FINAL REPORT—BERKELEY PIT INNOVATIVE TECHNOLOGIES PROJECT, PHASE II: GEO2 LIMITED DEMONSTRATION

MINE WASTE TECHNOLOGY PROGRAM ACTIVITY IV, PROJECT 7

#### **Prepared by:**

Montana Tech of The University of Montana 1300 West Park Street Butte, Montana 59701

and

MSE Technology Applications 200 Technology Way P.O. Box 4078 Butte, Montana 59702 IAG ID No. DW89938513-01-0

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### **REVIEWS AND APPROVALS:**

Prepared by:

Project Engineer

Reviewed by:

Project Manager

Program Manager

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# Berkeley Pit Innovative Technologies Project—Phase II Geo2 Limited Report on the Demonstration of the Green Precipitate (GP) Process

**Demonstration performed by:** 

Geo2 Limited

#### Written by:

R. M. Taylor, R. W. Arthur, and C. J. Restarick. Geo2 Limited Level 2, 155 Queen Street, Melbourne, VIC 3000 Australia

#### **Principal Investigators:**

Dr. Reginald M. Taylor Mr. Ronald W. Arthur

#### **Project Coordination:**

Dr. Gottfried Lichti Mr. Ian D. Ennis

#### **Prepared by:**

MSE Technology Applications, Inc. 200 Technology Way P.O. Box 4078 Butte Montana 59702

and

Montana Tech of the University of Montana 1300 W. Park Street

#### Butte, Montana 59701 Foreword

Today industries are developing and modifying technologies to more efficiently produce their products. The waste generated by these industries, if improperly dealt with, can threaten public health and degrade the environment. The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the 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 the EPA to perform research to define, measure the impacts, and search for solutions to environmental problems.

The National Risk Management Research Laboratory (NRMRL) of the EPA is responsible for planning, implementing, and managing research, development, and demonstration programs to provide an authoritative, defensible engineering basis. This supports the policies, programs, and regulations of the EPA with respect to drinking water, wastewater, pesticides, toxic substances, solid and hazardous wastes, and Superfund-related activities. The Federal Energy Technology Center (FETC) of the U.S. Department of Energy (DOE) has responsibilities similar to the NRMRL in that FETC is one of several DOE centers responsible for planning, implementing, and managing research and development programs. In June 1991, an Interagency Agreement (IAG) was signed between EPA and DOE that made funds available to support the Western Environmental Technology Office's (WETO) operating contractor, MSE Technology Applications, Inc. (MSE), and Montana Tech of The University of Montana (Montana Tech) for the development of the Mine Waste Technology Program (MWTP). This publication is one of the products of the research conducted by the MWTP through these two Federal organizations and provides a vital communication link between the researcher and the user community.

The objective of this demonstration was to remediate Berkeley Pit water to the extent that it could be safely used for agricultural applications, to demonstrate the suitability of the technology in allowing continuous online treatment that could be scaled up to match present inflows into the Berkeley Pit, and to evaluate the possible recovery of the economic minerals in the compacted precipitate.

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#### **Executive Summary**

The primary objective of this demonstration was the treatment of Berkeley Pit water to the extent that it could be safely used for agricultural application. Secondary objectives were to:

- demonstrate the adaptability of the technology to a continuous treatment that is scalable to match or exceed present inflow rates into the Berkeley Pit and is cost effective;
- evaluate the possible recovery of commercial metals in the compacted precipitate; and
- investigate parameters such as the settling rate of the precipitate and its physical properties that may enhance or reduce the economic attractiveness of the process.

In August 1996, it was demonstrated that dissolved metals in the Berkeley Pit water could, to a large extent, be made to react with each other under suitable conditions to form a precipitate similar to this family of minerals. In the demonstration, the average concentration of heavy and toxic metals was significantly reduced and the average pH of the treated water was less than 8, making it suitable for agricultural applications.

The initial experiments were carried out on Berkeley Pit water that had been taken from the 200-foot level of the Berkeley Pit and stored under nitrogen at MSE for one year. Although this water had a slightly different composition from that of the fresh Pit water taken from the same level, especially with respect to iron concentration, the parameters chosen to apply to the remediation of fresh Pit water samples in the official trials proved satisfactory. Two of the official samples were fresh Pit water; the third is a further sample of the stored water.

The 1997 demonstration was conducted in a 40-liter (L) cylinder in which the water to be treated could be efficiently stirred at different rates under a blanket of nitrogen, and the dark green precipitate (GP) in suspension could be removed through a tap at the bottom of the cylinder for filtration. The addition of flocculating agents would not add to the efficiency of precipitation or dewatering; therefore, they were not added to the process.

The results of analyses of the treated water from the three batch experiments yielded arsenic, chromium, copper, iron, and zinc concentrations below the detection limits. Cadmium for two samples was 0.006 and 0.016 parts per million (ppm) respectively, while in the third sample it was below the detection limit. Similarly, aluminum was measured at 0.3 ppm in two of the samples, just above its detection limit, whereas in the third sample it was below the detection limit. The manganese concentration in the three samples was determined at 3.3, 4.94 and 4.3 ppm respectively, slightly below the amended target level of 5 ppm. It is to be noted in the treatment of a much larger volume (500 L) in a pilot plant coiled pipe reactor that was a demonstration given as an adjunct to this work, the manganese level was generally below 1 ppm. Sulfate was slightly above the target level of 2 500 mm being measured at 2 080, 2 670 and 2 100 mm respectively.

2,500 ppm being measured at 3,080, 2,670 and 3,100 ppm respectively.

The flocculent nature of the layered lattice reaction products and the cohesive, rather than adhesive, nature demonstrated in the settling tests added to the ease of the separation of the residue from the treated supernatant water. Moreover, dewatering produces a compact filter cake that was shown to be suitable for the economic recovery of mineral values therein by direct leaching with an ammoniacal ammonium carbonate solution. In a series (5) of extractions on the three precipitates, an average of

91.7% of the total copper and 86.5% of the total zinc were successfully leached from the precipitate. Although the precipitate showed this cohesiveness, there was no cementation effect when the precipitate was allowed to sit in a closed circuit, 1-inch-internal-diameter tube for various periods up to one week. The precipitate was brought back into a fluid suspension as soon as an in-line pump was activated.

The direct use of deep Pit water without an initial oxidative stage represents an advantage by allowing the water to be drawn directly from the subsurface layers and treated directly in a pipe reactor whose length can be varied to increase the reaction time, if necessary. The presence of supersaturated calcium sulfate (gypsum) in the treated water presents no disadvantage for agricultural use. In alkaline soils, gypsum is commonly added to improve the soil structure.

Since no organic reagents are involved in the process, there is no fear of increased mutagenicity following chlorination if the treated water approaches drinking standards.

## Contents

|    |   | U               |  |  |  |  |  |  |  |  |
|----|---|-----------------|--|--|--|--|--|--|--|--|
| Ex | ewordeword  | ii<br>iii<br>vi |  |  |  |  |  |  |  |  |
| Та | Tables vi   |                 |  |  |  |  |  |  |  |  |
| Ac | knowledgments   | vii             |  |  |  |  |  |  |  |  |
| 1. | INTRODUCTION  | 1               |  |  |  |  |  |  |  |  |
|    | 1.1 Demonstration Personnel   |                 |  |  |  |  |  |  |  |  |
| 2. | PROCESS DESCRIPTION   | 3               |  |  |  |  |  |  |  |  |
| ∠. | 2.1 General Overview  |                 |  |  |  |  |  |  |  |  |
|    | 2.1       General Overview         2.2       Process Flow Sheet   | -               |  |  |  |  |  |  |  |  |
|    |   |                 |  |  |  |  |  |  |  |  |
|    | ······································  |                 |  |  |  |  |  |  |  |  |
|    | 2.4 Experimental Design   | 4               |  |  |  |  |  |  |  |  |
| 3. | EXPERIMENT DETAILS FOR GP PROCESS   |                 |  |  |  |  |  |  |  |  |
|    | 3.1 Experiment Geo2/1–September 26, 1997  | 5               |  |  |  |  |  |  |  |  |
|    | 3.1.1 Results   |                 |  |  |  |  |  |  |  |  |
|    | 3.2 Experiment Geo2/2–September 29, 1997  | 5               |  |  |  |  |  |  |  |  |
|    | 3.2         Experiment Geo2/2         September 29, 1997           3.3         Experiment Geo2/3–September 30, 1997 |                 |  |  |  |  |  |  |  |  |
|    | 3.4 Cementation Test of the GP in Pipe  |                 |  |  |  |  |  |  |  |  |
|    | 3.5 Settling Kinetics of GP Process   |                 |  |  |  |  |  |  |  |  |
|    | 3.6 Recovery of Copper and Zinc from the Precipitate  |                 |  |  |  |  |  |  |  |  |
|    | 3.6.1 Aim of Test Demonstration   |                 |  |  |  |  |  |  |  |  |
|    | 3.6.2 Background  |                 |  |  |  |  |  |  |  |  |
|    | 3.6.3 Procedure   |                 |  |  |  |  |  |  |  |  |
|    | 3.6.4 Results   |                 |  |  |  |  |  |  |  |  |
|    | 5.0.4 Results   | 0               |  |  |  |  |  |  |  |  |
| 4. | SUMMARY AND CONCLUSIONS   | 14              |  |  |  |  |  |  |  |  |
|    | 4.1 Economic Evaluation   | 14              |  |  |  |  |  |  |  |  |
|    | 4.2 Independent Technology Review   | 14              |  |  |  |  |  |  |  |  |
| 5. | 5. REFERENCES   |                 |  |  |  |  |  |  |  |  |
| Δī | PENDIX A: Data Validation Analysis  | 17              |  |  |  |  |  |  |  |  |
|    | PENDIX B: Preliminary Results Obtained from Montana Tech  | 141             |  |  |  |  |  |  |  |  |
|    | -   |                 |  |  |  |  |  |  |  |  |

