Controlling Mine Drainage Problems-
New Approaches for Waste Rock Stockpiles

A Final Report to the Minerals Coordinating Committee

June 30, 2003

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Division of Lands and Minerals
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0. EXECUTIVE SUMMARY

Four field test piles containing Archean greenstone rock (38 cubic meters ~ 63 tons) were constructed to provide data to aid in predicting drainage quality from greenstone waste rock. The four piles were constructed from 21 July to 19 September 2000 and had sulfur contents of 0.02%, 0.20%, 0.39% and 0.67%. The piles were instrumented to provide data on the temperature and gas composition within the pile, collect drainage from the piles, and determine the amount of drainage from the piles. Six tanks containing three different mixtures of rock and limestone were also used to examine the effectiveness of limestone addition in limiting release of acid from rock with a sulfur content of 0.49%. Funding for the construction was provided by the Minnesota Department of Natural Resources Division of Lands and Minerals (MN DNR).

Rock samples were characterized for particle size and chemistry. Roughly 75% of the rock in test piles 1 and 2 was coarser than 3/4-inch, as compared to about 55% of the rock in test piles 3 and 4. The -100 mesh fraction for the four piles ranged from 0.8% to 1.9%. Drainage quality data were collected for only one to three months in 2000. All drainages were in the circumneutral pH range, with elevated concentrations of sulfate. Due to problems with flow measurement, no rates of chemical release were determined. Funding for the initial solid-phase characterization and drainage quality analyses was provided by the Minerals Coordinating Committee (MCC), Minnesota Environmental Cooperative Research Program, and MN DNR Division of Lands and Minerals (Lapakko et al. 2001).

The Minerals Coordinating Committee provided funding to continue field experiments from 1 July 2001 to 30 June 2003, the period addressed in the present report. The objectives of this phase were to 1) conduct additional analyses on the rock used in field tests, 2) continue field scale predictive tests on Archean greenstone waste rock samples to evaluate the effects of solid-phase composition and time of reaction on drainage quality, and 3) continue investigating the feasibility of adding fine grained limestone to acid generating Archean greenstone waste rock to control acid release with drainage from the rock.

Environmentally sound management of waste rock generated by mining must consider the quantity and quality of drainage generated by waste rock piles. The quantity of drainage generated by waste rock piles is a function of physical properties of the rock and climatic conditions. Drainage quality from waste rock is a function of solid-phase composition, reaction conditions, and time. The solid-phase, gaseous-phase, and temperature data collected in the present study will provide essential data on the reactants present and the reaction conditions in the field. This information will aid in the comparative analysis of laboratory and field results and extrapolation of results to operational conditions.

Water yield coefficients changed substantially from 2001 to 2002, and the 2002 values are more consistent with previously reported data. Additional data collection will further increase confidence in these values. Drainage quality data suggests that sulfide oxidation rates in the 0.39% and 0.67% sulfur piles are increasing. If this trend continues, these drainages may acidify in the upcoming years. Additional data on drainage quantity and quality are necessary to evaluate the long-term

behavior of waste rock in the environment. These data, in conjunction with laboratory data, will provide considerable insight into the long-term behavior of operational-scale waste rock. This insight will allow for the efficient and environmentally effective management of these waste rock from mining operations in the Archean greenstones.

Chemical analyses were conducted to determine the variation of chemistry as a function of particle size. Sulfur contents in the 0.02% and 0.39% sulfur piles tended to increase as particle size decreased, and in the 0.67% S pile tended to decrease as particle size decreased. In the 0.20% S pile, sulfur content was elevated in the +1/4-inch fractions and relatively constant in the finer fractions. The carbon dioxide content of all piles increased as particle size decreased, indicating any acid-neutralizing carbonate minerals were concentrated in the fine fractions. Analyses were also conducted to determine the mineral content of the four piles, chemistry of individual minerals, and the degrees of sulfide and carbonate mineral liberation. The degrees of liberation are key factors influencing, respectively, rates of acid production and acid neutralization. The mineralogical analyses were not completed in time for the present report and will be presented in the future.

Measurement of the gas-phase composition within the piles in 2000 suggested that oxygen was not substantially depleted within the piles in 2000. Measurements in 2001 and 2002 indicated that oxygen content of the gas phase in the piles was essentially the same as that in the atmosphere. The temperature within the piles was determined concurrently with gas-phase composition and ranged from -2 to -23°C. It should be noted that no measurements were made between 13 December and 27 March.

Drainage volumes in 2001 ranged from 73 to 85 percent of the input precipitation, which was the only input to the piles. In contrast, drainage volumes in 2002 ranged from 51 to 67 percent of the input precipitation, and this range is more consistent with previously reported values.

Drainage pH values from all piles typically ranged from 7.5 to 8.0 over the course of the study, and concentrations of trace metals (Cu, Ni, Co, Zn) were typically less than 0.02 mg L^{-1}. Sulfate concentrations were elevated in drainages from all four piles, indicating that the pyrite present was oxidizing and, consequently, acid was being produced. The neutral drainage pH values indicate that the acid produced was neutralized by other reactions within the piles. Dissolution of calcium bearing minerals neutralized the majority of acid from all but the 0.02% S pile, in which sodium bearing minerals dominated acid neutralization. From 2001 to 2002, rates of sulfate release from the two low sulfur piles decreased by roughly 25 percent, and rates of sulfate release from the two high sulfur piles increased by 60 to 70 percent. This suggests that rates of sulfide oxidation, and attendant acid production, in the two higher sulfide piles are increasing. Furthermore, this may indicate that drainage from these piles may acidify in the future. The first two samples in 2003 indicate the pH of drainage from the two high sulfur piles remains in the range observed in 2001 and 2002.

Fine grained limestone (manufactured sand) was added to Archean greenstone rock (0.49% S, 0.39% CO_2, NP_{Sobek} = 6.3 g $CaCO_3$ eq (kg rock)$^{-1}$) obtained from Soudan State Park. The mixtures each weighed approximately 3500 pounds and were contained in polyethylene tanks (d = 48 in, h = 42 in).

The acid neutralization potential to acid production potential ratios (NP_{Sobek}:$AP(S_T)$) of the controls and two treatments, each of which were run in duplicate, were 0.41, 1.4:1, and 2.5:1, respectively.

In 2001 the volume of drainage from the tanks ranged from 85 to 98 percent of the input precipitation, which was the only input to the tanks. In 2002 the observed range decreased to 48 to 53 percent. As mentioned in the previous discussion, this is more consistent with previously reported values.

After two full years of operation, drainage from the controls and treatments remains in the circumneutral range. Drainage pH from the controls is on the lower end of the observed range, and several values below 7.5 have been observed. Drainage pH values from the rock to which limestone was added have typically ranged from 7.7 to 8.4. Concentrations of alkalinity in the drainages tended to increase with limestone loading. The pH and alkalinity trends indicate limestone dissolution is affecting drainage quality. Acidification of the control tanks in the future will be necessary to provide an assessment of the practical effectiveness of this treatment.

1. INTRODUCTION

Greenstone belts are hosts to numerous gold and base metal deposits. There is presently no mineral development in the Archean greenstone belts of northern Minnesota. However, these greenstone belts extend north and northeast into Ontario, where a number of gold and base metal mines are located. Due to the promising mineral potential of Minnesota's greenstone belts, there are presently 37 state metallic mineral exploration leases covering more than 13,000 acres in these areas. There are also private metallic mineral exploration leases, although the number and extent of these leases are not public information.

The Minnesota Department of Natural Resources (MN DNR) is charged with both encouraging mineral resource development and protecting other natural resources, including water quality. If mineral development occurs in Minnesota's greenstone belts, characterization and dissolution testing of mine wastes will be necessary to determine the quality of drainage that would be generated. Rock that would not impair water quality, or have other deleterious environmental effects, could be put to productive use, in construction for example. Rock with potential to adversely affect water quality would require mitigative measures to prevent such impacts.

Determination of the quality of drainage from a mine waste is a complex process and can require subjecting a variety of samples from a specific rock type to long-term dissolution testing. To gain insight into the quality of drainage from Archean greenstone waste rock a laboratory dissolution study was initiated in January 2000. Fourteen samples were characterized (particle size, chemistry, mineralogy) and subjected to laboratory dissolution testing for 100 weeks (Lapakko et al. 2002). Sulfur contents of the samples ranged from 0.04% to 1.22% and carbonate was detected in only two samples. Samples for which sulfur content did not exceed 0.16% sulfur produced drainage pH values above 6.0, a common water quality standard, and samples containing 0.20% sulfur or more produced drainage pH values below 6.0. Rates of pyrite oxidation were calculated using sulfur content as a function of particle size and degree of sulfide mineral liberation to determine pyrite surface area. These rates were in good agreement with published rates.

Samples used in the laboratory studies were collected during excavation of a cavern, unrelated to mining, in the greenstone formation near Soudan, MN (www.hep.umn.edu/minos). Rock from this excavation was also collected to construct four field test piles of variable sulfur contents. These piles will provide field data for correlation of laboratory data. Additional samples were collected to fill six tanks. These tanks were used to examine the effectiveness of blending limestone with acid-producing rock to control generation of acidic drainage. These two studies were initiated in the second half of 2000 and data generated through 2001 are described in this report.

It is important to note that the major mineral components of greenstone rocks vary even within the state of Minnesota. Furthermore, the samples used in this study were not taken from an area with economic levels of base or precious metals. Consequently, trace elements present in the samples tested do not simulate those commonly associated with economic ore deposits. Such mineralogical and chemical variations must be considered when applying data from this study to other areas in greenstone belts.

2. OBJECTIVES

The objectives of the field studies using Archean greenstone rock are as follows.

1. Determine the variation of field drainage pH and pyrite oxidation rates with solid-phase sulfur content of greenstone rock samples and dissolution time.

2. Compare the relationship between solid-phase composition of drainage quality in the field with that observed in the laboratory.

3. Determine, as a function of limestone loading, the effectiveness of limestone addition to waste rock in controlling acid generation.

3. BACKGROUND

3.1. Hydrology of Waste Rock Piles

The hydrologic focus of the present study is limited to amount of drainage from a waste rock pile relative to the amount of precipitation falling on the pile. This can be presented as a yield coefficient, which is the ratio of drainage volume from a waste rock pile to the volume of precipitation onto the pile. Two uncovered waste rock test piles in northeastern Minnesota had average yield coefficients over six years of 0.44 and 0.58 (Eger and Lapakko 1985). More recently Smith and Beckie (2003) reported a yield coefficient of 0.55 for a test pile in northern Saskatchewan.

