alginate Acid, high viscosity	2	Low	High	4	4	3
Bulk Sodium Alginate	4	Low	Low	4	4	4
Agarose	4	Low	Med	2	2	2
Carrageen	3	Low	Low	1	1	2
Polyacrylamide	1	High*	Med	1	1	1
Nitrocellulose membrane	1	Low	High	2	2	2
Granular Activated Carbon*	5	Low	Low	N/D	5	5*

\* Tested with microbial system only

#### Electron Donor System Testing

Various electron donor systems tested in the laboratory include cellular components, nutrient components, and electron-carrying dyes. An electro-bioreactor test system was designed to provide a constant supply of electrons to the matrix as an attempt to increase the operational longevity of the system. Test material was prepared for the system by incorporating the electron-carrying dyes (Azur A and Bromophenol Blue) into an alginate matrix. A bead preparation without an electron-carrying dye (enzyme extract only) served as a control. The supplied DC current, with and without the electron carrying dyes did not appear to have an appreciable effect on enhancement of the longevity of selenium reduction (data not shown). None of the other tested electron donor systems, including the nutrient components (acetate, H<sub>2</sub> and molasses) increased the operational longevity of the enzymatic matrix.

#### Reactor Testing

Bench scale testing has demonstrated the proof of concept for use of enzyme technologies for water treatment. Bench-scale up-flow columns were set up as demonstrated in Figure 5. Selenium removal from the mining process solutions has been tested using a consortium of

selenium-reducing bacteria, both as live microbes and as an immobilized enzyme preparation. The data indicates that the selenium concentration was reduced to approximately the same levels in both the live immobilized microbe column and the immobilized enzyme column. The selenium concentrations were lowered from 23.1 mg/L in the feed, to <0.10 mg/L in the effluent in 9hr, Figure 6. For this testing a 9-hour retention time was used.

A second test series was conducted using immobilized enzymes to determine if cyanide and selenium could be removed simultaneously from the process solution. Enzyme preparations used in proprietary cyanide-oxidizing enzyme preparations and the selenium-reducing enzyme preparations were immobilized separately and combined in a column for testing. Test results, presented below show that the cyanide level decreased from 102 to <1 mg/L and the selenium concentration decreased from 31.1 to 1.6 mg/L, Figure 7. Simultaneous removal using live microbes would not be possible because the cyanide level of ~100 mg/L is toxic for the live selenium-reducing bacteria. An 18-hour retention time was used to allow contaminant diffusion into the alginate beads.

Enzymatic selenium reduction was compared with an enhanced encapsulated live microbial biofilm preparation, Figure 8. Stabilized microbial enzyme preparations were able to remove selenium to or below 0.01 mg/L in a single-pass reactor from a feed solution containing 0.62-mg/L selenium for over four months. In comparison, the enhanced immobilized biofilm also reduced selenium in these tests to below 0.01 mg/L for over nine months.

## Discussion

Applied Biosciences has demonstrated, at bench scale, a proof-of-concept proprietary enzyme technology for selenium reduction and removal. The prototype functioned equally well in both synthetic and various actual wastewaters for limited times. This metal reducing technology is based on proprietary enzyme extraction/purification methods combined with unique immobilization/encapsulation techniques that keep the selenium reducing enzyme(s) in a functional arrangement within an immobilized/encapsulated matrix.

Advantages of cell-free systems over live systems include (1) the potential for greatly increased kinetics, (2) nutrients are not required, and (3) the effects of toxic process solutions can be eliminated. Cell-free bioreactors can be engineered to be resistant to microbial overgrowth and degradation. To construct an enzyme bioreactor, one needs readily available sources of the stable enzymes. Although several enzymes of microbial origin have been isolated and characterized, some are membrane-bound and difficult to purify and retain activity *in vitro*. Pure enzymatic metal reduction systems are currently cost prohibitive to treat large water volumes

Because enzymes are biological catalysts, they promote the rate of reactions and are not themselves consumed in the reactions; they may be used repeatedly for as long as they remain active. However, in most industrial, analytical, and clinical enzymatic processes, enzymes are mixed in a solution with substrates and cannot be economically recovered after the exhaustion of the substrates. This single use approach is obviously quite wasteful when the cost of enzymes is considered. Thus, there is an incentive to use enzymes in an

immobilized form so that they may be retained in a bioreactor to catalyze a feed stream. The use of immobilized enzymes would make it economically feasible to operate an enzymatic process in a continuous mode.