## Figures

|                                      | Page    |
|--------------------------------------|---------|
| 2-1. Batch Version of the GP Process | <br>. 4 |

## Tables

## Page

| 3-1. Geo2/1–26th September 97  | 9  |
|--|----|
| 3-2. Results for Exp Geo2/1 and the Untreated Freshly Obtained Pit Water               | 10 |
| 3-3. Geo2/2–29th September 97  | 10 |
| 3-4. Results for Exp Geo2/2 and the Untreated Freshly Obtained Pit Water               | 11 |
| 3-5. Geo2/3–30th September 97  | 11 |
| 3-6. Results for Exp Geo2/3 and the Untreated Freshly Obtained Pit Water               | 12 |
| 3-7. Solution Concentrations of Leachate and Acid Dissolution of Precipitates from the |    |
| GP Process Remediation of Berkeley Pit Water   | 13 |
| 3-8. Calculation of Cu and Zn recoveries Using Direct Ammoniacal-Ammonium              |    |
| Carbonate Leaching of the Precipitates   | 13 |
| 4-1. Summary of Geo2 Limited Demonstration Results                                     | 15 |
|  |    |

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### 1. Introduction

In August 1996, Geo2 of Melbourne, Australia, successfully demonstrated at Montana Tech of the University of Montana (Montana Tech) their patented "Green Precipitate" (GP) process, an acid mine remediation technology, on a sample of the Berkeley Pit water. Geo2's GP Process technology arose from earlier studies on the formation of naturally occurring double cation hydroxy mineral compounds (e.g., pyroaurite, hydrotalcite, takovite, "Green Rust,"etc.) at or near the surface of the earth (Ref. 1).

Geo2 Limited is an Australian company listed on the Australian Stock Exchange. The company has some 3,000 shareholders and has been in operation for 10 years. Apart from the GP Process, Geo2 Limited is active in several major research and commercial projects in Australia and Asia.

Following the 1996 demonstration at Montana Tech, Geo2 used the GP process at their laboratories in Australia to treat other acid mine wastewaters with similar but variable compositions. Similar reductions in heavy and toxic metal concentrations were achieved, as in the earlier Berkeley Pit water trials. Uranium and rare earth concentrations in these waters were markedly reduced.

Concurrent with these developments, Geo2 engineers successfully constructed a pilot-plant continuous flow pipe reactor that would use the chemical principles of the GP process to demonstrate the commercial application of the technology.

The next stage of the Berkeley Pit Innovative Technologies (BPIT) Project conducted at Montana Tech called for submissions to treat 30 liters (L) of Berkeley Pit water as an advance beyond the small laboratory-scale demonstration of 1996.

Geo2 was successful in receiving a further invitation to take part in this new demonstration and used a partial approach to their pilot-scale pipe reactor to treat the 30 L of Pit water.

The demonstrations on the Montana Tech campus followed a Quality Assurance Project Plan (QAPP) prepared by Montana Tech and Geo2 Limited and approved by the U.S. Environmental Protection Agency's (EPA) National Risk Management Research Laboratory (NRMRL). The demonstrations were part of the Mine Waste Technology Program (MWTP) and the BPIT Project under the supervision of Dr. Karl Burgher (Project Manager, Montana Tech, MWTP), Mr. Steve Anderson (Manager, BPIT Project), and members of the Academic Staff of Montana Tech and were carried out by Dr. Reginald Taylor and Mr. Ronald Arthur (Principal Investigators) and Mr. Cliff Restarick.

#### **1.1 Demonstration Personnel**

Dr. Reginald M. Taylor, a recently retired Chief Research Scientist in the Australian Commonwealth Scientific and Industrial Research Organization (CSIRO) and an elected Fellow of the Australian Academy of Technological Science and Engineering, invented the GP Process, having previously researched the conditions of the formation of naturally occurring and synthetic members of the pyroaurite (Mg<sub>6</sub>Fe(III)<sub>2</sub> (OH)<sub>16</sub>.CO<sub>3</sub>) group of minerals (Ref. 2, 3, and 4).

Dr. Taylor postulated, from the published composition of the Berkeley Pit water, that by adjusting the environmental conditions, it should be possible to cause a large proportion of the metal cations in solution to react with one another to form a crystalline precipitate of such a Metal(II)Metal(III) hydroxy compound where the Metal(III) can comprise iron (Fe)(III) and aluminum (Al)(III) and the various divalent cations contribute to the Metal(II) composition.

Following initial tests in Australia using samples of Berkeley Pit water, Dr. Taylor successfully demonstrated this process (now patented as the GP process) to support his hypothesis. The precipitate formed was filtered, and the filtrate was found by Inductively Coupled Plasma (ICP) analysis to contain very low (often less than 1 ppm) metal concentrations compared with the original concentrations of many hundreds of ppm.

X-ray diffraction analysis of the filter residue confirmed that the major precipitate was a crystalline member of the pyroaurite group. The ease of synthesizing this type of compound endorses the comment of H. F. W. Taylor, who stated that it is only necessary to bring together in solution appropriate amounts of a divalent and trivalent metal at the right pH and these pyroaurite-type compounds form very readily (Ref. 5).

For most of his professional life, Mr. Clifford J. Restarick, was an Experimental Scientist in the Australian CSIRO's Divisions of Mineral Engineering. His experience embraces fluid bed reactions and particle comminution processes with particular emphasis on the use of hydrocyclones. He is also skilled in metallurgical audits of operating plants and the design and operation of pilot plants. Mr. Restarich is an Associate of the School of Mines at Ballarat in Metallurgy and prior to his CSIRO appointment, worked as a Mill Shift Boss in East and Central Africa. Since 1994, he has been a Private Consultant Metallurgist in all aspects of his previous experience.

Mr. Ronald W. Arthur, B.E. (Mining), B.Sc., F.S.V., Justice of the Peace, Mining Engineer, and the Senior Engineer for Geo2 Limited, is in charge of the Geo2 Research Facility in Geelong, Victoria, Australia. He was one of the founding directors of Murasap Industries Limited, an Australian Public Company engaged in technology research and development of leading edge water and wastewater treatment technologies. His most recent research projects involve "Automatic Self Cleaning Micro-Ceramic Filters" for low and high temperature micro-filtration of industrial and domestic water supplies (R.W. Arthur), "Photocatalytic Oxidation of Water Species" for the removal and destruction of organic toxins, (hydrofluorocarbon) HFC and polychlorinated biphenyl (PCB) in water (R.W. Arthur and Dr. B.H.Boyden), and a special environmental project (classified) with the Royal Australian Navy for the Separation of Hydrocarbons and water (R.W. Arthur and Dr. R.A. Creelman).

### 2. Process Description

The Geo2 Limited bench-scale technology test demonstrated the removal of metal ions from Berkeley Pit water using milk of lime as the pH modifying agent. The precipitate formed during the process was comprised of double or mixed cation hydroxy salts that lend themselves to traditional leaching techniques for metals recovery.

#### 2.1 General Overview

The project addresses the resource recovery, scaleup, and process engineering issues of remediation of the acid mine drainage (AMD) waters of the Berkeley Pit. The project methodology involves:

- demonstration of process to scaleup the GP Process from 400 (milliliters) mL to 30 L;
- the precipitation from the AMD of double or mixed cation hydroxy salts having a pyroauritelike structure in a batch reactor; and
- the execution of metal recovery processes on the precipitate.

To obtain samples of treated water for analysis and precipitate for resource recovery processes, it is proposed to provide a large batch version of the GP process to demonstrate the ability to generate large quantities of the precipitate from fresh samples of the Berkeley Pit water.

The products of the GP process will also be used to show the process engineering issues to be considered in scaling up the process to pilot scale.

#### 2.2 Process Flow Sheet

The process flow sheet for the proposed project work program is provided in Figure 2-1.

## **2.3** Statement of Project Goals and Objectives

The primary objective of this demonstration is to use the GP technology package at a 30-L scale to precipitate heavy metal species from Berkeley Pit water. The anticipated treatment results (ppm in treated water) are as follows:

| 0.03-0.14   |
|-------------|
| < 0.04      |
| 0.002-0.009 |
| < 0.04      |
| 0.01-0.03   |
| < 0.04      |
| 5           |
| 2500        |
| < 0.04      |
| 8.5-8.8     |
|             |

The secondary objectives of the demonstration are given below.

- C To demonstrate without optimization that the precipitate as formed can be leached directly with a suitable reagent to achieve an economical level of Cu and Zn retrieval. It is expected at least 80% of Cu and Zn values in the GP will be extracted. Subsequent research may show how the leach values may be converted into value-added saleable products.
- C To establish the flow characteristics of pre-formed GP slurry in a one-inch diameter pipe system with and without static mixing.
- <sup>C</sup> To quantify aspects of the settling kinetics of the GP in its original suspension.

#### 2.4 Experimental Design

For each trial run of the batch GP process, the following measurements will be taken:

Critical Measurements:

- concentrations of dissolved metals (Al, As, Cd, Cr, Cu, Fe, Mn, and Zn) in remediated water;
- concentrations of Cu and Zn in the precipitate leach solution; and
- concentration of sulfate in remediated water.