3.2. Mine Waste Dissolution

3.2.1. Acid Production

The major water quality concern regarding mine waste drainage quality is generation of acidic drainage, although release of metals in neutral drainage can also adversely impact water quality. Acid is released as a result of the oxidation of iron sulfide minerals (equation 1), which are common in both hydrothermal quartz carbonate gold deposits and base metal deposits in greenstones.

$$FeS_2\,(s) + (15/4)\,O_2 + (5/2)\,H_2O = FeOOH(s) + 2SO_4^{2-}\,(aq) + 4H^+\,(aq) \qquad [1]$$

Williamson and Rimstidt (1994) used literature data (Smith and Shumate 1970, McKibben 1984, Nicholson et al. 1988, Moses and Herman 1991) to derive the rate law for the abiotic rate of pyrite oxidation by oxygen at 25°C, defined by the equation

$$dFeS_2/dt = 10^{-8.19\,(\pm 0.10)}\, m_{DO}^{\,0.5\,(\pm 0.04)} m_{H+}^{\,(-0.11\pm 0.01)} \qquad [2]$$

where, m_{DO} and m_{H+} are molalities of dissolved oxygen and H^+ in units of mol kg^{-1}, and where the rate of pyrite destruction is expressed in mol m^{-2} s^{-1}. Ranges of m_{DO} and pH for which the expression is applicable are approximately $10^{-5.5}$ to $10^{-1.5}$ and 2 to 10, respectively. For oxygen saturation at 25°C at pH 3 and pH 7, this yields respective rates of 2.2×10^{-10} and 6.2×10^{-10} mol m^{-2} s^{-1}.

In the environment the rate of sulfide mineral oxidation increases as pH decreases into a range conducive to bacterial mediation of ferrous iron oxidation. Nordstrom (1982) reported that as pH decreases to 4.5, ferric iron becomes more soluble and begins to act as an oxidizing agent. As pH further decreases, bacterial oxidation of ferrous iron becomes the rate limiting step in the oxidation of pyrite by ferric iron (Singer and Stumm 1970), which is the only significant oxidizing agent in this pH range (Nordstrom 1982; Singer and Stumm 1970; Kleinmann et al. 1981). The bacterially mediated rate of pyrite oxidation by ferric iron is roughly two to three orders of magnitude faster than the abiotic oxidation by oxygen at pH 2 (Nordstrom and Alpers 1999). In laboratory experiments conducted on hydrothermal quartz carbonate tailings (Lapakko and Wessels 1995) the sulfate release rate from pyrite in the pH range of 3.0 to 3.2 was approximately 13 times that at pH 8 (MN DNR 2000).

3.2.2. Acid Neutralization

Some or all of the acid generated as a result of iron sulfide oxidation may be neutralized by dissolution of other minerals present in a mine waste. Calcium and magnesium carbonates are the most effective of these neutralizing minerals and may be associated with greenstone ore deposits. Calcite ($CaCO_3$) is the most reactive carbonate, with a reported dissolution rate of approximately 2.4×10^{-3} mol m^{-2} s^{-1} at pH 6 ($p_{CO2} = 0.1$ atm, 25°C; Busenberg and Plummer 1986). Relative to calcite dissolution at pH 6, siderite dissolution under anoxic conditions is about three orders of magnitude slower (Greenberg and Tomson 1992). Dissolution of silicate minerals will also neutralize acid, but this dissolution is much slower than that of calcium and magnesium carbonates.

Chlorite, sericite and albite are three silicate minerals that can occur in greenstones. Chlorite is commonly associated with greenstones, forming as a result of low-temperature metamorphism (Klein and Hurlbutt 1985), and is one of the minerals that lend the color for which greenstones are named (Bayly 1968). Sverdrup (1990) presented the following equations as a possible stoichiometry for the initial protonation of the chlorite surface and the reaction of the partially protonated surface.

$$Mg_3Fe_3AlSi_3O_{10}(OH)_7 + 11.2\ H^+ \rightarrow 3\ Mg^{2+} + 2.6\ Fe^{2+} + H_{11.2}Fe_{0.4}AlSi_3O_{10}(OH)_7 \quad [3]$$

$$H_{11.2}Fe_{0.4}AlSi_3O_{10}(OH)_7 + 2.8H^+ \rightarrow Al^{3+} + 0.4Fe^{2+} + 7H_2O{\bullet}H_7Si_3O_{10}^- \quad [4]$$

A chlorite dissolution rate of 7.6×10^{-13} mol m^{-2} s^{-1} (25°C, pH 5) was calculated using the chlorite composition and rate of base metal cation release reported by Sverdrup (1990). May et al. (1995) reported a chlorite dissolution rate of 3.0×10^{-13} mol m^{-2} s^{-1} based on silica release for the same reaction conditions. Malmström et al. (1996) used magnesium release to determine a rate of 5.8×10^{-13} mol m^{-2} s^{-1} after about 25 days of dissolution at 25°C and pH 8.2. The rate after three days of dissolution was about 2.8 times this value. The order of the rate with respect to $[H^+]$ over the approximate pH range of 3 to 5 was reported as approximately 0.5 by May et al. (1995) and 0.7 by Sverdrup (1990).

Sericite is a fine-grained muscovite ($KAl_2(AlSi_3O_{10})(OH)_2$), which has a specific gravity of 2.76 to 2.88, a hardness of 2 to 2.5 (Klein and Hurlbutt 1985) and a reported surface roughness factor of 71 (Nickel 1973). Rates of dissolution have been reported based on observed release of its component elements. Reported rates (pH 5 - 5.6, 22-25°C) range from 1.2×10^{-14} to 1.7×10^{-12} mol m^{-2} sec^{-1}, with four of the six values ranging from 1×10^{-13} to 2.4×10^{-13} mol m^{-2} sec^{-1} (Nickel 1973; Lin and Clemency 1981; Stumm et al. 1987; Kalinowski and Schweda 1996). The dependence of the rate on pH was reported as 0.1 by Nickel (1973, pH 0.2-5.5), 0.08 by Stumm et al. (1987, pH 3-5), and 0.2 by Kalinowski and Schweda (1996, pH 1-4).

Albite is a sodium silicate ($NaAlSi_3O_8$) with a specific gravity of 2.62 and a hardness of 6 (Klein and Hurlbutt 1985). Blum and Stillings (1995) reported the surface roughness factor of freshly ground and washed feldspars averaged 9 ± 6, based on data from Blum (1994). Blum and Stillings (1995) compiled published data on albite dissolution (Chou 1985; Chou and Wollast 1985 and Sverdrup 1990) and expressed it in the form

$$\log \text{rate} = \log k_{H+} - n\text{pH} \qquad [5]$$

The values of k_{H+} ranged from -9.66 to -9.5 and n from 0.49 to 0.5. For application in the present study, values of -9.67 and 0.5 were chosen for k_{H+} and n. These represent the averages of values reported by Chou (1985) and Chou and Wollast (1985).

Mine waste drainage will acidify if the rate of acid production exceeds the rate of acid neutralization. In the present study acidification is considered to occur when pH decreases below 6.0, a common water quality standard in the United States. Equation 2 implies a pyrite oxidation rate of 4.8×10^{-10} mol m^{-2} s^{-1} at pH 6 and an associated rate of acid production of 1.9×10^{-9} mol m^{-2} s^{-1}. The rate of acid neutralization by calcite is roughly six orders of magnitude faster, while that by siderite under anoxic conditions is about three orders of magnitude faster. In contrast, the rates of chlorite, sericite and albite dissolution at pH 6.0 are on the order of 1×10^{-13} mol m^{-2} s^{-1}. This yields a rate of acid neutralization roughly four orders of magnitude lower than the rate of acid production by pyrite oxidation at pH 6. Thus, in a system containing only pyrite and these silicate minerals, the silicate mineral surface area must be roughly four orders of magnitude higher than that of pyrite in order to maintain pH in excess of 6.0.

3.3. Blending Alkaline Solids with Acid-Producing Rock

Acid release from reactive mine waste may be decreased by the mixing of alkaline solids with the rock. The alkaline solids neutralize acid produced by the oxidation of sulfide minerals. This neutralization has three secondary effects. First, the elevated pH yields an environment which is unsuitable for *Acidithiobacillus ferrooxidans*, a strain of bacteria which catalyzes sulfide mineral oxidation. The elimination of these bacteria limits the rate of sulfide oxidation, and therefore, the rate of acid production. Second, the elevated pH enhances the oxidation of ferrous iron and the subsequent precipitation of ferric oxyhydroxides. If the pH is elevated in the immediate neighborhood of iron sulfide mineral surfaces, precipitates will form on the mineral surface. This would impede chemical transport to and from the iron sulfide mineral surface, and consequently, inhibit iron sulfide oxidation and the attendant acid production. Third, as pH increases the equilibrium concentrations of trace metals decrease. The decrease in concentrations is due to increased trace metal precipitation (as hydroxides, oxides, and/or carbonates) and adsorption.

Laboratory data have been generated on drainage quality from sulfidic mine wastes containing naturally-occurring calcium and magnesium carbonates. Finely-crushed Duluth Complex rock containing naturally occurring calcite was subjected to laboratory dissolution. An NP:AP ratio of 0.8 was determined using the calcite and sulfur contents (3% and 1.17%, respectively), and the sample produced neutral drainage over a period of 33 weeks of wet/dry cycle leaching (Lapakko 1988). However, over a longer period the calcium carbonate may have been depleted or rendered ineffective by precipitate coating. If this occurred, and iron sulfide minerals remained and oxidized, the drainage would have become acidic. Such depletion and acidification was reported after a period of 122 weeks for pyritic tailings (5% sulfide) containing 1.4% calcite (Lapakko and Wessels 1995).

In mitigation design the balance between acid production and acid neutralization is affected by the amount of alkaline solids added relative to the amount of iron sulfide present. This is often expressed as the neutralization potential:acid production potential ratio, or NP:AP. The alkaline solids requirement can be estimated based on theory or empirical evidence. Calculation of the acid-producing sulfur content should be based on sulfur associated with iron sulfide minerals (and alunite-jarosite minerals, if present). The theoretical alkalinity requirement can be calculated assuming that each mole of sulfur associated with iron sulfides produces two moles of acid (H^+, reaction 1). It can also be assumed that each mole of calcium carbonate consumes one or two moles of acid. It is necessary to assume how much of the AP and NP will react. The neutralization provided by host rock minerals has also been used to calculate the loading of alkaline solids required (Lapakko et al. 1997).

$$CaCO_3(s) + 2H^+(aq) = H_2CO_3(aq) + Ca^{2+}(aq) \qquad [6]$$

$$CaCO_3(s) + H^+(aq) = HCO_3^-(aq) + Ca^{2+}(aq) \qquad [7]$$

Analysis of field data from coal mining areas, in conjunction with consideration of aqueous carbonate equilibria, suggests that one mole of calcium or magnesium carbonate will neutralize one mole of acid (reaction 7; diPretorio and Rauch 1988; Cravotta III et al. 1990; Brady et al. 1990). diPretorio and Rauch (1988) found that neutral drainage was consistently produced by coal seams containing "greater than 40 tons $CaCO_3$ equivalent of total NP per thousand tons of overburden." This value was subsequently modified to 30 tons/1000 tons "with fizz" by Brady and Hornberger (1990). The "with fizz" provision was added to ensure than the NP was present as calcium and magnesium carbonates as opposed to iron carbonates.