Numerous methods exist for microbe and enzyme immobilization. These include biofilms, matrix entrapment, micro-encapsulation, adsorption, and covalent binding. Many entrapment methods are used today, and all are based on the physical occlusion of live microbes and/or enzyme molecules within a "caged" gel structure such that the diffusion of active components to the surrounding medium is severely limited, if not rendered totally impossible. What creates the "wires" of the cage is the cross-linking of polymers. A highly cross-linked gel has a fine "wire mesh" structure and can more effectively hold smaller enzymes in its cages. The degree of cross-linking depends on the condition at which polymerization is carried out. Ideally the network of cross-linking should be coarse enough so that the passage of substrate and product molecules in and out of a gel bead is as unhindered as possible.

With any oxidation/reduction reaction, an electron donor or acceptor must be present to complete the desired contaminant transformation. In a living microbial system, the electrons are provided as carbon substrates are oxidized. One can anticipate both live microbial and enzymatic systems to function only as long as a suitable electron donor/acceptor system is available. Many materials can function at electron donors both for live microbial cells and for immobilized enzyme systems. These materials include many metal ions, microbial cellular components, nutrient components, dyes, and direct electric current.

#### Scale Up Recommendations

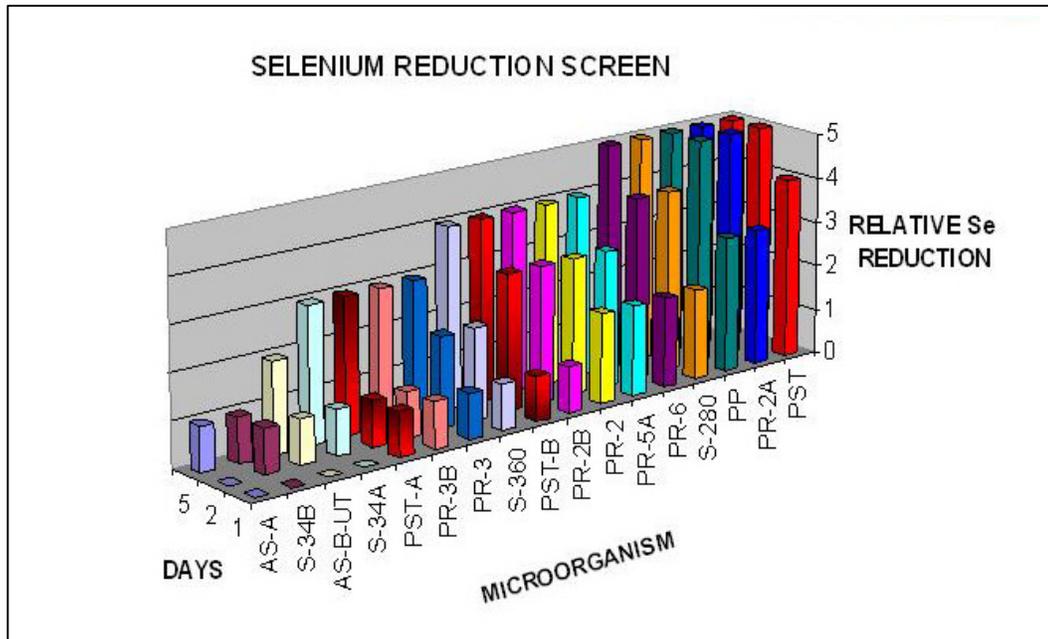
Due to the instability or lack of an appropriate electron donor system, of the enzymatic reactor matrix, enzymatic selenium removal cannot be recommended as an economical process at this time, nor is it ready to be recommended for pilot-scale testing. The current limitation to the deployment of an enzymatic selenium reduction system lies in the cost and/or ability to produce a stable enzymatic reactor matrix.