Noncritical Measurements:

- room temperature;
- pH of reaction vessel contents on a continuing basis;
- calibration of pH electrodes at room temperature with two standard buffers;
- volume of deep Pit water transferred to reactor;

- timing and volume of multistage addition of pH modifying agents and nucleating agents;
- time to end of reaction; and
- volume of filtrate/sludge.

The critical measurements are the measurements that will be used to choose optimal process control values and to establish reproducibility parameters.

The batch GP reactor will be operated under various conditions of timed multistage addition of pH modifying agents and nucleating agents; it is not possible to anticipate in advance what these conditions will be as optimization proceeds.

One 30-L sample of deep Pit water will be used for each trial experiment of the GP process, and it is planned that up to seven such trial experiments be conducted in Butte, Montana. Three of these trial experiments will be replicates run under optimum process conditions.

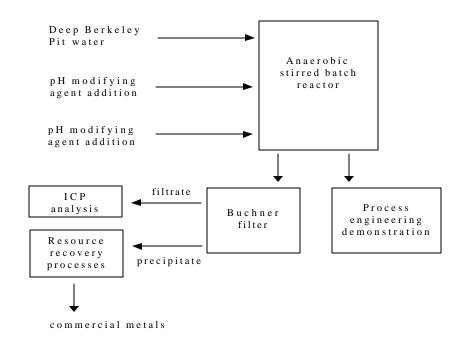


Figure 2-1. Batch version of the GP process.

### 3. Experiment Details for GP Process

The precipitation of a 30-L batch of Berkeley Pit water produced double or mixed cation hydroxy salts displaying the properties of the pyroaurite-like GP obtained in the laboratory-scale demonstration done by Geo2 at Montana Tech in 1996. Three experiments designated Geo2/1, Geo2/2, and Geo2/3 will be described on the following pages. The data obtained from the experiments conducted during this demonstration was validated by standard methods. The results of this validation are contained in Appendix A.

## 3.1 Experiment Geo2/1—September 26, 1997

In this first demonstration of a batch reaction, 30 L of fresh Berkeley Pit water from the 200-foot level were treated. The conditions established in preliminary Experiment 2 were used because these conditions gave cleaner treated water than the other two preliminary tests (see Appendix B). The water, which had been stored at approximately 7 EC prior to its use in this experiment, was syphoned from the sealed container into the reaction vessel that had been previously filled with nitrogen. A calibrated pH probe was inserted below the liquid surface, and the solution was stirred by the blade stirrer. A stirred suspension of lime [approximately 3.44%] calcium oxide (CaO)] also under nitrogen, was delivered by a peristaltic pump at the rate of 270 mL per minute into the bottom level of the Pit water at the level of the stirrer blade. A log recording the time, pH, color changes, and the times that the periostitic pump was turned on and off is given in Table 3-1.

Approximately 1.7 L of the slaked lime suspension were consumed in this experiment, giving a dilution factor of 1.056 (= 31.7/30.0) if it is necessary to convert the analytical data back to the original volume of Pit water used.

Immediately after the termination of the experiment, approximately 2 L of the suspension were withdrawn and filtered through a fast filter (number 101). The clear filtrate showed a lower pH than was recorded when the experiment was terminated. The addition of carbon dioxide  $(CO_2)$ to accelerate the deposition of a calcium sulfate hydrate species in the preliminary experiments did not show any clear advantage over the non-CO<sub>2</sub> treated aliquot after standing overnight (see Appendix B). It was therefore decided to just stir the filtrate vigorously to introduce a small amount of  $CO_2$ . The filtrate was stored overnight in a refrigerator, and after filtering through a 0.45micron filter, the filtrate was sent for official analysis together with a sample of the Pit water used. The results of these analyses are given in Table 3-2, together with the target values and the composition of the Pit water used. Three samples of approximately 5 grams (g) were taken from the fresh green filter cake and used for the tests to show the economic metal values contained within the precipitate could be recovered to a large extent by simple leaching with conventional reagents (see Section 3.6). A sample of the fresh GP was taken and pressed into a plastic container so as to have minimal air space. The container was refilled and sealed for future investigations.

#### 3.1.1 Results

Three water samples were taken from the reactor vessel. The samples were filtered through a 0.45-micron filter, preserved, and sent off for analysis. Table 3-2 represents the results of the analysis of Exp Geo2/1.

## 3.2 Experiment Geo2/2—September 29, 1997

In this second trial, an additional 30 L of fresh Berkeley Pit water from the 200-foot level were treated. Just prior to its use in this experiment, the water had been stored at approximately 7 EC. The same conditions and techniques used in Geo2/1 were used. A calibrated pH probe was inserted below the liquid surface, and the solution was stirred by the blade stirrer. A stirred suspension of lime also under nitrogen (approximately 3.44% CaO) was delivered by a peristaltic pump at the rate of 270 mL per minute into the bottom level of the Pit water at the level of the stirrer blade. A log recording the time, pH, color changes, and the times the peristaltic pump was turned on and off is shown in Table 3-3.

Approximately 2.2 L of the slaked lime suspension were consumed in this experiment, giving a dilution factor of 1.073 (=32.2/30.0) if it is necessary to convert the analytical data back to the original volume of Pit water used.

Immediately after the termination of the experiment, approximately 4 L of the suspension were withdrawn and filtered through a fast filter (number 101). As previously, the clear filtrate showed a lower pH than was recorded when the experiment was terminated. The filtrate was stirred vigorously for approximately 2 hours to introduce a small amount of  $CO_2$  and was stored overnight in a refrigerator. After filtering through a <0.45-micron filter, the filtrate was sent for official analysis, together with a sample of the Pit water used. The results of these analyses are given in Table 3-4, together with the target values and the composition of the Pit water used.

Three samples of approximately 5 grams were taken from the fresh green filter cake and used for the tests to show the economic metal values contained within the precipitate could be recovered to a large extent by simple leaching with conventional reagents (see Section 3.6). A sample of the fresh GP was taken and pressed into a plastic container so as to have minimal air space. The container was refilled and sealed for future investigations.

## 3.3 Experiment Geo2/3—September 30, 1997

In this third remediation experiment, another 30 L of Berkeley Pit water from the 200-foot level stored for over 12 months was treated. Just prior to its use in this experiment, the water had been stored at approximately 7 EC. The same conditions and techniques used in Geo2/1 were used. A calibrated pH probe was inserted below the liquid surface and the solution was stirred by the blade stirrer. A stirred suspension of lime also under nitrogen (approximately 3.44% CaO) was delivered by a peristaltic pump at the rate of 270 mL per minute into the bottom level of the Pit water at the level of the stirrer blade. A log recording the time, pH, color changes, and the times the peristatic pump was turned on and off is given in Table 3-5.

Approximately 2.9 L of the slaked lime suspension were consumed in this experiment, giving a dilution factor of 1.096 (=32.9/30.0) if it is necessary to convert the analytical data back to the original volume of Pit water used.

Immediately after the termination of the experiment, approximately 4 L of the suspension were withdrawn and filtered through a fast filter (number 101). The clear filtrate showed a lower pH than was recorded when the experiment was terminated. The filtrate was stirred vigorously for approximately 2 hours to introduce a small amount of  $CO_2$  and was stored overnight in a refrigerator. After filtering three samples through a <0.45-micron filter, the filtrate was sent for official analysis, together with a sample of the Pit water used. The results of these analyses are given in Table 3-6, together with the target values and the composition of the Pit water used.

Three samples of approximately 5 grams were taken from the fresh green filter cake and used for the tests to show that the economic metal values contained within the precipitate could be recovered to a large extent by simple leaching with conventional reagents (see Section 3.6). A sample of the fresh GP was taken and pressed into a plastic container so as to have minimal air space. The container was refilled and sealed for future investigations.

#### **3.4** Cementation Test of the GP in Pipe

It is important to demonstrate that in commercialization of the GP process, a plant breakdown would not result in the precipitate cementing up the pipes. To this end, a closed circuit of on 1-inch-internal-diameter pipe was completely filled (to exclude all air) with a fresh suspension of the GP. This was allowed to settle out for periods of 1, 3, and 7 days. At the end of these periods, the in-line circulating pump was activated, and the precipitate was immediately brought into a fluidized suspension that circulated easily. There was no evidence of residual particles remaining as a sediment at the lowest flow rate of the pump.

Unfortunately, all air was not excluded, and the very slow oxidation of the GP led to the partial transformation of the precipitate to a black phase, presumably maghemite (Mg-Fe<sub>2</sub>O<sub>3</sub>) or magnetite (Fe<sub>3</sub>O<sub>4</sub>), because of the magnetic influence displayed when a magnetic needle was brought in proximity.

#### **3.5** Settling Kinetics of GP Process

The settling and sedimentation properties of the GP are quite complex, however, also quite reproducible. During the final stages of the precipitate formation, swirls of clear water form patterns in the stirred slurry. This swirl pattern behavior is typical of crystalline and granular particles and indicates the complete absence of ultrafine or submicron particles.

After settlement, the supernatant water is clear. When stirring ceases, the GP particles start to visibly agglomerate, and particle movement stops within 2 minutes. The rate of sedimentation of these flocs varies depending on the container size, shape, composition and slope, and the ease of forming water upflow channels. Sedimentation is faster when it occurs:

- in glass rather than in plastic;
- in small diameter tubes rather than in the 40-L container; and
- in a sloped (inclined) small diameter tube.