Numerous studies have been conducted on the application of alkaline materials to neutralize acid released from reactive mine wastes. With fine-grained mine wastes, such as tailings, and alkaline solids a homogeneous mixture can be more readily attained and, due to more uniform particle sizing, flow tends to be more uniform. These factors may be more conducive to neutralization of acid generated by sulfide-bearing mine wastes.

Studies have been conducted to examine the effectiveness of adding limestone (Lapakko et al.1997) and rotary kiln fines (Lapakko et al. 2000) to fine-grained ($0.053 < d \leq 0.149$ mm) acid producing Duluth Complex rock. Addition of limestone elevated drainage pH and alkalinity and reduced the rate of iron sulfide oxidation during the 397-week period of record. Drainage remained circumneutral even following the depletion of limestone because host rock mineral dissolution was adequately rapid to neutralize acid produced at the slower rate of iron sulfide oxidation. Addition of rotary kiln fines also elevated drainage pH and alkalinity and reduced the rate of iron sulfide oxidation. However, once these alkaline solids were depleted, drainage acidified and sulfate concentrations increased.

Factors other than NP:AP ratios may determine if mine waste drainage is maintained in the neutral range. With waste rock, the blending and layering of acid-neutralizing solids are reported to be of minimal mitigative success, due to problems such as inadequate homogeneity of mixtures and preferential flow through acid-generating layers (Mehling et al. 1997). The large particle sizes of waste rock (the dimensions of which can reach several feet) and the acid-neutralizing solids most likely contribute to these problems. Analyses by Kempton et al. (1997) and Morin and Hutt (2000) indicate that preferential flow has a dominant influence on the effectiveness of waste rock blending. The latter publication indicated that waste rock drainage acidity is dependent on the flow path length within acid neutralizing rock separating zones of acid generating rock. The authors' analysis indicated that waste rock with a bulk NP:AP ratio of 300:1 could release acidic drainage if appropriate neutralizing rock flow path length was not attained.

Day (1994) concluded that limestone ($d < 0.6$ mm) mixed with acid producing rock (2.1% sulfur) should provide neutralization potential at least twice the acid-producing potential of the rock in order to ensure neutral drainage. (It should be noted that MEND (1994) indicates that 60 percent of the limestone was finer than 0.6 mm, and that 81.9 percent of the rock was finer than 3.35 mm.) Day (1994) further noted that iron precipitate coatings did not reduce the availability of the limestone during the 5-year column study.

In column experiments lasting a total of 24 weeks, O'Hagan (1986) found that a 5 percent $CaCO_3$ addition was required to neutralize drainage from shale with 1 percent sulfur and 0.7 percent pyritic sulfur. The 2.0 to 5.6 mm limestone and shale particles were well blended. Rose and Daub (1994) conducted a 15-week column experiment, and concluded that the 2 to 5 mm limestone particles mixed with 7.0%-sulfur pyritic shale ($d < 1$ cm; $NP/AP = 1$) were ineffective because they did not impart adequate alkalinity to the pore water. This ineffectiveness was hypothesized as being due to their large size and observed coating by iron precipitates.

Donovan and Ziemkiewicz (1994) added limestone layers to 400-ton piles of sandstone and shale coal overburden roughly one to eight inches in diameter, to produce limestone contents of 0.46, 1.07, and 1.26% ($0.56 < NP/AP < 2.38$). The authors concluded that these and other layered alkaline additions did not consistently affect drainage quality during the year after construction "due to their inhomogeneous distribution, to heterogeneity in NP or MPA [Maximum Potential Acidity] within the piles, to hydroxide armoring of the amendment layers, or to time lag in reaching the outflow." The site was resampled 11 years later and drainage from the two higher limestone additions was "circumneutral" (Ziemkiewicz and Meek 1994).

In summary, the main problem in the field has been achieving a good mixture of the alkaline material with the waste rock and problems of preferential flow within large waste rock stockpiles. Most applications at metal mines have either layered acid producing with acid consuming rocks, or tried to blend materials by dumping alternate loads of acid consuming and acid producing material. The problem is that with the layered approach, acid is generated within the acid producing layer and due to preferential flow is not completely neutralized by the acid consuming layer. Similar problems occur in the approach where the loads are dumped in an alternate manner. The challenge is to develop an approach where the acid consuming material is well distributed throughout the pile and in intimate contact with the acid producing material.

Adding limestone to each haul truck as the truck leaves the pit may provide the correct limestone loading and an acceptable level of mixing. This could be accomplished by the truck driving under a hopper where the limestone would be added directly to the top of the load. This type of system is currently used at the Gold Quarry Mine in Nevada to add lime (CaO) to gold bearing sulfide rock to maintain neutral conditions in the leach pad (Bolin et al. 2000). The limestone would begin to mix with the waste rock as the truck drives to the waste dump and then would be further mixed as the material is dumped.

Although visually most stockpiles appear to consist solely of large particles, the interior of these piles contain substantial quantities of fine grained materials. For underground operations, based on the material removed from the AMAX exploration shaft near Babbitt, MN, 38% of the material was less than 1 inch; silt and clay size material comprised about 3% of the mass of the pile (Lapakko et al. 1986). Specific surface area increased from 0.6 $m^2 g^{-1}$ for the coarse sand fraction to 2.6 to 4.7 $m^2 g^{-1}$ for the silt and clay fraction. Sulfur content increased from 0.67% for coarse sand to 1.65-1.94% for the silt and clay sized material. As a result, most of the reactive sulfide surface area of the pile was contained within this fine grained material and generated the majority of the acid in the stockpile. Incorporating a fine grained limestone, in intimate contact with acid producing fines, into the stockpile may provide sufficient contact to neutralize a substantial fraction of the acid production.

4. METHODS

4.1. Prediction Test Piles

4.1.1. Experimental Apparatus

Four 20 ft. x 20 ft. bins to house the rock for the field waste characterization study were constructed using 8 in. x 8 in. x 20 ft. treated timbers stacked two high (Figure 1). The timbers were placed on a compacted sand pad. A one piece 36 mil Reinforced Polypropylene (RPP) liner was placed in the bin and covered with 2 to 4 inches of sand followed by a second one piece 36 mil RPP liner, sloped to one end of the bin, which was covered with six inches of sand (Figures 1, 2). A 11/2 in. slotted PVC pipe covered with a geotextile sleeve was installed between the two liners for use as a leak detection system. A 11/2 in. slotted PVC leachate collection pipe with a geotextile sleeve was placed on the top of the top liner. The pipe ran the entire length of the bin and exited in the center of the bin to a collection sump.

The collection sump as well as flow instrumentation was housed in a 60-gallon polyethylene plastic tank (Figures 3, 4). The leachate flowed into a 7-gallon polyethylene plastic sump equipped with two Madison Co. polypropylene hinged liquid level sensors. When the flow reached the upper sensor, a MARCH model LC-SCP-MD pump was triggered on and pumped the sump down until the water level reached the lower level sensor. The water was pumped through a JLC International Inc. IR-Opflow flow meter and flow was recorded on a Precision Digital model 94788 flow totalizer. A portion of each pump cycle was collected in a 2-liter sample bottle for analyses and the remainder of the flow was pumped to a treatment plant.

Before the rock was loaded into the bins a temperature and oxygen sampling apparatus was placed on the top sand layer (Figure 5). Rock was hand placed over the apparatus to prevent any damage while filling the bins (Figure 6). Test piles 2, 3, and 4 also had the apparatus installed at mid-pile (approximately 3 feet up). The apparatus consisted of a 2 in. PVC pipe ten feet long, which housed a temperature probe. For oxygen sampling a 3/16 inch I.D. plastic Tygon tubing with a 1/4 inch I.D. slotted PVC pipe attached to the end was secured to each side of the pipe. The slotted pipe was covered with a geotextile fabric to prevent plugging from fine rock particles. On one side of the pipe the sampling port was placed at 10 feet and the other at 5 feet.

4.1.2. Materials

4.1.2.1. Excavation

The University of Minnesota initiated a project to enlarge its underground physics laboratory at the Soudan Mine, which resulted in excavation of approximately 22,000 cubic yards of greenstone rock. Prior to excavation a drill hole was bored through the center of the cavern to characterize the rock. The rock was then blasted and removed in four lifts. The explosives used were 75% ANFO, a commercially-prepared combination of ammonium nitrate and fuel oil, and 25% Mine Rite, an ammonium nitrate based water gel. As the rock was removed the walls of the cavern were sealed with shotcrete, a mixture of portland cement containing 11% silica fume and –1/2" aggregate. Rock designated for field dissolution testing was trucked to the MN DNR research site in Hibbing, MN.

During the removal of the top lift of rock at the Soudan Mine, several drill cuttings samples were collected and along with the drill core were analyzed for percent sulfur. Based on these analyses, areas within the bottom three lifts were selected as target zones for obtaining a range of sulfur contents to be used for the field waste characterization tests at the DNR's field research site. Once an identified area had been blasted, approximately 50 cubic yards of rock was removed from the mine in approximately 1.5 cubic yard muck boxes.

4.1.2.2. Pile Construction and Sample Collection

Three types of samples were collected for characterization of rock placed into the prediction bins. First, as each muck box was emptied into a pile a random sample was taken and placed in one gallon plastic containers. Each time an identified area was removed from the shaft, 25 samples were collected and analyzed at Lerch Brothers Inc. (Hibbing, MN) to determine if they would produce a test plot of the desired sulfur content. If the results were suitable the rock was loaded into 10 cubic yard dump trucks and hauled to the research site. The 25 samples were retained and analyzed for total sulfur, sulfate, evolved carbon dioxide, and whole rock chemistry. Thirteen of the samples were analyzed for trace metal content. These analyses were conducted by ACTLABS.

A second sample was collected to determine particle size distribution and the variation of chemistry with particle size. The bins were loaded by placing the rock onto a conveyer, which piled the rock in the center of the bin (Figure 7). The rock was then leveled using a backhoe, forming a truncated pyramid (Figure 8). Based on the dimensions of the truncated pyramid it was determined that the piles contained approximately 39 cubic meters. Based on the bulk density determined for the limestone addition tanks the mass of the piles was estimated to be 63 metric tons. As the rock was loaded onto the conveyer, a random sample (approximately 1 cubic yard) was set aside for analyses of particle size distribution.

A third set of 20 samples was collected to further assess compositional variability, including modes of sulfide mineral occurrence, of rock in test piles 2, 3, and 4. Samples were not collected from test pile 1 since analyses of muck box samples indicated sulfur contents wee relatively uniform. Furthermore, visual examination of the rock revealed no concentrated pyrite occurrences.