Purified enzyme preparations of plant origin, are currently commercially available. However, these plant-based preparations are much too expensive to be applied to water treatment. The use microbial enzyme preparations are expected to eventually reduce these costs. However, more work is needed to gain a better understanding of what is occurring in the immobilization of the enzymes and the linking of electron donors with in any immobilization technique used. If the enzyme-matrix can be demonstrated to be stable for 6 to 9 months, the process could possibly be considered as an economical treatment alternative. However, with the current operational longevity at 3 weeks to just several months, treatment costs become prohibitive. The enzymatic system still has the potential to operate at higher kinetics and outperform live microbial systems in many ways. Enzymatic technologies are still in the prototype development stages, but are viewed by many to have the potential to revolutionize drinking water and wastewater treatment. .

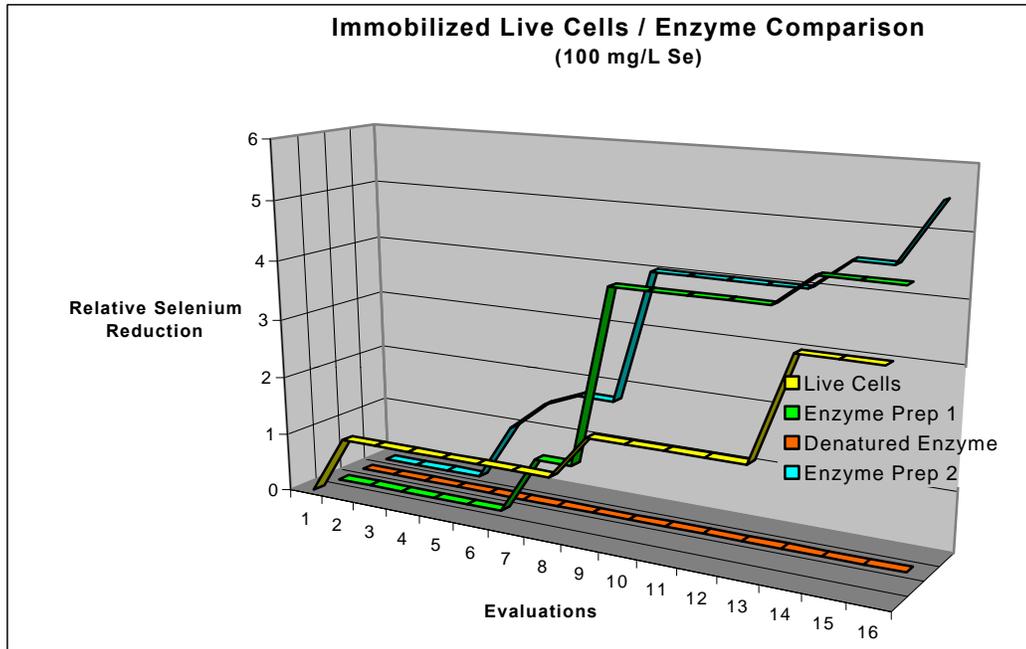
#### Conclusions

Based on this laboratory study, the following conclusions can be made:

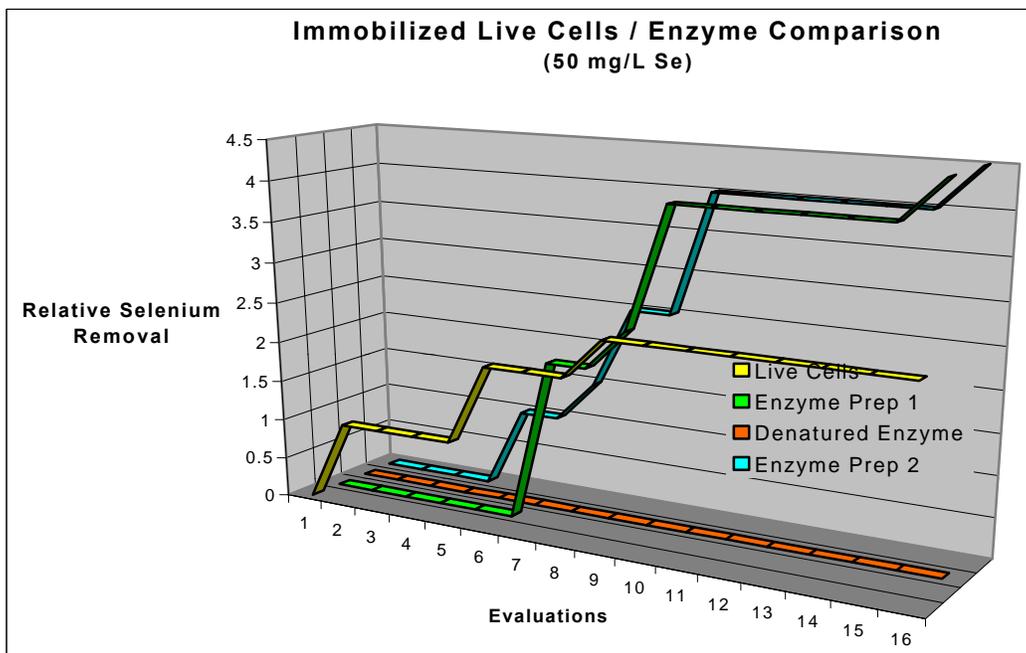
- Microorganisms are an alternative source for inorganic contaminant reducing enzymes
- Selenium reduction in the presence of cyanide is possible using select cell free preparations
- As an encapsulation polymer, calcium alginate performed the best in regards to ease of handling, toxicity, cost, and performance.
- Research to further develop the electron donor system and enhance the operational longevity of the system is needed to complete prototype development