The voluminous nature of the GP and the high entrainment of water within and around the floc is typical of the hindered, not-free settling mechanism found in the consolidation zone of a thickener. Disturbed consolidated GP settles faster than the original precipitate.

The hindered settling consolidation rates of fresh GP were compared in:

- 40-L plastic cylinder—30% of original volume after 16 hours;
- 4-L tall plastic cylinder—25% of original volume after 24 hours;
- 1-L glass measuring cylinder on a slope—50% of original volume after 1 hour;
- 200-mL glass measuring cylinder on a slope—30% of original volume after 1 hour; and
- 1-inch-diameter flat pipe unit—50% in 10 mins.

Even at the 30% consolidation level, the settled floc is estimated to contain > 90% water; therefore, it is quite fluid, does not cement permanently, and can be caused to flow easily even after 14 days.

## **3.6 Recovery of Copper and Zinc from the Precipitate**

#### 3.6.1 Aim of Test Demonstration

These experiments were directed towards the ultimate recovery of the economic metal values within the precipitate. Experiments described in this report were carried out on the precipitates formed in the treatments Geo2/1 and were to demonstrate that the precipitate formed in the GP process is suitable for successful leaching. No effort was made to optimize the Cu and Zn extraction. Only one leach solution was used, an ammoniacal-ammonium carbonate solution containing 100 grams of ammonium carbonate, 306 mL of 25% aqueous ammonia, and 594 mL of water.

#### 3.6.2 Background

The experimental design was based on preliminary experiments carried out by Geo2 in their Melbourne laboratories where 0.5 grams of precipitate was treated with approximately 50 mL of ammoniacal-ammonium carbonate solution to give over 80% recovery of the contained Cu and Zn.

#### 3.6.3 Procedure

Two leach extractions were carried out on each of the three precipitates. Approximately 5 grams of freshly formed, filtered, but moist precipitate was used in each experiment. The precipitates were dispersed in varying volumes of the leachate in conical flasks and placed in a reciprocating water bath at 33 EC for 2 hours, with the oscillation frequency fixed at either 100 or 120 per minute. Approximately 5 grams of each of the precipitates were also dissolved in a small amount of concentrated hydrogen chloride (HCl) to determine the absolute amounts of Cu and Zn per gram in each precipitate. The acid extracts were made up to a volume of 100 mL for analysis by ICP. From these values, expressed as milligrams per gram (mg/g) of precipitate, the amounts of Cu or Zn that could be expected in a known weight of the same precipitate used for the leaching can be calculated. It is to be noted in the series Geo2/1 and /2, freshly bottled Pit water was used, whereas for Geo2/3, water gathered over 1 year ago and stored under nitrogen at the MSE facility in Butte was used. The major difference in the two solutions is in the dissolved Fe concentrations.

The extractions carried out on Geo2/1 precipitates used a oscillation rate in the reciprocating water bath of 100/min. In the extraction of the Geo2/2, the 5-grams precipitates were dispersed in the leach solution by immersion of the precipitate and leach solution for approximately 2 minutes in an ultrasonic bath. In the extraction, the first hour was at 100 oscillations/minute and the second at 120 per min. The third series using the precipitate from Geo2/3 utilized 120 oscillations/min.

#### 3.6.4 Results

The results obtained for these extractions from ICP analyses performed at Montana Tech are listed in Tables 3-7 and 3-8.

This leads to the following percentage recovery for the two elements in the various volumes of leachate. The following demonstrates the method of calculation. The precipitate of Geo2/1 of 5.016 grams dissolved in HCl and made up to 100 mL contains 163 ppm Cu in a 100-mL volume. Therefore, a total of 16.3 milligrams (mg) of Cu arises from the 5.016 grams giving a Cu content of 3.24 mg/g of precipitate. Precipitate used in the first ammoniacal extraction of 4.972 grams should therefore contain 3.24 x 4.972 mg Cu, i.e., 16.15mg Cu. The 500-mL leachate from this sample had a concentration of 32.1 ppm; therefore, the total leachate contained 500 x 32.1, equal to 16.050-mg giving a 99.35% recovery.

These extractions have not been optimized with regard to concentrations, different leaching solutions, temperature, or times. The average recoveries in the five leaching experiments where the extraction did not exceed 100% were 91.7% Cu and 86.5% Zn. These results markedly demonstrate that the precipitate from the GP process, applied to the remediation of Berkeley Pit water, responds very well to direct conventional Cu and Zn extraction techniques.

The high percentages of Cu and Zn extracted from the first sample of Geo2/2 precipitate can

only indicate an incorrect measure of the weight of precipitate used in this extraction. The data used to derive these recovery values were obtained from the MSE-HKM laboratory of Butte, Montana, and ACZ Laboratories of Colorado. There were no systematic differences in the results obtained. It is also seen that the volume of leach solution used for approximately the same weight of precipitate did not seem to affect the efficiency of the extraction. This recovery procedure could be optimized with due regard to all suitable extractants and prior art, and the variations that may be introduced by oxidation and drying out of the precipitate.

| Table 3-1. Geo2/1-20 | September 97 |       |       |                                 |
|----------------------|--------------|-------|-------|---------------------------------|
| Elapsed Time (min)   | pH           | Mark  | рН    | Observation                     |
| 0.00                 | 2.89         |       |       | Lime ON - 6.0 EC.               |
| 1.00                 | 3.86         |       |       | Yellow/Green                    |
| 2.00                 | 4.67         |       |       | Distinct Yellow/Green           |
| 3.00                 | 5.20         |       |       | Light Olive Green               |
| 4.00                 | 5.83         |       |       | Apple Green                     |
|                      |              | 4.35  | 6.17  | Lime OFF                        |
| 5.00                 | 6.37         |       |       | Going darker                    |
|                      |              | 5.13  | 6.41  | Lime ON                         |
| 6.00                 | 7.42         |       |       |                                 |
|                      |              | 6.40  | 9.63  | Lime OFF                        |
| 7.00                 | 10.16        |       |       | Dark Green - 8.2 EC.            |
| 8.00                 | 10.02        |       |       |                                 |
| 9.00                 | 9.84         |       |       |                                 |
| 10.00                | 9.73         |       |       |                                 |
| 11.00                | 9.65         |       |       |                                 |
| 12.00                | 9.61         |       |       |                                 |
| 13.00                | 9.57         |       |       |                                 |
| 14.00                | 9.54         |       |       |                                 |
| 15.00                | 9.52         |       |       |                                 |
|                      |              | 15.05 | 9.52  | Lime ON                         |
|                      |              | 15.33 | 10.04 | Lime OFF - pH rose to 10.44     |
| 16.00                | 10.28        |       |       |                                 |
| 17.00                | 10.02        |       |       | 8.5 EC.                         |
| 18.00                | 9.90         |       |       |                                 |
| 19.00                | 9.82         |       |       |                                 |
| 20.00                | 9.77         |       |       |                                 |
| 21.00                | 9.74         |       |       |                                 |
| 22.00                | 9.71         |       |       |                                 |
| 23.00                | 9.69         |       |       |                                 |
| 24.00                | 9.66         |       |       |                                 |
| 27.00                | 9.63         |       |       |                                 |
| 28.00                | 9.61         |       |       |                                 |
| 29.00                | 9.60         |       |       |                                 |
| 30.00                | 9.59         |       |       | Experiment Terminated - 8.9 EC. |
|                      |              |       |       |                                 |

#### Table 3-1. Geo2/1—26<sup>th</sup> September 97

| Contaminant     | Sample #<br>Geo BP-1 | Sample #<br>Geo1-1 | Sample #<br>Geo1-2 | Sample#<br>Geo1-3 | Target<br>Concentration |
|-----------------|----------------------|--------------------|--------------------|-------------------|-------------------------|
| Al              | 305                  | 0.31               | 0.3                | 0.32              | 0.03 -                  |
|                 |                      |                    |                    |                   | 0.14                    |
| As              | <1                   | < 0.2              | < 0.2              | < 0.2             | < 0.04                  |
| Cd              | 2.46                 | 0.016              | 0.011              | 0.013             | 0.002                   |
|                 |                      |                    |                    |                   | - 0.009                 |
| Cr              | 0.06                 | 0.02               | < 0.02             | < 0.02            | < 0.04                  |
| Cu              | 189                  | 0.02               | < 0.02             | < 0.02            | 0.01 -                  |
|                 |                      |                    |                    |                   | 0.03                    |
| Fe              | 1,100                | < 0.02             | < 0.02             | < 0.02            | < 0.04                  |
| Mn              | 236                  | 4.94               | 4.72               | 4.66              | 5.0                     |
| Zn              | 626                  | < 0.05             | < 0.05             | < 0.05            | < 0.04                  |
| $\mathbf{SO}_4$ | 7,890                | 2,670              | 2,640              | 2,590             | 2,500                   |
| pН              | 3                    | 7                  | 7.3                | 7.3               | 8.5 - 8.8               |
|                 |                      |                    |                    |                   |                         |