These samples were collected as the bins were being loaded. For test piles 2 and 3, four samples were taken from the top of the pile after each 10 yards was added and leveled off, one from each side of the pile. For test pile 4, two samples were collected while the first three feet of rock was being added, eight random samples when the first three feet of rock had been leveled off, six random samples after an additional foot of rock had been added, and four samples when the pile was completed. These samples had not yet been analyzed at the time of this report.

4.1.3. Analytical Methods

4.1.3.1. Solid Phase Analyses

Particle size distribution of the one-cubic meter sample taken during bin construction was determined at the MN DNR. The +12 inch and -12 in./+6 in. fractions were manually measured, removed from the pile and weighed. The remaining sample was shoveled through two stacked screens to remove the -6in./+2.5 in. and -2.5 in./+0.75 in. fractions. Rock passing the 0.75 in. screen was collected in five-gallon buckets. For all four piles this fraction did not exceed three buckets.

One of the buckets was randomly selected, placed on a sheet of plastic and split using the four corners method (Scott 1942). One fourth of the sample (approximately one gallon) was used to determine the size distribution of the -0.75 inch rock using a Gilson Ro-Tap equipped with Tyler standard sieves. Sulfur, sulfide, sulfate, evolved carbon dioxide, whole rock and trace metal chemistry of the various size fractions were determined by ACTLABS using methods described below.

Chemical analyses of rock samples were conducted either by Lerch Brothers Inc. or ACTLABS. Lerch Brothers Inc. (Hibbing, MN) performed the initial sulfur determination on the 25 muck box samples using a LECO combustion furnace (method ASTM E395-95A). The mean of the sulfur determination conducted by Lerch Brothers was used to identify the test piles. The remaining chemical analyses were conducted by ACTLABS Inc. Sulfur, sulfate (sulfide was determined by difference), and evolved carbon dioxide were determined in Tucson, AZ using ASTM E-1915-97 (ASTM 2000). A 10 percent hydrochloric acid solution was used to solubilize the carbonate minerals, and the carbonate present was quantified as the difference between total carbon in the initial sample and that in the residue. The remaining solid-phase constituents were determined by ACTLABS Inc. in Ancaster, ON. Whole rock constituents were determined using a lithium tetraborate fusion modified from ASTM E886-94 (ASTM 2000) and analysis by inductively coupled plasma-atomic emission spectroscopy (ICP-AES) using a Thermo Jarrell-Ash ENVIRO II ICP. Concentrations of Ag, Cd, Cu, Ni, Pb, Zn, and Bi were determined using a total digestion method modified from Crock et al. (1983), with analysis by ICP-AES. Other trace elements were determined using instrumental neutron activation analysis (Hoffman 1992).

Mineralogical analyses are presently being conducted by McSwiggen & Associates in St. Paul, MN and were not completed in time for the present report and will be presented in the future. Analyses includes determining the mineral content of the four piles, chemistry of individual minerals, and the degrees of sulfide and carbonate mineral liberation.

4.1.3.2. Test Pile Drainage

Water input to and output from the test plots were determined. Water input to the piles consisted entirely of precipitation (Tables A2.1, A2.2). Precipitation was collected in a US Standard rain gage at the Hibbing field research site. Flow was recorded on a bi-weekly basis once the piles began to flow. Flow was measured using an IR-Opflow flow meter and was recorded using a Precision Digital model 94788 flow totalizer. During the initial startup of the experiment the flow meters were not functioning due to electrical problems. As a result, flow from 21 July, 2000 to 09 November, 2000 had to be estimated (see attachment A2.2 for details).

Composite **drainage quality** samples were collected on a bi-weekly basis once the piles began to flow. An additional grab sample on the first water that flowed into the sumps of test piles 1 and 4 was also analyzed. Samples were analyzed for specific conductance, pH, alkalinity, and acidity at the MN DNR lab. Specific conductance was analyzed using a Myron L conductivity meter, and an Orion SA720 meter, equipped with a Ross combination pH electrode (8165), was used for pH analyses. Alkalinity (for pH ≥ 6.3) and acidity were determined using standard titration techniques for endpoints of 4.5 and 8.3, respectively (APHA et al. 1992). The remaining sample was filtered for metals and sulfate analysis at MDA. Metal samples were acidified with 0.2 mL of Baker Instra-

Analyzed nitric acid per 50 mL. An additional 500 mL sample acidified with 1.0 mL of Baker Analyzed sulfuric acid was taken for nutrient analyses.

Ca, Mg, Na, and K were determined with a Varian 400 SPECTRAA; inductively coupled plasma mass spectrometry (ICP-MS, Hewlett Packard HP4500 Series, model #G1820A) was used for the remaining metals analyses. Sulfate concentrations were determined using a Lachat QuickChem 8000 or, for $[SO_4] < 5$ mg/L, a Dionex ion chromatograph. Nutrients were analyzed at MDA using the Automated Cadmium Reduction Method (Wastewater Method 4500-NO_3 F) on a Technicon AA11 for Nitrate + Nitrite Nitrogen, the Ammonia-Selective Electrode Method (Wastewater Method 4500-NH_3 F) on an Accumet 950 pH/ion meter for Ammonia Nitrogen, the Ascorbic Acid Method (Wastewater Method 4500-P E) on a Perkin Elmer 552 Spectrophotometer for Total Phosphorus, and the Semi-Automated Colorimetric Method (EPA 351.2) with a Bran&Luebbe Traacs 800 for Total Kjeldahl Nitrogen.

4.1.3.3. Temperature and Oxygen within Test Piles

Temperature was determined using a Cole-Parmer model 8402-00 meter. Oxygen readings were determined using a GC Industries model GC-502 meter and a YSI model 57 as a comparison to verify results. (See attachment A9.3 for temperature and oxygen notes and comparisons.) The method for oxygen sampling consisted of inserting the probe into an oxygen chamber which was connected to a vacuum pump on one end and to the oxygen sampling port on the other. The pump was turned on and the valve to the oxygen port was opened. Oxygen measurements were read after a five minute purge time. Temperature was read at the same time as the oxygen readings. The sampling apparatus is illustrated in Figure 9.

4.1.4. Calculations

Yield coefficients were calculated to express the amount of flow as a fraction of the precipitation falling on the pile.

$$Y_{b,t} = V_{b,t}/[(P_t/12) \times A_b \times 28.2)], \text{ where} \qquad [8]$$

$Y_{b,t}$ = yield coefficient for pile b for flow period t, dimensionless;
$V_{b,t}$ = volume of flow from pile b during flow period t, L;
P_t = precipitation during flow period t, inches (division by 12 converts to feet);
A_b = horizontal cross-sectional area of pile b, ft^2; and
28.2 = factor to convert cubic feet to liters.

Since the base of each pile was 20 feet square, the area A_b can be calculated and inserted into equation 8.

$$Y_{b,t} = V_{b,t}/[940\ P_t] \qquad [9]$$

For periods flow during which flow was not measured from a specific pile (e.g. due to failure of equipment), flow was estimated as the product of the yield coefficient for the pile and the volume of precipitation onto the pile during the period of unmeasured flow.

Mass release during individual flow periods was calculated as the product of the volume of flow and the concentration at the end of the flow period. Mass release during the entire year was calculated as the sum of the releases from the individual flow periods. Rates of release were calculated by dividing mass release for a period by its duration. To determine rates over a longer time frame, the time-weighted average release rate of periods during the time frame was determined.

4.2. Limestone Addition Tanks

4.2.1. Materials

The 0.67% sulfur rock used in test pile 4 of the prediction study was selected for use in the alkaline mixing experiment (see sections 4.1.1. and 4.1.3 for sample collection details). The rock was screened to pass a 1.5 inch screen at Casper Construction Inc. in Grand Rapids, MN (Figures 10 and 11). A bobcat loader was used to fill the tanks with the exception of tank 5 which could not be reached with the loader and was filled using five gallon buckets. Three bucket loads were placed in each tank, the loader positioned the bucket just above the tank and the rock was randomly shoveled into the tanks (Figure 12).

For the tanks that had the rock mixed with limestone, the loader scooped a bucket of rock and then the limestone (1/3 of the total addition), which had been weighed, was added to the bucket (Figures 13, 14 and 15). A sample of rock was taken from each loader bucket, prior to adding the limestone for the alkaline mixing tanks, for a total of 3 samples per tank. A total of 55 pounds of limestone was added to the 1:1 ratio tanks and 165 pounds to the 3:1 ratio tanks.

4.2.2. Experimental Apparatus

The limestone mixing tests are being conducted in six polyethylene plastic tanks (d = 48 in., h = 42 in.). The tanks are housed in a 20' x 20' lined bin that serves as a double containment (Figures 16 and 17). Two control tanks (tanks 1 and 6), two tanks with limestone mixed at a 1:1 ratio (tanks 2 and 5), and two tanks with limestone mixed at a 3:1 ratio (tanks 3 and 4). The tanks were fitted with 2 -inch slotted PVC outlet pipe on the bottom, which drained into a 22 gallon polyethylene plastic sample collection sump (Figure 18). The bottom of the outlet pipe was installed approximately 2 inches above the bottom of the tank which created a zone of saturation, so as not to allow the waste rock to be within this zone three inches of silica sand was placed in the bottom of each tank. The tanks were then filled to a depth of approximately 31 inches with either rock (control tanks) or rock mixed with limestone, yielding a bed volume of 32.4 cubic feet. Bulk density of the rock was subsequently determined as 108.2 lbs/ft^3, yielding a bed mass of 3506 pounds. The limestone had a bulk density of 86.6 lbs/ft^3. The tanks were filled on 24 October, 2000 and input to the tanks is limited to precipitation.

4.2.3. Analytical Methods

4.2.3.1. Solid Phase Analyses

The three rock samples from each tank were composited. A representative split and a sample of the limestone were sent for analyses. Samples were analyzed for total sulfur, sulfate, evolved carbon dioxide as well as whole rock and trace element concentrations at ACTLABS. Total sulfur for the six samples ranged from 0.40% to 0.56%. The limestone used as described by the company was "manufactured sand" and was obtained from CAMAS, Shiely Division in Eagan, MN. Particle size distribution for the rock samples and limestone was determined by Lerch Brothers. Percent moisture of the limestone (5 %) was determined at the MN DNR. See section 4.1.4.1. for analytical methods. The mineralogical composition and pyrite liberation were determined for splits of samples from the two control tanks. Analyses were conducted by Mineralogical Consulting Service, Pengilly, MN (Appendix 1, Attachment A1.1.).

4.2.3.2. Aqueous Analyses

The sample schedule was designed to collect samples on a bi-weekly basis and after selected large rain events. Water input to the tanks will consist entirely of precipitation. The collection sump was calibrated in five liter increments and total flow was measured with a ruler. One inch of precipitation is about the equivalent to 30 liters of input water. A grab sample for analyses was collected directly from the sump and the sump was then emptied. A 250 mL sample was taken for pH, specific conductance, alkalinity (if pH exceeded 6.30) or acidity, metals, and sulfate analysis. These samples were analyzed for pH and specific conductance directly in the bottle. A 20 mL sample was then taken for analysis of alkalinity or acidity. The remaining sample was filtered for metals and sulfate analysis. Metal samples were acidified with 0.2 mL of Baker Instra-Analyzed nitric acid per 50 mLs. Periodic samples for nutrients (500 mLs) were also taken. Nutrient samples were acidified with 2 mLs of Baker Analyzed sulfuric acid per 500 mLs. See section 4.1.4.2. for analytical methods.