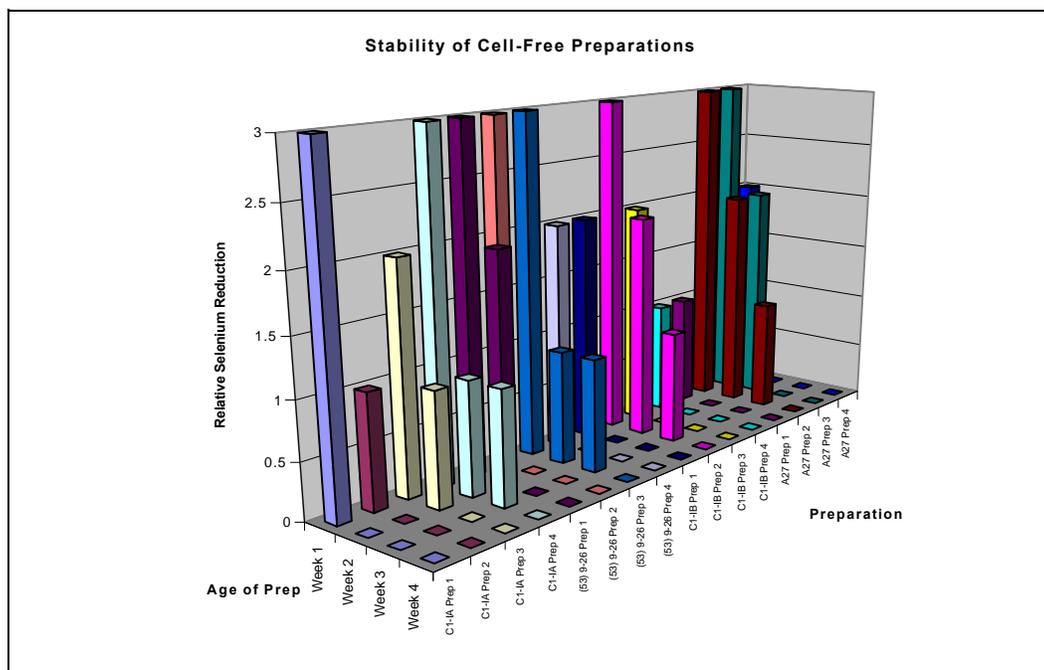
**Figure 1.** Multiple microbial isolates were tested for their ability to reduce selenium in synthetic and actual mining waters.