Table 3-2. Results for Exp Geo2/1 and the Untreated Freshly Obtained Pit Water

#### Table 3-3. Geo2/2—29th September 97

| Elapsed Time (min) | pН    | Mark  | pН    | Observation              |
|--------------------|-------|-------|-------|--------------------------|
| 0.00               | 2.90  |       |       | Lime turned ON - 8.4 EC. |
| 1.00               | 3.73  |       |       | Yellow color             |
| 2.00               | 4.54  |       |       | Yellow/Green             |
| 3.00               | 4.96  |       |       | Yellow/Apple Green       |
| 4.00               | 5.44  |       |       | Apple Green - 9.5 EC.    |
| 5.00               | 5.86  |       |       |                          |
|                    |       | 5.21  | 6.00  | Lime OFF                 |
| 6.00               | 6.24  |       |       |                          |
|                    |       | 6.47  | 6.41  | Lime ON                  |
| 7.00               | 6.48  |       |       |                          |
| 8.00               | 7.56  |       |       | Dark Olive Green         |
|                    |       | 8.53  | 9.78  | Lime OFF                 |
| 9.00               | 10.13 |       |       |                          |
| 10.00              | 10.04 |       |       |                          |
| 11.00              | 9.82  |       |       |                          |
| 12.00              | 9.69  |       |       | 10.5 EC.                 |
| 13.00              | 9.60  |       |       |                          |
| 14.00              | 9.54  |       |       |                          |
|                    |       | 14.30 | 9.52  | Lime ON                  |
| 15.00              | 10.05 | 15.00 | 10.05 | Lime OFF                 |

| 16.00 | 10.11 |
|-------|-------|
| 17.00 | 9.92  |
| 18.00 | 9.81  |

#### Table 3-3. Continued

| Elapsed Time (min) | рН   | Mark | рН | Observation           |
|--------------------|------|------|----|-----------------------|
| 19.00              | 9.75 |      |    |                       |
| 20.00              | 9.71 |      |    |                       |
| 21.00              | 9.67 |      |    |                       |
| 24.00              | 9.61 |      |    |                       |
| 25.00              | 9.60 |      |    |                       |
| 26.00              | 9.59 |      |    |                       |
| 27.00              | 9.58 |      |    |                       |
| 28.00              | 9.56 |      |    |                       |
| 29.00              | 9.56 |      |    | 11.0 EC.              |
| 30.00              | 9.55 |      |    | Experiment Terminated |

#### Table 3-4. Results for Exp Geo2/2 and the Untreated Freshly Obtained Pit Water

| Contaminant     | Sample #<br>Geo BP-2 | Sample #<br>Geo2-1 | Sample #<br>Geo2-2 | Sample#<br>Geo2-3 | Target<br>Concentration |
|-----------------|----------------------|--------------------|--------------------|-------------------|-------------------------|
| Al              | 303                  | 0.3                | 0.2                | <0.2              | 0.03 -<br>0.14          |
| As              | <2                   | < 0.2              | < 0.2              | < 0.4             | < 0.04                  |
| Cd              | 2.4                  | < 0.02             | < 0.02             | <0.03             | 0.002 -<br>0.009        |
| Cr              | < 0.5                | < 0.05             | < 0.05             | < 0.1             | < 0.04                  |
| Cu              | 205                  | <0.1               | <0.1               | <0.1              | 0.01 -<br>0.03          |
| Fe              | 1,140                | < 0.05             | < 0.05             | < 0.1             | < 0.04                  |
| Mn              | 244                  | 3.83               | 3.8                | 4.02              | 5.0                     |
| Zn              | 673                  | 0.06               | 0.05               | 0.1               | < 0.04                  |
| $\mathbf{SO}_4$ | 9,530                | 3,080              | 3,040              | 3,170             | 2,500                   |
| рН              | 2.9                  | 8.2                | 8.3                | 8.3               | 8.5 - 8.8               |

#### Table 3-5. Geo2/3—30<sup>th</sup> September 97

| Elapsed Time (min) | pН   | Mark | pH | Observation             |
|--------------------|------|------|----|-------------------------|
| 0.00               | 2.42 |      |    | Lime ON - Temp. 5.8 EC. |
| 1.00               | 2.96 |      |    | Yellow/Brown            |
| 2.00               | 3.99 |      |    | Yellow                  |
| 3.00               | 4.67 |      |    | Yellow                  |
| 4.00               | 4.98 |      |    | Yellow/Green - 7.3 EC.  |
| 5.00               | 5.40 |      |    | Yellow/Green            |
|                    |      |      |    |                         |

| 6.00 | 5.74 |      |      | Green                          |
|------|------|------|------|--------------------------------|
|      |      | 6.48 | 6.00 | Lime OFF - pH rising - 8.0 EC. |

#### Table 3-5. Continued

| Elapsed Time (min) | рН    | Mark  | рН    | Observation                |
|--------------------|-------|-------|-------|----------------------------|
| 7.00               | 6.09  |       |       |                            |
| 8.00               | 6.17  |       |       |                            |
|                    |       | 8.15  | 6.20  | Lime ON                    |
| 9.00               | 6.38  |       |       | Dark Olive Green - 8.1 EC. |
| 10.00              | 7.07  |       |       |                            |
|                    |       | 10.05 | 7.07  | Lime OFF                   |
| 11.00              | 7.58  |       |       | Dark Green                 |
| 12.00              | 7.84  |       |       |                            |
| 14.00              | 9.64  |       |       | 8.7 EC.                    |
|                    |       | 14.14 | 10.00 | Lime OFF                   |
| 13.00              | 8.04  |       |       | Dark Green - 8.5 EC.       |
| 15.00              | 10.16 |       |       |                            |
| 16.00              | 9.91  |       |       |                            |
| 17.00              | 9.75  |       |       |                            |
| 18.00              | 9.66  |       |       |                            |
|                    |       | 18.31 | 9.63  | Lime ON                    |
| 19.00              | 9.92  |       |       |                            |
|                    |       | 19.06 | 10.02 | Lime Off - 8.9 EC.         |
|                    |       | 19.30 | 10.27 | Highest pH peak            |
| 20.00              | 10.10 |       |       |                            |
| 21.00              | 9.92  |       |       |                            |
| 22.00              | 9.81  |       |       |                            |
| 23.00              | 9.76  |       |       |                            |
| 24.00              | 9.72  |       |       |                            |
| 25.00              | 9.69  |       |       | 9.1 EC.                    |
| 26.00              | 9.67  |       |       |                            |
| 27.00              | 9.65  |       |       |                            |
| 28.00              | 9.63  |       |       |                            |
| 29.00              | 9.62  |       |       |                            |
| 30.00              | 9.61  |       |       | Experiment Terminated      |
|                    |       |       |       |                            |

### Table 3-6. Results for Exp Geo2/3 and the Untreated Freshly Obtained Pit Water

| Contaminant | Sample # | Sample # | Sample # | Sample# | Target        |
|-------------|----------|----------|----------|---------|---------------|
|             | Geo BP-3 | Geo3-1   | Geo3-2   | Geo3-3  | Concentration |
| Al          | 304      | <0.3     | <0.3     | <0.3    | 0.03 - 0.14   |

| As               | <2                   | <0.4               | <0.4               | <0.4              | < 0.04                  |
|------------------|----------------------|--------------------|--------------------|-------------------|-------------------------|
| Cd               | 2.39                 | 0.006              | 0.006              | 0.006             | 0.002 - 0.009           |
| Cr               | <0.5                 | < 0.1              | < 0.1              | < 0.1             | < 0.04                  |
| Table 3-6. Conti | nued                 |                    |                    |                   |                         |
| Contaminant      | Sample #<br>Geo BP-3 | Sample #<br>Geo3-1 | Sample #<br>Geo3-2 | Sample#<br>Geo3-3 | Target<br>Concentration |
| Cu               | 192                  | < 0.1              | < 0.1              | <0.1              | 0.01 - 0.03             |
| Fe               | 835                  | < 0.1              | < 0.1              | < 0.1             | < 0.04                  |
| Mn               | 227                  | 4.33               | 4.32               | 4.26              | 5.0                     |
| Zn               | 637                  | < 0.1              | < 0.1              | < 0.1             | < 0.04                  |
| ${ m SO}_4$      | 10,100               | 3,100              | 3,520              | 3,150             | 2,500                   |
| pН               | 2.5                  | 7.3                | 7.4                | 7.3               | 8.5 - 8.8               |

| Table 3-7. Solution Concentrations of Leachates and Acid Dissolution of Precipitates from the GP Process |
|--|
| Remediation of Berkeley Pit Water  |

| Sample  | Volume of<br>Leach Soln.<br>(mL) | Final Volume<br>(mL) | Weight of<br>Precipitate<br>Leached (g) | Concentration<br>Cu (ppm) | Concentration<br>Zn (ppm) |
|---------|----------------------------------|----------------------|---|---------------------------|---------------------------|
| GeoAL-1 |                                  | 100                  | 5.016                                   | 163                       | 500                       |
| GeoL-1  | 250                              | 500                  | 4.972                                   | 32.1                      | 97.6                      |
| GeoL-1A | 250                              | 500                  | 5.16                                    | 25.9                      | 81.4                      |
| GeoAL-2 |                                  | 100                  | 5.032                                   | 122                       | 372                       |
| GeoL-2  | 170                              | 500                  | 4.984                                   | 26.6                      | 79                        |
| GeoL-2A | 250                              | 500                  | 5.051                                   | 24.3                      | 71.3                      |
| GeoAL-3 |                                  | 100                  | 5.037                                   | 113                       | 350                       |
| GeoL-3  | 100                              | 500                  | 5.135                                   | 21.2                      | 63.7                      |
| GeoL-3A | 200                              | 500                  | 5.307                                   | 21.6                      | 51.2                      |