5. RESULTS

5.1. Prediction Test Piles

5.1.1. Introduction

Four field test piles were constructed to examine the variation of the quality of drainage from Archean greenstone rock as a function of sulfur content. Construction of individual piles was completed from 21 July to 19 September 2000. These piles provide data for only four sulfur contents, as opposed to the 14 different samples examined in the laboratory.

However, the field tests are more representative of waste rock dissolution under operational conditions. Relative to the laboratory phase, the rock size used in this phase is more representative of that generated during mining and, therefore, more accurately simulates movement of air and water within the rock. In addition, it is being subjected to dissolution under actual environmental conditions. The data generated will be used to aid in extrapolating data from the more intensive laboratory study to field conditions.

5.1.2. Solid-Phase Analyses

Roughly 75% of the rock in test piles 1 and 2 was coarser than 3/4-inch, as compared to about 55% of the rock in test piles 3 and 4 (Table 1). The -100 mesh fraction for the four piles ranged from 0.8% to 1.9%. The respective mean sulfur analyses for test piles 1 - 4 were 0.02%, 0.20%, 0.39%, and 0.67% (Table 2). The major whole rock components (and approximate range of average values) were SiO_2 (52-68%), Al_2O_3 (13-20%), FeO (9-11%), MgO (3-6%), and K_2O (1.7-2.5%). Contents of CO_2 (0.02-0.5%), CaO (0.23-0.77%), and Na_2O (0.2-0.4%) were low (Table 3).

As was the case with laboratory solids, most trace metal concentrations in the field rock were less than 20 mg kg^{-1}. Elements with concentrations above 20 mg kg^{-1} (and their range in mg kg^{-1}) were La (13-30), Nd (15-35), Co (20-40), Ce (30-70), Rb (60-75), Cu (20-110), Zn (80-160), Ni (70-190), and Cr (100-400). Additional data on trace metal contents for all bins and Sobek NP values for Bin 4 are presented in Appendix 1.

The mass-weighted average compositions determined for the particle size samples were typically in close agreement with compositions determined by analysis of the 25 muck box samples (Table 4). This suggests that the one-ton particle size sample was fairly representative of the piles. Notable exceptions to this agreement were the sulfur contents from the two low sulfur bins. In both cases the mass-weighted average compositions for the particle size samples yielded sulfur concentrations more than twice those for the muck box samples. In both cases the sulfur content of each particle size fraction was higher than that of the muck box sample average. It is possible that some anomalously high sulfur rock was included in the random sample collected for particle size analysis.

There were no consistent trends in the variation of sulfur content with particle size. For the 0.02% and 0.39% sulfur piles, sulfur content tended to increase as particle size decreased (Table A1.12). In contrast, the sulfur content in the 0.67% sulfur pile tended to decrease as particle size decreased. For the 0.20% sulfur pile, sulfur content was elevated in the +1/4-inch fractions and fairly stable in the finer fractions.

For all four piles carbon dioxide content tended to increase with decreasing particle size, and the CO_2 content of the -100 fraction was roughly an order of magnitude higher than the mass-weighted mean. In fact, almost all fractions finer than 0.25 inches had CO_2 contents at least three times the mass-weighted mean. Calcium concentrations also increased with decreasing particle size, suggesting (although not conclusively) that a calcium carbonate phase may be present. Consequently, the effectiveness of the fine fraction for neutralizing acid would be enhanced not only by the greater degree of carbonate mineral liberation and elevated specific surface area, but also by the preferential concentration of carbonates in this fraction.

Analyses are presently in progress to determine the mineral content of the four piles, the chemistries of the individual minerals, the variation of mineral content with particle size, and the variation of sulfide and carbonate mineral liberation with particle size.

5.1.3. Temperature and Oxygen Profiles

Variations in temperature and oxygen affect the rate of sulfide mineral oxidation. Temperature and oxygen content within piles 1, 3, and 4 were measured two to four times from 2 August to 26 September 2000. All four piles were sampled eight to thirteen times from 27 March to 17 October 2001 and three times between 15 May and 26 August 2002. Start-up problems were encountered with both sampling and measuring oxygen content (appendix 2, attachment A2.3). Some sampling problems due to obstructed tubing also occurred in test piles 3 and 4 late in 2001 (see appendix 2, attachment A2.3. for field notes).

Temperature was measured from March to December and ranged from approximately -2°C to 23°C. Temporal trends were similar in all piles, with temperatures lowest in March and highest in July and August (Table 5). Temperatures at the bottom of the piles typically ranged from approximately 0°C to 18°C. Piles 2-4 were sampled both at the bottom and approximately three feet from the bottom. The high and low temperatures for the upper sampling ports were roughly 2-4 degrees more extreme than those from the lower ports. The greater degree of insulation at the bottom of the piles probably resulted in more moderate variations in temperature.

Oxygen contents in the gas phase were measured 6 to 15 times at the 14 ports. At two of the ports only six measurements were made because the tubing used for measurements became obstructed. Of the167 measurements, 78% (130 of 167) of the values were in the range of 16% to 21% of the gas phase, as compared with 21% for the oxygen content of the atmosphere. Of the 37 values out of the typical range, 21 were determined on two days. Ten values determined on 27 March 2001 ranged from approximately 7% to 12%, and 11 values determined on 17 October 2001 ranged from roughly 23% to 26%. It is conceivable, if not likely, that these measurements are in error.

Average oxygen concentrations at the 12 ports that functioned throughout the study ranged from 18.1% to 19.8%. Average oxygen concentrations were examined as a function of vertical depth in the pile (top vs bottom port for five and ten foot distances into pile), horizontal distance into pile (five vs ten foot distance for top and bottom port) and sulfur content (top port five feet into pile, top port ten feet into pile, bottom port five feet into pile, bottom port ten feet into pile). Since the range in concentrations was fairly small and some problems were encountered in measurement, the following comparisons are tentative.

In three of four cases, average oxygen concentrations at the top of the pile were 0.1% to 1.1% higher than at the top of the pile. In all four cases assessing horizontal distance into the pile, average oxygen concentrations five feet into the pile were 0.1% to 0.8% higher than those 10 feet into the pile. There was no consistent dependence of oxygen concentrations on sulfur content. Considering the data collected to date it appears oxygen concentrations in gas within all four piles are near atmospheric and the variation of these concentrations within the pile is small. It is unlikely that possible variations of this magnitude will have a substantial effect on rates of sulfide mineral oxidation within the pile. The fact that oxygen concentrations are near atmospheric indicates that rates of sulfide mineral oxidation (per unit sulfide mineral surface area) should be close to those observed in the laboratory.

5.1.4. Flow

The four test piles were constructed late in 2000, and the periods of flow were roughly two to four months. Flow for the first partial year was assumed to cease on 20 November 2000, although a small amount of flow occurred subsequently (Attachment A2.1). Due to equipment problems flow was not determined until November 1 (piles 1-3) or 9 (pile 4), 2000, providing only one month of measured flow for the two to four month records for that year. Yield coefficients (the ratio of drainage volume to input precipitation volume) of 0.50 to 0.63 were calculated for one- to three-week periods after flow meters were installed in 2000. The short period of record over which these values were determined must be considered when evaluating their merit. Flows for the remaining one to three months were estimated using the observed precipitation during the period of unmeasured flow and yield coefficients determined for each pile for the period from August to November 2001 (see Attachment A2.2). The estimated flows ranged from 3700 to 7000 liters for 2000.

In 2001, precipitation was 26.06 inches (Table A2.2) and the four piles produced drainage from the middle of April to the middle of December. Total flows ranged from approximately 17,500 to 20,400 liters. Yield coefficients of 0.73 to 0.85 were calculated and these values are considerably higher than yield coefficients of 0.44 and 0.58 reported by Eger and Lapakko (1985) and 0.55 reported by Smith and Beckie (2003) (Table 6). The yield coefficients for piles 1,3 and 4 from 2 August - 28 November 2001 ranged from 0.50 to 0.63. These yields are in good agreement with previously reported values. The yield coefficient for pile 2 from August to November 2001 was higher (0.81) (Appendix 2, Attachment A2.2).

In 2002, precipitation was 27.21 inches and the four piles produced drainage from the middle of April to the end of November. Total flows ranged from approximately 13,000 to 17,500 liters, representing 51% to 67% of the precipitation input to the piles (yield coefficients of 0.51 to 0.67). The yield coefficients determined for 2002 are more consistent with previously reported values (Eger and Lapakko 1985; Smith and Beckie 2003).

The yield coefficients determined for 2001 seem unusually high when compared to the 2002 values and those reported previously. It is possible that the apparently high yields in 2001 are reasonable because the size of the piles are so small. If so, then a problem exists with the low coefficients observed in 2002. The reason for the large variation in drainage yields between 2001 and 2002 is unclear, especially considering the variation was observed for all four piles. As is disscussed in a later section, this large variation was also observed for the six limestone addition tanks.

Error in the determination of yield coefficients could be the result of errors in 1) measuring precipitation, 2) measuring drainage volume, or 3) error in calculation. Precipitation measurements at the site were in reasonable agreement with those reported for the Hibbing airport. It seems unlikely that errors in measuring drainage volume would occur uniformly at all 10 measurement locations. The same calculation was used for both years. Yield coefficients calculated in the future may lend insight into the large variation observed over the present two-year period of record.

5.1.5. Drainage Quality and Chemical Mass Release

In 2000, three to six drainage quality samples were collected from each pile between 14 August and 20 November. In 2001 (9 April - 28 November) and 2002 (28 April - 28 October), 11 and 10 samples per pile, respectively, were collected. These samples were composites that represented the flow weighted mean drainage quality over a typical period of two to three weeks. Chemical mass release was calculated based on concentrations in test pile drainage and the drainage volume. Rates of release were calculated for 2001 and 2002 by dividing chemical mass release by the time over which release occurred and the mass of the pile.

Actual flows in 2001 were 20 to 30 percent higher than those used to calculate rates. Consequently, chemical mass release and rates of release in 2001 were approximately 20 to 30 percent higher than those presented in this report. In the upcoming year, estimates will be made to incorporate all flow into the 2001 rates of release. The flow volumes used to calculate rates in 2001 were roughly equal to those measured in 2002. Therefore, changes in rates between the two years reflect change in drainage concentrations.