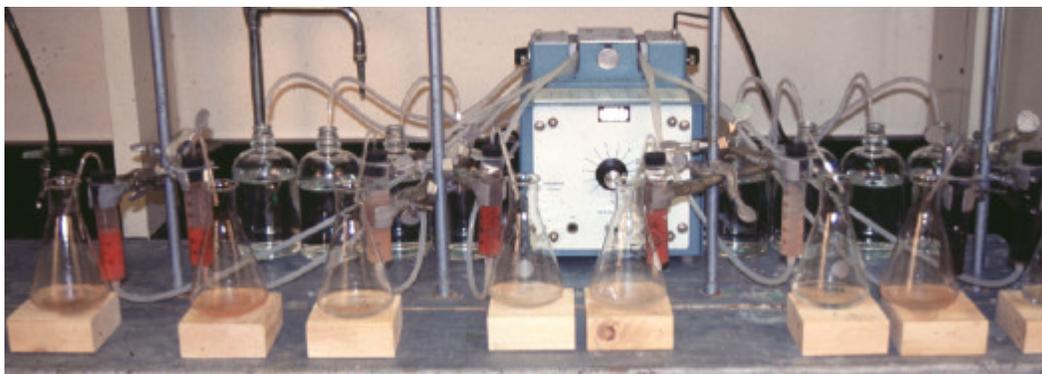
**Figures 2 and 3.** Enhanced selenium removal was observed in cell free preparations when compared to a live microbial system. Testing used actual KUCC mining water spiked to 50 and 100 mg/L Se.



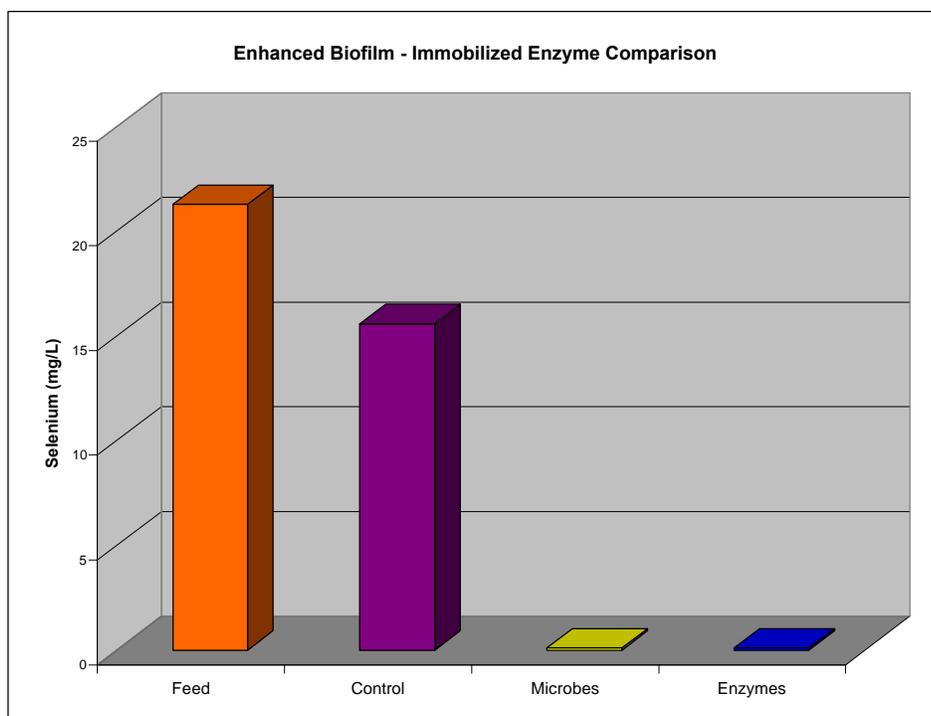
**Figure 3.** (See caption in Figure 2).