 Table 3-8. Calculation of Cu and Zn Recoveries Using Direct Ammoniacal-Ammonium Carbonate Leaching of the Precipitates

| Sample<br>Number | Volume<br>Leach<br>solution | Cu mg in<br>amount of<br>precipitate | mg Cu<br>leached | % Cu<br>Recovery | Zn mg in<br>amount of<br>precipitate. | mg Zn<br>leached | % Zn<br>recovery |
|------------------|-----------------------------|--------------------------------------|------------------|------------------|---------------------------------------|------------------|------------------|
| GeoL-1           | 250 mL                      | 16.154                               | 16.050           | 99.35            | 49.561                                | 48.8             | 99.67            |
| GeoL-1A          | 250 mL                      | 16.764                               | 12.950           | 77.20            | 51.534                                | 40.700           | 78.9             |
| GeoL-2           | 170mL                       | 12.083                               | 13.30            | 110              | 36.845                                | 39.51            | 107              |
| GeoL-2A          | 250mL                       | 12.243                               | 12.150           | 99.24            | 37.326                                | 35.65            | 95.5             |
| GeoL-3           | 100 mL                      | 11.519                               | 10.6             | 92.02            | 35.729                                | 31.85            | 89.14            |
| GeoL-3A          | 200 mL                      | 11.887                               | 10.75            | 90.85            | 36.926                                | 25.600           | 69.32            |

### 4. Summary and Conclusions

The primary objective of this demonstration was the treatment of Berkeley Pit water to the extent that it could be safely used for agricultural application. Secondary objectives were to:

- demonstrate the adaptability of the technology to a continuous treatment that is scalable to match or exceed present inflow rates into the Berkeley Pit and is cost effective;
- evaluate the possible recovery of commercial metals in the compacted precipitate; and
- investigate parameters such as the settling rate of the precipitate and its physical properties that may enhance or reduce the economic attractiveness of the process.

The results of analyses from the three experiments indicate that the technology is capable of removing the majority of dissolved metals from the Berkeley Pit water. The detection limits of some of the metals are higher than normal due to interferences encountered during the analysis, probably due to the high concentration of calcium present in the treated water. However, the presence of supersaturated calcium sulfate (gypsum) in the treated water presents no disadvantage for agricultural use. In alkaline soils, gypsum is commonly added to improve the soil structure. Table 4-1 summarizes the results of the Geo2 Limited technology demonstration.

#### 4.1 Economic Evaluation

Because of the small scale of the technology demonstration performed at Montana Tech, a complete economic evaluation is not possible. However, by comparing the amount of lime used in the GP process with the amount used in the MWTP Activity IV, Project 1, Berkeley Pit Water Treatment Research project that became the treatment technology specified in the record of decision (ROD) for remediating the Berkeley Pit, a cost comparison can be made. Data presented in the Final Report for Activity IV, Project 1 of the MWTP indicated the amount of lime needed to raise the final pH of the Berkeley Pit water to approximately 9.6 was 4.35 grams per liter (g/L). The Geo2 GP Process, to achieve a similar final pH, needed an average lime ratio of approximately 2.7 g/l. This is approximately 40% less lime being used than with the technology specified in the ROD, thus resulting in a savings in lime costs as well as sludge disposal costs.

Also, the sludge created during the process is capable of being leached to recover Cu and Zn. Demonstration results indicate over 90% Cu recovery and approximately 86% Zn recovery. The recovery process has not been optimized; therefore, it is impossible to determine the exact economic benefit of employing this option on the sludge produced.

#### 4.2 Independent Technology Review

The Geo2 GP Process performed up to expectation in most areas of the demonstration. The process removed Al, As, Cd, Cu, Cr, and Zn to levels low enough to meet the most stringent standards that apply to Montana. Sulfate and Mn were greatly reduced; however, they were not removed to levels relative to the other metals analyzed. Sulfate was reduced by approximately 75%, while 98% of the Mn was removed.

The technology is based on using unoxidized Berkeley Pit water and adjusting the pH using lime. By not using an oxidation step in the process, chemical equations indicated that less lime need to be used to raise the pH to specific levels. This technology did reduce the lime consumed to 40% of that of the technology indicated in the ROD for Berkeley Pit cleanup. Overall, the technology performed as expected, based on the data that was collected during the demonstration. To better assess this technology, the following additional information needs to be investigated:

- more work in the recovery of metals from the sludge; and
- a method to remove sulfate.

This technology does show promise in the treatment of many different mine waste waters.

- a complete economic evaluation for a scale-up to treat larger flows;
- Toxicity characteristic leaching procedure (TCLP) tests on the sludge produced;

| Tuble I I | Summu |       | Emitted De | monstratio | II Itebuits |        |      |        |         |     |
|-----------|-------|-------|------------|------------|-------------|--------|------|--------|---------|-----|
| Sample    | Al    | As    | Cd         | Cr         | Cu          | Fe     | Mn   | Zn     | Sulfate | рН  |
| Number    | ppm   | ppm   | ppm        | ppm        | ppm         | ppm    | ppm  | ppm    | ppm     |     |
| Geo1-1    | 0.31  | < 0.2 | 0.016      | 0.02       | 0.02        | < 0.02 | 4.94 | < 0.05 | 2,670   | 7.0 |
| Geo1-2    | 0.30  | < 0.2 | 0.011      | < 0.02     | < 0.02      | < 0.02 | 4.72 | 0.05   | 2,640   | 7.3 |
| Geo1-3    | 0.32  | < 0.2 | 0.013      | 0.02       | < 0.02      | < 0.02 | 4.66 | 0.05   | 2,590   | 7.3 |
| Geo2-1    | 0.3   | < 0.2 | < 0.02     | < 0.05     | < 0.1       | < 0.05 | 3.83 | 0.06   | 3,080   | 8.2 |
| Geo2-2    | 0.2   | < 0.2 | < 0.02     | < 0.05     | < 0.1       | < 0.05 | 3.80 | 0.05   | 3,040   | 8.3 |
| Geo2-3    | < 0.2 | < 0.2 | < 0.02     | < 0.05     | < 0.1       | < 0.05 | 4.02 | < 0.1  | 3,170   | 8.3 |
| Geo3-1    | < 0.3 | < 0.4 | 0.006      | < 0.1      | < 0.1       | < 0.1  | 4.33 | < 0.1  | 3,100   | 7.3 |
| Geo3-2    | < 0.3 | < 0.4 | 0.006      | < 0.1      | < 0.1       | < 0.1  | 4.32 | < 0.1  | 3,520   | 7.3 |
| Geo3-3    | < 0.3 | < 0.4 | 0.006      | < 0.1      | < 0.1       | < 0.1  | 4.26 | < 0.1  | 3,150   | 7.3 |
| GeoL-1    |       |       |            |            | 32.2        |        |      | 97.6   |         |     |
| GeoL-2    |       |       |            |            | 26.0        |        |      | 79.0   |         |     |
| GeoL-3    |       |       |            |            | 21.2        |        |      | 63.7   |         |     |
| GeoL-1A   |       |       |            |            | 25.9        |        |      | 81.4   |         |     |
| GeoL-2A   |       |       |            |            | 24.3        |        |      | 71.3   |         |     |
| GeoL-3A   |       |       |            |            | 21.6        |        |      | 51.2   |         |     |
| GeoAL-1   |       |       |            |            | 163         |        |      | 500    |         |     |
| GeoAL-2   |       |       |            |            | 122         |        |      | 372    |         |     |
| GeoAL-3   |       |       |            |            | 113         |        |      | 350    |         |     |
| GeoBP-1   | 305   | <1    | 2.46       | 0.06       | 189         | 1,100  | 236  | 626    | 7,890   | 3.0 |
| GeoBP-2   | 303   | <2    | 2.4        | < 0.5      | 205         | 1,140  | 244  | 673    | 9,530   | 2.9 |
| GeoBP-3   | 304   | <2    | 2.39       | < 0.5      | 192         | 835    | 227  | 637    | 10,100  | 2.5 |
|           |       |       |            |            |             |        |      |        |         |     |

#### Table 4-1. Summary of Geo2 Limited Demonstration Results

## 5. References

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Appendix A Data Validation

## Appendix B Preliminary Results Obtained From Montana Tech

Three initial experiments were conducted for the purpose of modifying the apparatus and experimental techniques to optimize the process. The rates of delivery of the pH modifying agent, the rate of stirring, and techniques to reduce the residual calcium in the filtrate were investigated. The experiments are designated by a number, the date that the experiments were conducted, and whether or not the filtrates from the treated samples were treated with carbon dioxide ( $CO_2$ ) to accelerate calcium (Ca) deposition as calcium carbonate and gypsum.

In these preliminary experiments, Pit water stored under nitrogen at MSE Technology Applications, Inc. (MSE) was used. The composition of this water varied from that of the fresh Pit water that was used in the three main experiments sampled for this work on September 8, 1997. A comparison between the composition of the stored and fresh Pit waters is given in Table B-1 of this Appendix.

Measured quantities of the stored water were added to a cylindrical perspex container that was 300-millimeters (mm) internal diameter (ID) by 600-mm high that was graduated to mark off 5-liter (L) intervals up to 35 liters. The top was fitted with five ports that were used respectively to:

- introduce the slaked lime suspension pH modifying agent;
- allow the insertion of a pH probe;
- permit nitrogen to flow into the cylinder and form a blanket above the liquid interface;
- insert a blade stirrer fixed to an external electric motor; and
- permit a bubbler to allow the nitrogen to escape if necessary.