Drainage pH values typically ranged from 7.5 to 8.4 and **alkalinities** from roughly 40 to 80 mgL^{-1} as $CaCO_3$. Drainage pH values from the two lower sulfur piles tended to oscillate in the approximate range of 7.5-8.5, and exhibited no distinct temporal trend over the course of the study (Figures 19, 20). Typical drainage pH ranges for the 0.39% (7.5-7.9) and 0.67% (7.4-8.2) sulfur piles were lower and both displayed a slightly decreasing trend over the 2001 and 2002 field seasons (Figures 21, 22). Alkalinities from the 0.02% and 0.20% sulfur piles tended to oscillate within respective ranges of 40-75 and 60-80 mg $CaCO_3$ L^{-1}, with no distinct temporal trend over the course of the study (Tables A3.1, A3.2, respectively). Alkalinities from the 0.39% and 0.67% piles typically ranged from 45-70 and 40-85 mg $CaCO_3$ L^{-1}, respectively, and tended to be lower during 2002 than in the earlier part of the study (Tables A3.3, A3.4).

Sulfate release reflects acid production (equation 1). Sulfate concentrations from the four piles typically ranged from 20 to 300 mgL^{-1}and generally increased as the sulfur content of the pile increased. All piles exhibited a sulfate concentration peak in 2000, probably due to the release of oxidation products accumulated after the rock was blasted. Concentrations from the 0.02% and 0.20% piles tended to decrease slightly over time and plateau during 2002 (Figures 19, 20). In contrast, sulfate concentrations in drainage from the 0.39% and 0.67% sulfur piles tended to increase over time (Figures 21, 22).

It was assumed that all chemical release was the result of reactions in the pile. Because sulfate concentrations in precipitation can be elevated, data from precipitation monitoring stations in the region were checked to determine their contribution to drainages. For the 2001 calendar year, average sulfate concentrations in precipitation at Marcel, Ely, Wolf Ridge and Fond du Lac were in the range of 0.59 to 0.97 mg L^{-1} (http://nadp.sws.uiuc.edu/nadpdata/state.asp?state=MN). Drainage from the piles was approximately 60 percent of precipitation (Table 6). Assuming sulfate was conserved in solution, the aforementioned concentrations would increase to the range of 1.0 to 1.6 mg L^{-1}. Sulfate concentrations in drainage from the 0.02% S pile were the lowest of the four piles, typically ranging from 17 to 21 mg L^{-1} in 2002. Comparing adjusted sulfate concentrations in the precipitation with those in the drainage, precipitation contributed roughly five to nine percent of the sulfate release from the pile (100 x 1/21 ~ 5, 100 x 1.6/17 ~ 9). The fraction of sulfate release contributed by precipitation for the remaining three piles would be less than this value because sulfate concentrations in drainages from these piles was higher. Sulfate concentrations from precipitation are not considered further in the discussion of sulfate release.

Sulfate release rates calculated for discrete time intervals during 2001 and 2002 ranged over about one order of magnitude for each of the piles (Table 7). These variations were due to variations in drainage volume (Tables A4.1-A4.4) and sulfate concentrations. From 2001 to 2002, rates of sulfate release from the 0.02% and 0.20% sulfur piles decreased by roughly 25%, and this reflects a corresponding decrease in sulfate concentrations (Figures 19, 20). Rates from the 0.39% and 0.67% sulfur piles in 2002 were approximately 65% higher than those in 2001 (Table 8). These increases were largely the result of sulfate concentrations increasing over time (Figures 21, 22).

Rates of sulfate release in 2002 increased linearly with the sulfur content of the rock (Figure 23). The slope of the line presented in Figure 23 was determined to be 32.2 μmol $(\text{kg week})^{-1}$ $(\text{pct S})^{-1}$ (r^2 = 0.98). The slope for a similar graph depicting laboratory data was reported as 224 μmol $(\text{kg week})^{-1}$ $(\text{pct S})^{-1}$ (Lapakko et al. 2002, Figure 21). The ratio of the field slope to that from the laboratory data suggests that sulfate release rates in the field were 0.14 times those in the laboratory.

Retardation factors were also calculated using sulfate release rates from the individual piles. In 2001 and 2002, these values ranged from 0.055 to 0.55 (Table 7), in comparison with values of 0.096 to 0.33 reported for Duluth Complex rock (Lapakko 1994). The retardation factor for the 0.02% S pile was unusually high, particularly because it was compared to a sample with a sulfur content of 0.04% S. It should be noted that the 0.02% S content for this pile was determined based on analysis of 25 muck box samples (Table 2). Sulfur concentrations reported for size fractions of the bulk sample suggest the sulfur content of the pile may actually be in the neighborhood of 0.04% (Table 4, Table A1.12).

Calcium, magnesium, sodium and potassium were released from minerals that dissolved and neutralized acid. Release of one mole of calcium or magnesium represents neutralization of two moles of acid and release of one mole of sodium or potassium represents neutralization of one mole of acid. Concentrations of these metals peaked in 2000 and generally tended to decrease with time (Figures 19-22). Concentrations of calcium, magnesium and potassium tended to increase as the sulfur content of the pile increased, and the opposite trend was observed for sodium.

Molar concentrations generally decreased in the order Ca > Na > Mg ~ K. In contrast, solid-phase concentrations (and their approximate ranges) decreased in the order MgO (3 - 6%) > K_2O (1.5 - 3.5%) > CaO (0.2 - 0.8%) ~ Na_2O (0.2 - 0.5%) (Table 4). High aqueous phase calcium concentrations relative to solid-phase calcium concentrations indicates the solid phase in which calcium was present dissolved relatively rapidly. Conversely, magnesium and potassium concentrations in drainages were relatively low and their solid-phase concentrations were relatively high. These elements were apparently present in mineral phases that dissolved relatively slowly.

Calcium release rates tended to increase with increasing sulfur content. This trend was also observed for sulfate release rates and suggests that calcium release rates were driven by the rate of acid production. Rates of calcium release from the 0.02%, 0.20%, and 0.39% S piles in 2002 were roughly 50% to 75% of those in 2001 (Table 8), reflecting the previously mentioned decrease of calcium concentrations over time (Figures 19, 20). The rate of calcium release from the 0.67% S pile in 2002 was roughly 1.2 times that in 2001, and this indicates calcium concentrations increased slightly from 2001 to 2002. Sulfate release from this pile also increased in 2002, and the increase in calcium release may have been the result of increased acid production.

The decreasing calcium rates observed for the 0.02% and 0.20% S piles suggests there may have been a small amount of a more reactive phase containing calcium, such as fine-grained carbonate minerals or residue from shotcrete used in the Soudan Mine cavern. The carbon dioxide contents of the rock ranged from 0.054 to 0.46, which is higher than values typically observed for the laboratory samples ($CO_2 < 0.05\%$).

Although magnesium release rates in 2001 exhibited no distinct dependence on solid-phase sulfur content, release rates in 2002 strictly increased with sulfur content. Release rates for the 0.02%, 0.20% and 0.39% piles in 2002 were roughly 40 to 60 percent of those in 2001 (Table 8), reflecting a decrease in magnesium concentrations from 2001 to 2002. The rate of magnesium release from the 0.67% S pile in 2002 was the same as that in 2001. The rate of sulfate release from this pile increased in 2002, and magnesium release may have increased, in part, due to increased acid production (as was suggested previously for calcium release).

Sodium release tended to decrease with increasing sulfur content. The average annual sodium release rates in 2002 were roughly 30 to 50 percent of those in 2001 and indicate a pronounced decrease in sodium concentrations. The extent to which sodium release decreased over this period was greater than or equal to that for calcium, magnesium and potassium.

Potassium release demonstrated a mild tendency to increase with increasing solid-phase sulfur content. Annual potassium release from the piles in 2002 ranged from approximately 60 to 90 percent of those in 2001. The extent to which potassium release decreased over this period was generally lower than that for calcium, magnesium and sodium.

Concentrations of **trace metals** (Cu, Ni, Co, Zn) were generally very low. Only copper was detectable (> 0.002 mgL^{-1}) in more than half the cases. Concentrations in 2002 typically did not exceed 0.006 mgL^{-1} and exhibited no distinct dependence on sulfur content of the pile (Tables A3.1-A3.4).

In 2002, iron concentrations were generally below 0.01 mgL^{-1} and tended to increase as the sulfur content of the piles increased (Tables A3.1-A3.4). Manganese concentrations were typically below detection (0.002 mgL^{-1}). Aluminum concentrations were generally below 0.01 mgL^{-1} and were highest in the 0.02% sulfur pile. Ammonia and nitrate concentrations were elevated in 2000, most likely due to the presence of residual blasting agents in the rock, and showed a decreasing trend over time (Tables A3.1-A3.4). This trend suggests the ammonia and nitrate were being removed from the piles and that concentrations would approach zero in the fairly near future.

5.1.6. Comparison of Field and Laboratory Sulfate Release Rates

Rates of sulfate release in 2002 increased linearly with the sulfur content of the rock (Figure 23). The slope of the line presented in Figure 23 was determined to be 32.2 μmol (kg week)$^{-1}$ (pct S)$^{-1}$ ($r^2 = 0.98$). The slope for a similar graph depicting laboratory data was reported as 224 μmol (kg week)$^{-1}$ (pct S)$^{-1}$ (Lapakko et al. 2002, Figure 21). The ratio of the field slope to that from the laboratory data suggests that sulfate release rates in the field were 0.14 times those in the laboratory. Retardation factors calculated using sulfate release rates from the individual piles in 2001 and 2002 ranged from 0.055 to 0.55 (Table 7). These values, as would be expected, are in reasonable agreement with the 0.14 value.

Assuming sulfate release was not limiting in either the laboratory or field and ignoring effects of temperature on oxidation rates, the difference between laboratory and field rates would be due to differences in pyrite surface area. (Note that field data indicate the oxygen content of the gas phase in the piles was near atmospheric concentrations.) A rough estimate of the relative pyrite surface areas in the laboratory and field can be made by assuming the variation of pyrite liberation as a function of particle size in the field was similar to that in the laboratory. For the laboratory solids, the degree of pyrite liberation increased with decreasing particle size, and the average degree of liberation particles in the -100/+150 size fraction ($105 < d < 149$ μm) was 81% (range of 67% - 90%). The percentage of laboratory solids in the -100 mesh fraction averaged 9.5%, ranging from 7.6% to 11.2% (Lapakko et al. 2002, Table 2). The percentage of field solids in this size fraction averaged 1.4%, ranging from 0.8% to 1.9% (Table 1). The ratio of the -100 mesh percentage in the field to that in the laboratory can be approximated using the quotient of average values, or $1.4/9.5 = 0.15$.

Given the assumptions stated at the beginning of the previous paragraph, the sulfate release rate in the field would be 0.15 times that in the laboratory. This is reasonably close to the 0.14 retardation factor determined based on slopes of graphs depicting sulfate release rates vs percent solid-phase sulfur. Furthermore, this suggests that available pyrite surface area is the dominant controlling factor in both the laboratory and field. Temperature differences between the field and laboratory and reduced sulfate removal in the field would also be expected to affect rates of sulfate release. The extent of pyrite liberation in the field solids is presently being determined and will provide additional insight on the pyrite surface area in the field relative to that in the laboratory. The influence of available surface area on observed sulfate release rates will be further assessed when these data are available.