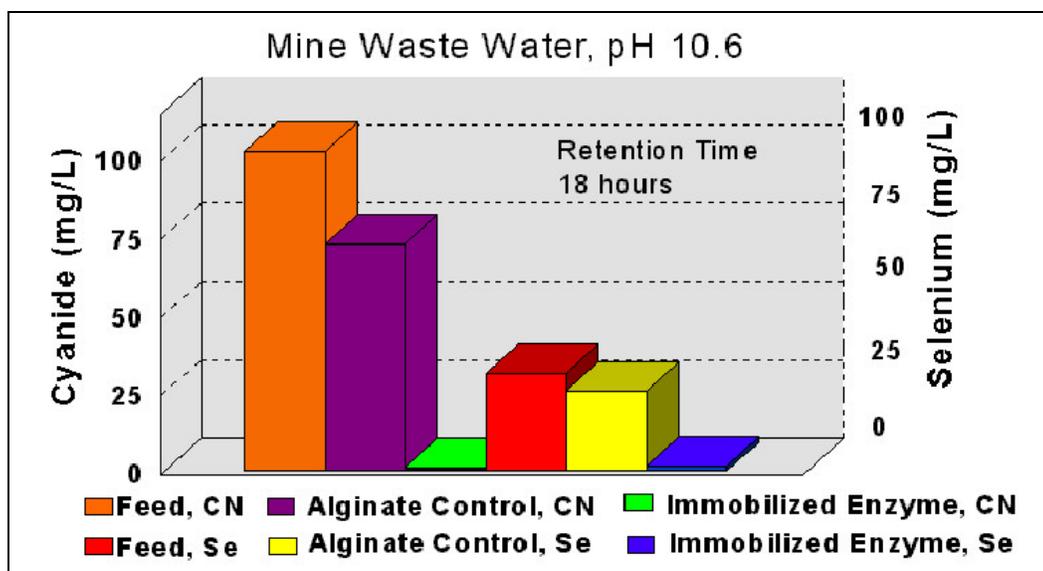
**Figure 4.** Multiple preparations were tested for stability over time. Preps were allocated and placed into selenium containing water at 1 week intervals. By the fourth week, all preps had lost selenium reducing capabilities.



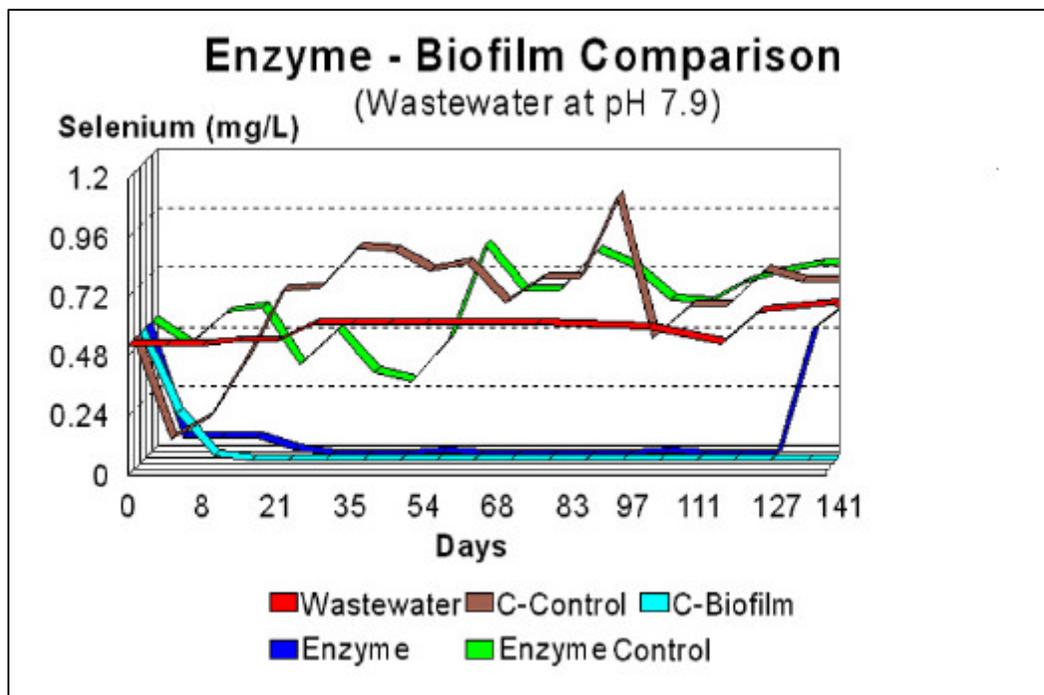
**Figure 5.** Bench scale reactor test apparatus.



**Figure 6.** Enhanced Biofilm and Immobilized Enzyme Comparison



**Figure 7.** Cell free systems can provide for simultaneous contaminant removal in environments that are inhibitory to live microbial systems. Cyanide at this concentration is toxic to all selenium reducing microbes tested. An 18 hour retention time was used to allow contaminant diffusion into the alginate beads.



**Figure 8.** Proof-of concept reactor testing compares enzymatic selenium reduction to live microbial systems.