A slaked lime suspension of known concentration also under a nitrogen atmosphere was added to a particular volume of the Pit water in the cylinder. The lime suspension reservoir was also graduated so that the volume used for a particular experimental method could be gauged. At the conclusion of the experiment, the volume of lime suspension could be used to obtain a dilution factor for an accurate determination of the elemental concentrations in the treated water samples for comparison of efficiencies of the different technique modifications. The optimum conditions were to be used in the final three experiments that were to determine reproducibility of the technique.

## Preliminary Experiment 1—September 18, 1997

In this first trial, 15.1 liters of the stored Berkeley Pit water taken from a depth of 200 feet were used. The water was syphoned from the sealed container into the reaction vessel that had been previously filled with nitrogen. A calibrated pH probe was inserted below the liquid surface and the solution was stirred by the blade stirrer. A stirred suspension of lime (3.33% Ca) also under nitrogen was delivered through a peristaltic pump at the rate of 102 mL per minute above the top of the Pit water. A log recording the time, pH, color changes, and the times at which the peristatic pump was turned on and off is presented in Table B-2.

A volume of 850 mL of the 3.33% Ca slaked lime suspension were used in this experiment, and with the 10 mL of water used for washing, a dilution factor of 1.0569 (=15960/15100) must be used to modify the measured elemental concentrations that are shown in the following Table B-3. Immediately after the termination of the experiment, approximately 2 liters of the suspension were withdrawn and filtered through a fast filter. The clear filtrate showed a lower pH than was recorded when the experiment was terminated (8.85 vs. 9.08). An aliquot of the filtrate was divided into two portions. In one portion,  $CO_2$  was passed until the pH had dropped to approximately 6.2. This produced an immediate cloudiness that accelerated the subsequent precipitation of calcium sulfate in one of its hydrated forms. The aliquot without the  $CO_2$  was much slower in exhibiting a cloudiness and calcium sulfate precipitation. After standing 16 hours, both portions of the filtrate had large amounts of the white crystalline precipitate.

Although it appeared that the sample treated with  $CO_2$  had a larger amount of precipitation, the subsequent analysis showed there was, in fact, very little difference in the amounts of Ca still in solution or in the sulfate levels. Most of the elements originally present in the stored Pit water, with the exception of manganese (Mn), are seen to be markedly reduced. No explanation is offered for the high residual Mn content.

#### Preliminary Experiment 2—September 22,

**1997** In this second trial, 30 liters of the stored 200-foot Berkeley Pit water were used. The water was syphoned from the sealed container into the reaction vessel that had been previously filled with nitrogen. A calibrated pH probe was inserted below the liquid surface, and the solution was stirred by the blade stirrer. A stirred suspension of lime (3.33% Ca), also under nitrogen, was delivered through a peristaltic pump at the rate of 200 mL per minute into the bottom level of the Pit water at the level of the stirrer blade. A log recording the time, pH, color changes, and the times that the peristatic pump was turned on and off is given in Table B-4.

In this experiment, 2.25 liters of the 3.33% Ca slaked lime suspension was used, giving a dilution factor of 1.075 (=15.950/15.10) that must be used to modify the measured elemental concentrations

for the filtered sample given in Table B-5. Immediately after the termination of the experiment, approximately 2 liters of the suspension were withdrawn and filtered through a fast filter. As previously, the clear filtrate showed a lower pH than was recorded when the experiment was terminated (9.31 vs. 9.81). An aliquot of the filtrate was divided into two portions. In one portion,  $CO_2$  was passed until the pH had dropped to approximately 7.24. This produced an immediate cloudiness that accelerated the subsequent precipitation of calcium sulfate in one of its hydrated forms. The aliquot without the CO<sub>2</sub> was much slower in exhibiting a cloudiness and calcium sulfate precipitation. After standing 16 hours, both portions of the filtrate had large amounts of the white crystalline precipitate.

## Preliminary Experiment 3—September 23, 1997

In this second trial, 24.8 liters of the stored 200-foot Berkeley Pit water were used. The water was syphoned from the sealed container into the reaction vessel that had been previously filled with nitrogen. A calibrated pH probe was inserted below the liquid surface and the solution was stirred by the blade stirrer. A stirred suspension of lime (3.33% Ca), also under nitrogen, was delivered through a peristaltic pump at the rate of 100 mL per minute into the bottom level of the Pit water at the level of the stirrer blade. A log recording the time, pH, color changes, and the times that the peristatic pump was turned on and off is given in Table B-6.

In this experiment, 1.49 liters of the 3.33% Ca slaked lime suspension was used, and with the 10 mLs water used for washing, a dilution factor of 1.06 (=26.370/24.880) must be used to modify the measured elemental concentrations that are shown in Table B-7. Immediately after the termination of the experiment, approximately 2 liters of the suspension were withdrawn and filtered through a fast filter. The clear filtrate

showed a lower pH than was recorded when the experiment was terminated (8.72 vs. 9.33). An aliquot of the filtrate was divided into two portions. In one portion,  $CO_2$  was passed until the pH had dropped to approximately 4.7. This produced an immediate cloudiness that accelerated the subsequent precipitation of calcium sulfate in one of its hydrated forms. The aliquot without the  $CO_2$  was much slower is exhibiting a cloudiness and calcium sulfate precipitation. After standing 16 hours, both portions of the filtrate had large amounts of the white crystalline precipitate and the pH of the sample was 5.12, whereas the aliquot without  $CO_2$  had dropped to 6.24.

All analyses in this Appendix were carried out by Dr. Bill Chattam of the Department of Chemistry and Geochemistry of the Montana Tech.

#### Conclusions

From the results of the above three preliminary experiments, it was concluded that, given the time delay between filtration and analysis, there is no real benefit in the initial treatment with  $CO_2$ . The elemental concentrations in the filtrate of Experiment 2 showed that the conditions used, such as the rate and method of delivery of the lime and the stirring rate, should be the parameters used in the official runs Geo2/1, Geo2/2, and Geo2/3 given in this report.

| Table B-1. Comparison of Composition of Berkeley Pit Waters Collected from 200 Feet, Stored at MSE for |
|--|
| Approximately 1 Year, Compared to Freshly Collected 11 Sept 1997                                       |

| Elements | Sample: Stored Pit Water (ppm) | Sample: Fresh Pit Water (ppm) |
|----------|--------------------------------|-------------------------------|
| Al       | 275.8                          | 290.2                         |
| As       | 0.0406                         | 0.594                         |
| Ca       | 440.5                          | 444.34                        |
| Cd       | 2.177                          | 2.260                         |
| Cr       | 0.0758                         | U                             |
| Cu       | 190.46                         | 198.839                       |
| Fe       | 758.5 8                        | 1,067.058                     |
| Mg       | 440.556                        | 444.338                       |
| Mn       | 200.779                        | 227.52                        |
| Ni       | 1.234                          | 1.209                         |
| S        | 2,467                          | 2,645                         |
| Zn       | 605.6                          | 607.7                         |

| Table B-2. | Peristatic | Pump Log | g—Experiment 1 | L |
|------------|------------|----------|----------------|---|
|------------|------------|----------|----------------|---|

| Elapsed in Mins | Mark Time | рН   | Observation                            |
|-----------------|-----------|------|--|
| 0.00            |           | 2.55 | Raw Berkeley Pit water at commencement |
| 1.00            |           | 3.24 | Yellow /Green                          |
| 2.00            |           | 4.14 | Yellow /Green                          |
| 3.00            |           | 4.70 | Yellow /Green                          |
| 4.00            |           | 5.30 | Yellow going Greener                   |
| 5.00            |           | 6.17 | Lime turned OFF                        |
|                 |           |      |  |

| 6.00 | 6.30 |
|------|------|
| 7.00 | 7.48 |
| 8.00 | 7.53 |

#### Table B-2. Continued

| Table B-2. Continued |           |       |  |  |  |  |  |  |  |
|----------------------|-----------|-------|--|--|--|--|--|--|--|
| Elapsed in Mins      | Mark Time | pH    |  |  |  |  |  |  |  |
| 9.00                 |           | 7.61  |  |  |  |  |  |  |  |
| 10.00                |           | 7.65  |  |  |  |  |  |  |  |
| 10.30                | 10.30     | 9.01  |  |  |  |  |  |  |  |
| 11.00                |           | 9.01  |  |  |  |  |  |  |  |
| 12.00                |           | 8.87  |  |  |  |  |  |  |  |
| 13.00                |           | 8.77  |  |  |  |  |  |  |  |
| 14.00                |           | 8.71  |  |  |  |  |  |  |  |
| 14.19                | 14.19     | 10.37 |  |  |  |  |  |  |  |
| 15.00                |           | 9.40  |  |  |  |  |  |  |  |
| 16.00                |           | 9.15  |  |  |  |  |  |  |  |
| 17.00                |           | 9.04  |  |  |  |  |  |  |  |
| 18.00                |           | 8.97  |  |  |  |  |  |  |  |
| 19.00                |           | 8.93  |  |  |  |  |  |  |  |
| 20.00                |           | 8.90  |  |  |  |  |  |  |  |
| 20.26                | 20.26     | 10.28 |  |  |  |  |  |  |  |
| 21.00                |           | 9.88  |  |  |  |  |  |  |  |
| 22.00                |           | 9.58  |  |  |  |  |  |  |  |
| 23.00                |           | 9.44  |  |  |  |  |  |  |  |
| 24.00                |           | 9.35  |  |  |  |  |  |  |  |
| 25.00                |           | 9.30  |  |  |  |  |  |  |  |
| 26.00                |           | 9.25  |  |  |  |  |  |  |  |
| 27.00                |           | 9.22  |  |  |  |  |  |  |  |
| 28.00                |           | 9.20  |  |  |  |  |  |  |  |
| 29.00                |           | 9.17  |  |  |  |  |  |  |  |
| 30.00                |           | 9.15  |  |  |  |  |  |  |  |
| 31.00                |           | 9.14  |  |  |  |  |  |  |  |
| 32.00                |           | 9.12  |  |  |  |  |  |  |  |
| 33.00                |           | 9.11  |  |  |  |  |  |  |  |
| 34.00                |           | 9.10  |  |  |  |  |  |  |  |
| 35.00                |           | 9.08  |  |  |  |  |  |  |  |
| 36.00                |           | 9.08  |  |  |  |  |  |  |  |
|                      |           |       |  |  |  |  |  |  |  |

| Lime turned ON                        |
|---------------------------------------|
| Lime turned OFF/Beautiful Green color |