The calcium retardation factors for 2001 and 2002 were near one, except for values from the 0.20%

S pile. These factors indicate the field rates were roughly equal to or greater than laboratory rates. In contrast a range of 0.14 to 0.46 was reported for calcium retardation factors for Duluth Complex rock (Lapakko 1994). The high values are partly due to a higher range of CaO contents in the field rock than in the laboratory rock (0.23%-0.76% vs 0.04%-0.29%). The very high retardation factors also suggest the presence of a highly soluble calcium phase present in the field rock but absent in the laboratory samples. As noted above, the CO_2 content of the field rock was higher than that typically observed in the laboratory, indicating the possibility of a calcium carbonate phase in the field rock.

Retardation factors for magnesium ranged from 0.008 to 0.6. The extremely low Mg retardation factor of 0.008 was strongly influenced by the elevated magnesium release rate from the corresponding laboratory sample which had a siderite content of 17.9%. Retardation factors for the remaining three piles (0.1-0.6) compare favorably to the 0.053 to 0.36 range reported for Duluth Complex rock (Lapakko 1994).

Release rates for sodium in the field were higher than those observed in the laboratory, as reflected by retardation factors ranging from approximately 3 to 30 (Table 7). This suggests the presence of a rapidly dissolving solid-phase form of sodium in the field that was not present in laboratory solids. Based on the large decrease in sodium release from 2001 to 2002, the amount of the reactive sodium-bearing phase may be relatively small.

Potassium release rates in the field ranged form roughly 0.1 to 0.9, with values for the 0.20%, 0.39% and 0.67% S piles ranging from 0.6 to 0.9 (Table 6). This indicates that potassium release rates in the field were similar to those in the laboratory.

5.2. Limestone Addition Tank Results

5.2.1. Introduction

Six field tanks were used to determine, as a function of limestone loading, the effectiveness of blending limestone with waste rock in controlling acid generation. The experiment began on 24 October 2000. In addition to two controls, duplicate tanks with limestone additions producing NP:AP ratios of 1:1 and 3:1 were examined. The average sulfur content of rock in the six tanks was 0.49%.

5.2.2. Solid Phase Analyses

Particle size distribution for rock in tanks 1 - 5 was determined using a dry screening method and a wet screening method was used for tank 6. Although 40% to 60% of all samples were finer than 0.5 inches, the wet screening produced a finer particle size distribution. The wet screening yielded 10.4% finer than 100 mesh as opposed to approximately 5% for the dry screening. This was considerably finer than the 0.8% to 1.9% finer than 100 mesh reported for dry screening of rock in the prediction piles. The limestone was 57% -20 mesh and 4.5% -100 mesh (Table 9).

The respective averages for total sulfur, sulfate, and CO_2 contents of the rock were 0.49%, 0.019%, and 0.39%, respectively. The approximate averages for major whole rock components for the tank samples were SiO_2 (66%), Al_2O_3 (13%), FeO (9%), MgO (3%), and K_2O (2%). CaO and Na_2O contents were both about 0.3% (Table 10). All values were within the range reported for the prediction piles. Copper, nickel, cobalt and zinc concentrations were similar to those reported for the 0.67% S prediction pile (Table A1.17. vs Table A1.10). The CO_2 content of the limestone was 41.56% (estimated as LOI), slightly less than the 44% expected for pure calcite.

Mineralogical analysis of two samples from the tanks indicated that the major minerals present in the rock were quartz (~50%), chlorite (~25%), and sericite (~20%). The samples contained roughly 1% pyrite and 1.2% siderite. The pyrite was reported to be fine grained and liberated only in rocks finer than 28 mesh (600 μm). Most of the liberated pyrite occurs in fractions finer than 48 mesh (300 μm). The total pyrite liberation is approximately 12% (Appendix 1, Attachment A1.1).

5.2.3. Flow

Drainage volumes from the limestone tanks in 2001 ranged from roughly 550 to 640 liters. These volumes represented 85% to 98% of the precipitation input of 649 liters (Table 11). Drainage volumes in 2002 were lower, ranging from approximately 360 to 390 liters. The yields, relative to input precipitation, in 2002 were considerably lower than in 2001. All values were near 50% (Table 11) and this is consistent with values reported in previous studies (Eger and Lapakko 1985; Smith and Beckie 2003). The decrease of yields was similar to that observed for the field piles. This behavior is difficult to explain and suggests an error in measurement of precipitation or flow, or a calculational error. No error was found and additional effort will be directed toward resolving this anomaly in the upcoming year.

5.2.4. Drainage Quality and Chemical Mass Release

Each tank was sampled once in 2000, thirteen times in 2001, and nine times in 2002. Drainage pH typically ranged from 7.5 - 8.5, peaked from June to August, and was in essentially the same range for all tanks to which limestone was added. The pH of drainage from the control tanks was at the lower end of this range and several values were below 7.5.

Alkalinity from all tanks decreased over the course of the study and tended to increase with increasing limestone loading (Tables A3.5-A3.10). Concentrations from the controls reached the range of 10 to 25 mg L^{-1} as $CaCO_3$ during June 2002. Concentrations from the 1:1 and 3:1 limestone additions stabilize in approximate respective ranges of 40-60 and 50-70 mg L^{-1} as $CaCO_3$ after September 2001.

Sulfate concentrations displayed no obvious, consistent temporal trends (Figures 24-29). Concentrations from the controls were higher than those from the tanks to which limestone was added (Tables A3.5-A3.10). In 2002, the average sulfate concentration from the controls was around 350 mg L^{-1} compared to and average of 290 mg L^{-1} from the treated tanks. These differences in sulfate concentration were reflected in the rates of sulfate release (Table 12), because the volume of drainage did not vary greatly among the tanks. The elevated sulfate concentrations from the controls suggest that there may be areas of accelerated pyrite oxidation within the tank. These could be areas in which acidic conditions have developed and pyrite oxidation is accelerated by bacterial mediation.

After elevated concentrations in the first three samples, calcium and magnesium concentrations typically ranged from 100 - 200 mg L^{-1} and 10 - 20 mg L^{-1}, respectively. Rates of calcium release were typically slightly higher than rates of sulfate release (Table 12). Although the average calcium release rate from the controls was slightly higher than that from the treated tanks, differences in both calcium and magnesium release rates among the tanks were relatively small (Table 12). The calcium release from the controls may have been slightly accelerated by the elevated rate of acid production in these tanks, as indicated by higher sulfate release rates.

Nitrate concentrations were elevated in 2000, likely due to the presence of residual blasting agents in the rock but showed a decreasing trend throughout the 2001 field season (Tables A3.5 - A3.10). Concentrations during 2002 typically ranged from 10 to 15 mg L^{-1}.

5.2.5. Comparison of Field and Laboratory Chemical Release Rates

Rates of chemical release from the limestone addition tanks were determined for sulfate, calcium and magnesium and compared to rates of release observed for a greenstone sample containing 0.50% S in the laboratory. For weeks 60-100, rates of sulfate, calcium and magnesium release were 116, 6.37, and 72.6 μmol (kg rock week)$^{-1}$ (Lapakko et al. 2002, Table 9). Although sulfate release rates from the controls were higher than those from the treated tanks, rates did not vary greatly among the various tanks. The observed range was 27 to 41 μmol (kg rock week)$^{-1}$, yielding retardation factors of 0.23 to 0.35. These factors are similar to those calculated for the field piles.

The -100 mesh fraction was about 5% for the controls and 11.2% for the 0.50% S sample in the laboratory. Normalizing the retardation factors for the difference in -100 mesh fractions (see section 5.1.6) yields adjusted normalized retardation factors of 0.52 and 0.78. This indicates that after an estimated adjustment for available pyrite surface area the rates of sulfate release from the field tank controls were roughly 50% to 75% of laboratory rates.

Sulfate release rates from the alkaline addition tank controls were also compared to those observed from the test piles. The average sulfur content of the six tanks was 0.49% (Table 10) and five percent of the rock in control tanks 1 and 2 was finer than 100 mesh (Table 9). Using the slope of the line presented in Figure 23, a sulfate release rate of 16 μmol (kg rock week)$^{-1}$ was calculated (32.2 x 0.49 = 16). Accounting for the difference in the -100 fraction (5% for the tanks vs 1.4% for the field piles) yields a predicted rate of (5/1.4) x 16 = 56 μmol (kg rock week)$^{-1}$. This is roughly 40% higher than the average value of 40 μmol (kg rock week)$^{-1}$ observed for the controls.

Calcium release rates also fell into a fairly small range of 33 to 43 μmol (kg rock week)$^{-1}$, and appeared to be independent of limestone loading (Table 12, p. 2). Retardation factors of 5.2 to 6.8 indicated the field rates were higher than those in the laboratory. This suggests a soluble calcium phase, as well as the limestone added, was present in the field rock but not in the laboratory sample. The average Ca and CO_2 contents of the field rock were 0.39 and 0.38 percent (Table 10), indicating that some calcium may be present as a carbonate. The corresponding values for the laboratory rock were 1.76 and 0.03 percent (Lapakko et al. 2002).

Magnesium release rates ranged from 6.3 to 7.8 μmol (kg rock week)$^{-1}$ and demonstrated no strong dependence on limestone loading. Retardation factors ranged from 0.087 to 0.11. These factors were relatively low due to the high rates of magnesium release from the siderite-bearing sample in the laboratory.

6. PLANS

The following tasks have been identified for completion in the upcoming biennium.

1. Extend data collection and interpretation through the 2004 field season.
2. Determine the chemistry of additional samples collected from field piles to assess compositional variability within individual piles.
3. Determine the Sobek NP values for suites of samples from piles 1, 2, and 3.
4. Determine the variation in modes of occurrence of sulfide and carbonate minerals within individual test piles.
5. Determine the mineralogical composition of rock used in field tests.
6. Determine the composition of carbonate minerals in field tests.
7. Determine the extent of NP depletion from test piles and tanks.
8. Investigate possible reasons for the large change from 2001 to 2002 in yield coefficients from both test piles and limestone addition tanks.
9. Make estimations to consider flow not included in mass release rate calculations for test piles in 2001.
10. Determine the relationship between field rates of chemical release and flow.
11. Continue the comparison of field and laboratory rates and rates reported in the literature.

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Johnson, with assistance from Doug Rosnau, was responsible for collection and analyses of field test pile samples. Andrea was also responsible for producing appendix tables and generating tables and figures throughout the report. Sue Backe and Sue Saban provided data input. Lyn Leopold assisted in obtaining silicate mineral dissolution literature.

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Table 1. Particle size distribution of rock used in field test piles (percent passing).