**Observation** Stable Dark Green Lime turned ON

Lime turned OFF pH dropping

Lime turned ON

Lime turned OFF

Lime turned ON

Lime turned OFF

## Experiment Terminated

|                       | Al    | As    | Ca  | Cd    | Cr    | Cu    | Fe    | Mn    | Ni    | $SO_4$ | Zn    |
|-----------------------|-------|-------|-----|-------|-------|-------|-------|-------|-------|--------|-------|
|                       | ppm   | ppm   | ppm | ppm   | ppm   | ppm   | ppm   | ppm   | ppm   | ppm    | ppm   |
| Exp 1 CO <sub>2</sub> | 0.371 | 0.002 | 453 | 0.03  | U     | 0.048 | 0.047 | 13.38 | 0.005 | 1,905  | 0.448 |
| Exp 1 No CO2          | 0.067 | 0.003 | 429 | 0.031 | U     | 0.021 | 0.031 | 13.18 | 0.004 | 1,947  | 0.182 |
| Stored Pit Water      | 277   | 0.058 | 437 | 2.247 | 0.230 | 188   | 773   | 205   | 1.269 | 7,284  | 613   |

Table B-3. Comparison of Compositions of CO<sub>2</sub> and Non CO<sub>2</sub> Treated Filtrates from Preliminary Experiment 1 and Untreated Stored Pit Water

SO4 calculated from ICP estimation of sulfur

|                 | iic Puilip Log—Expe |       |                                  |
|-----------------|---------------------|-------|----------------------------------|
| Elapsed in Mins | Mark Time           | рН    | Observation                      |
| 0.00            |                     | 2.47  | Lime turned ON                   |
| 1.00            |                     | 3.05  | Yellow/Brown precipitate forming |
| 2.00            |                     | 3.95  | Yellow/Brown precipitate forming |
| 3.00            |                     | 4.44  | Yellow/Green precipitate forming |
| 4.00            |                     | 4.83  | Green/Yellow precipitate forming |
| 5.00            |                     | 5.39  |                                  |
| 6.00            |                     | 5.80  |                                  |
| 6.54            | 6.54                | 6.17  | Lime turned OFF                  |
| 7.00            |                     | 6.17  | Olive Green precipitate formed   |
| 8.00            |                     | 6.41  | Lime turned ON                   |
| 9.00            |                     | 6.90  |                                  |
| 9.30            | 9.30                | 7.39  |                                  |
| 10.00           |                     | 7.88  |                                  |
| 11.00           |                     | 9.00  | Lime turned OFF                  |
| 12.00           |                     | 9.28  |                                  |
| 13.00           |                     | 9.28  | Lime turned ON                   |
| 14.00           |                     | 9.93  |                                  |
| 14.40           | 14.40               | 10.42 | Lime turned OFF                  |
| 15.00           |                     | 10.38 |                                  |
| 16.00           |                     | 10.05 |                                  |
| 17.00           |                     | 9.98  |                                  |
| 18.00           |                     | 9.92  |                                  |
| 19.00           |                     | 9.89  |                                  |
| 20.00           |                     | 9.88  |                                  |
| 21.00           |                     | 9.87  |                                  |
| 22.00           |                     | 9.86  |                                  |
| 23.00           |                     | 9.85  |                                  |
|                 |                     |       |                                  |

#### Table B-4. Peristatic Pump Log—Experiment 2

| 24.00 | 9.84 |
|-------|------|
| 25.00 | 9.83 |

#### Table B-4. Continued

| Elapsed in Mins | Mark Time | pH   | Observation           |
|-----------------|-----------|------|-----------------------|
| 26.00           |           | 9.83 |                       |
| 27.00           |           | 9.82 |                       |
| 28.00           |           | 9.82 |                       |
| 29.00           |           | 9.82 |                       |
| 30.00           |           | 9.81 | Experiment Terminated |

Table B-5. Comparison of Compositions of CO<sub>2</sub> and Non-CO<sub>2</sub> Treated Filtrates from Preliminary Experiment 2 and Untreated Stored Pit Water

|                          | Al    | As    | Ca  | Cd ppm | Cr    | Cu    | Fe    | Mn    | Ni    | $SO_4$ | Zn    |
|--------------------------|-------|-------|-----|--------|-------|-------|-------|-------|-------|--------|-------|
|                          | ppm   | ppm   | ppm |        | ppm   | ppm   | ppm   | ppm   | ppm   | ppm    | ppm   |
| Exp 2 CO <sub>2</sub>    | 0.329 | U     | 704 | 0.004  | U     | 0.021 | 0.016 | 0.637 | 0.014 | 1,962  | 078   |
| Exp 2 No CO <sub>2</sub> | 0.245 | U     | 734 | 0.004  | U     | 0.011 | U     | 0.645 | 0.011 | 1,800  | 0.048 |
| Stored Pit Water         | 277   | 0.058 | 437 | 2.247  | 0.230 | 188   | 773   | 205   | 1.269 | 7,284  | 613   |

SO4 calculated from ICP estimation of sulfur

#### Table B-6. Peristatic Pump Log—Experiment 3

| -           |       | Observation                  |
|-------------|-------|------------------------------|
| 0.00        | 2.62  | Lime turned ON               |
| 1.00        | 3.12  | Yellow color                 |
| 2.00        | 3.78  | Golden Yellow                |
| 3.00        | 4.44  | Yellow/slight tinge of Green |
| 4.00        | 4.70  | Yellow/Green tint            |
| 5.00        | 5.02  | Greenish brown               |
| 6.00        | 5.48  | Greener                      |
| 7.00        | 5.85  | Greener                      |
| 8.00        | 6.15  | Yellow Green                 |
| 9.00        | 6.63. | Olive Green                  |
| 9.37 9.37   | 7.04  | Lime OFF                     |
| 10.00       | 7.09  |                              |
| 11.00       | 7.57  |                              |
| 12.00       | 7.73  | Lime ON                      |
| 12.56 12.56 | 8.74  | Lime OFF                     |
| 13.00       | 8.74  |                              |
| 14.00       | 9.07  |                              |
| 15.00       | 8.98  | Lime ON                      |

| 15.42 | 15.42 | 9.59 | Lime OFF |
|-------|-------|------|----------|
| 16.00 |       | 9.59 |          |
| 17.00 |       | 9.34 |          |
| 18.00 |       | 9.21 | Lime ON  |

#### Table B-6. Continued

| Elapsed in Mins | Mark  | рН   | Observation           |
|-----------------|-------|------|-----------------------|
| 18.42           | 18.42 | 9.81 | Lime OFF              |
| 19.00           |       | 9.80 |                       |
| 20.00           |       | 9.47 |                       |
| 21.00           |       | 9.34 |                       |
| 22.00           |       | 9.27 |                       |
| 23.00           |       | 9.23 | Lime ON               |
| 23.30           | 23.30 | 9.88 | Lime OFF              |
| 24.00           |       | 9.80 |                       |
| 25.00           |       | 9.57 |                       |
| 26.00           |       | 9.47 |                       |
| 27.00           |       | 9.41 |                       |
| 28.00           |       | 9.37 |                       |
| 29.00           |       | 9.35 |                       |
| 30.00           |       | 9.33 | Experiment Terminated |
|                 |       |      |                       |

Table B-7. Comparison of Compositions of CO<sub>2</sub> and Non-CO<sub>2</sub> Treated Filtrates from Preliminary Experiment 3 and Untreated Stored Pit Water

|                       | Al    | As    | Ca  | Cd    | Cr    | Cu    | Fe    | Mn    | Ni    | $SO_4$ | Zn  |
|-----------------------|-------|-------|-----|-------|-------|-------|-------|-------|-------|--------|-----|
|                       | ppm   | ppm   | ppm | ppm   | ppm   | ppm   | ppm   | ppm   | ppm   | ppm    | ppm |
| Exp 3 CO <sub>2</sub> | 0.435 | U     | 672 | U     | 0.006 | 0.064 | 0.037 | 2.227 | U     | 2,223  | U   |
| Exp 3 No CO2          | 0.301 | U     | 681 | U     | 0.006 | 0.055 | 0.111 | 2.136 | U     | 2,331  | U   |
| Stored Pit<br>Water   | 277   | 0.058 | 437 | 2.247 | 0.230 | 188   | 773   | 205   | 1.269 | 7,284  | 613 |

SO<sub>4</sub> calculated from ICP estimation of sulfur