SIZE FRACTION	TEST PILE 1	TEST PILE 2	TEST PILE 3	TEST PILE 4
+ 12"	12.2	8.7	3.0	7.9
- 12" / +6"	10.6	8.9	6.2	6.1
- 6" / +21/2"	21.4	23.3	12.5	11.1
- 21/2" / +3/4"	33.0	32.7	30.9	32.7
-3/4" / +1/4"	11.4	10.5	22.1	19.3
-1/4" / +10 mesh	5.2	6.2	12.1	11.7
-10 / +35 mesh	3.8	6.2	8.4	6.9
-35 / +100 mesh	1.5	2.3	3.1	2.4
-100 / +200 mesh	0.3	0.4	0.7	0.7
-200 mesh	0.5	0.8	1.0	1.2

Note: All size fractions were dry sieved.

Table 2. Sulfur analyses of muck box samples for field test piles 1 – 4 (n = 25). Analyses by Lerch Bros.

TEST PILE 1 MEAN % S = 0.02, SD = 0.019	TEST PILE 2 MEAN % S = 0.20, SD = 0.095	TEST PILE 3 MEAN % S = 0.39, SD = 0.226	TEST PILE 4 MEAN % S = 0.67, SD = 0.223
0.005	0.08	0.20	0.29
0.006	0.10	0.20	0.33
0.01	0.13	0.22	0.42
0.01	0.13	0.23	0.45
0.01	0.13	0.23	0.46
0.01	0.13	0.24	0.47
0.01	0.14	0.24	0.48
0.01	0.18	0.25	0.50
0.01	0.18	0.28	0.56
0.01	0.19	0.29	0.58
0.01	0.20	0.29	0.59
0.01	0.20	0.30	0.64
0.01	0.21	0.32	0.66
0.02	0.21	0.33	0.69
0.02	0.21	0.34	0.74
0.02	0.21	0.36	0.75
0.02	0.22	0.36	0.76
0.02	0.25	0.39	0.77
0.02	0.26	0.41	0.79
0.03	0.32	0.49	0.80
0.03	0.32	0.50	0.94
0.04	0.33	0.51	0.94
0.05	0.37	0.61	1.02
0.07	0.40	0.81	1.04
0.08	0.46	1.47	1.09

Table 3. Average whole rock composition of field test piles. Analyses by ACTLABS.

Sample	0.02% S[1]	0.20% S[1]	0.39% S[1]	0.67% S[1]
%S	0.012	0.222	0.374	0.634
SO_4 as S	0.016	0.025	0.028	0.032
CO_2	0.124	0.239	0.054	0.462
S_iO_2	52.05	60.76	67.92	65.99
Al_2O_3	19.74	14.96	13.01	12.85
Fe_2O_3	11.27	10.10	8.66	10.48
MnO	0.133	0.092	0.073	0.089
MgO	6.35	5.26	3.25	3.38
CaO	0.723	0.765	0.378	0.226
Na_2O	0.402	0.283	0.394	0.203
K_2O	2.54	1.83	1.70	1.90
T_iO_2	0.870	0.667	0.538	0.515
P_2O_5	0.403	0.411	0.257	0.132
LOI	5.80	4.78	3.53	4.02
Total	100.28	99.89	99.78	99.78

1 - Average sulfur content of the 25 muck box samples determined by Lerch Brothers.

Table 4. Average whole rock composition from 25 muck box samples and of the particle size sample from each field test pile. Analyses by ACTLABS.

PARAMETER	Pile 1 (0.02 %S)		Pile 2 (0.20 %S)		Pile 3 (0.39 %S)		Pile 4 (0.67 %S)	
	Average value	Mass weighted average	Average value	Mass weighted average	Average value	Mass weighted average	Average value	Mass weighted average
%S	0.012	0.04	0.222	0.532	0.374	0.363	0.634	0.548
SO_4 as S	0.016	0.043	0.025	0.025	0.028	0.030	0.032	0.051
CO_2	0.124	0.119	0.239	0.255	0.054	0.132	0.462	0.082
S_iO_2	52.05	51.839	60.76	67.010	67.92	68.149	65.99	71.423
Al_2O_3	19.74	19.663	14.96	13.123	13.01	12.004	12.85	11.830
Fe_2O_3	11.27	11.627	10.10	9.205	8.66	8.982	10.48	7.834
MnO	0.133	0.130	0.092	0.081	0.073	0.063	0.089	0.041
MgO	6.35	6.526	5.26	3.198	3.25	3.937	3.38	2.822
CaO	0.723	0.596	0.765	0.591	0.378	0.559	0.226	0.187
Na_2O	0.402	0.491	0.283	0.236	0.394	0.281	0.203	0.222
K_2O	2.54	2.294	1.83	2.010	1.70	1.383	1.90	1.896
T_iO_2	0.870	0.842	0.667	0.483	0.538	0.507	0.515	0.417
P_2O_5	0.403	0.305	0.411	0.201	0.257	0.312	0.132	0.093

NOTE: The mass weighted values do not include the +12" and -12" / + 6" size fractions. These two size fractions were not analyzed.

Table 5. Page 1 of 4. Dissolved oxygen and temperature data for field piles.

Date	0.02% Sulfur (Piles 1) Lower sampling port		
	Temperature (C)	5' DO (%)	10' DO (%)
8/2/00	18.1	22.0	20.4
8/15/00	18.0	16.8	17.3
8/18/00	17.1	19.1	19.1
9/26/00	11.3	15.6	15.7
3/27/01	-0.5	9.6	9.4
6/21/01	11.8	20.2	19.5
7/11/01	14.6	23.5	19.9
7/24/01	18.1	21.0	21.1
8/02/01	17.7	18.6	16.6
8/23/01	16.7	18.2	13.9
9/25/01	13.8	20.5	20.4
10/17/01	11.7	24.2	24.5
5/15/02	0.7	19.1	19.4
7/11/02	17.0	17.2	17.2
8/26/02	15.6	17.4	17.4
Average	12.7	18.9	18.1

Table 5. Page 2 of 4. Dissolved oxygen and temperature data for field piles.

Date	0.20% Sulfur (Pile 2)					
	Lower sampling port			Upper sampling port		
	Temperature (C)	5' DO (%)	10' DO (%)	Temperature (C)	5' DO (%)	10' DO (%)
3/27/01	-0.1	16.9	8.1	-1.6	19.3	19.1
6/21/01	12.4	20.5	20.1	14.4	21.5	20.6
7/11/01	14.7	19.9	19.9	17.6	20.2	19.9
7/24/01	17.4	21.4	21.4	21.4	21.3	21.3
8/02/01	17.1	16.6	16.6	19.0	17.1	16.8
8/23/01	17.4	13.9	18.2	18.6	18.2	18.0
9/25/01	14.4	20.5	20.5	12.9	20.7	20.2
10/17/01	11.5	24.2	24.2	9.2	24.5	24.2
5/12/02	0.9	18.9	19.4	3.0	20.1	18.9
7/11/02	16.8	16.7	17.2	19.9	17.4	16.9
8/26/02	16.4	16.1	15.6	17.8	17.9	17.4
Average	11.9	18.7	18.3	12.8	19.8	19.4

Table 5. Page 3 of 4. Dissolved oxygen and temperature data for field piles.

Date	0.39% Sulfur (Pile 3)					
	Lower sampling port			Upper sampling port		
	Temperature (C)	5' DO (%)	10' DO (%)	Temperature (C)	5' DO (%)	10' DO (%)
9/26/00	14.6	8.8	16.5	12.2	16.4	13.6
3/27/01	-0.1	6.7	NA[1]	-1.6	12.0	11.8
6/21/01	12.8	16.9	19.2	14.5	20.8	20.2
7/11/01	15.1	20.2	20.1	17.6	20.4	20.5
7/24/01	17.7	21.7	21.9	21.4	21.6	21.7
8/02/01	17.6	15.3	16.8	19.3	19.4	18.4
8/23/01	17.8	NA[2]	NA[2]	18.8	18.2	22.2
9/25/01	15.1	NA[2]	20.9	12.9	20.7	20.5
10/17/01	11.8	NA[2]	24.5	9.2	25.2	25.5
5/15/02	1.7	NA[2]	19.4	3.2	20.1	19.4
7/11/02	17.0	NA[2]	18.2	19.8	19.2	19.4
8/26/02	16.8	NA[2]	17.9	18.2	18.2	17.9
Average	13.2	14.9	19.5	13.8	19.4	19.3

[1] Oxygen not sampled due to water in tubing.
[2] Oxygen not sampled due to obstructed tubing.

Table 5. Page 4 of 4. Dissolved oxygen and temperature data for field piles.

Date	0.67% Sulfur (Pile 4)					
	Lower sampling port			Upper sampling port		
	Temperature (C)	5' DO (%)	10' DO (%)	Temperature (C)	5' DO (%)	10' DO (%)
8/2/00	19.6	20.5	20.9	23.9	19.7	19.6
8/15/00	18.7	8.5	15.2	21.2	15.7	15.5
8/18/00	18.2	16.9	19.1	19.8	19.2	22.8
9/26/00	14.5	9.0	17.3	11.5	16.2	15.3
3/27/01	-0.1	10.3	8.6	-2.2	9.0	9.8
6/21/01	13.1	16.7	NA[1]	15.5	22.0	21.2
7/11/01	15.6	19.4	18.8	20.0	19.7	19.4
7/24/01	18.4	20.5	20.5	23.2	21.3	20.8
8/02/01	17.9	15.6	15.6	20.6	16.6	16.6
8/23/01	18.0	NA[2]	21.1	20.0	22.2	22.2
9/25/01	15.1	NA[2]	20.2	12.8	20.2	20.2
10/17/01	11.7	NA[2]	22.9	8.5	24.0	15.6
5/15/02	2.7	NA[2]	20.1	4.4	20.4	20.6
7/11/02	17.5	NA[2]	18.9	20.7	19.4	19.2
8/26/02	16.9	NA[2]	19.0	19.1	19.0	19.0
Average	14.5	15.3	18.4	15.3	19.0	18.5

[1] Oxygen not sampled due to water in tubing.
[2] Oxygen not sampled due to obstructed tubing.

Table 6. 2000, 2001, and 2002 total input and output flow volumes in liters and yield coefficients for prediction piles.

Pile	INPUT FLOW VOLUME	OUTPUT FLOW VOLUME	YIELD COEFFICIENT
2000			
1	11,392	7,029	NA
2	5,254	3,808	NA
3	6,617	3,660	NA
4	11,534	7,021	NA
2001			
1	24,023	20,383	85 %
2	24,023	20,029	83 %
3	24,023	17,486	73 %
4	24,023	19,606	81 %
2002			
1	25963	16337	63%
2	25963	15772	61%
3	25963	13237	51%
4	25963	17360	67%

Table 7. Page 1 of 5. Sulfate, calcium, magnesium, sodium, and potassium release rates from greenstone prediction field piles (µmol (kg rock week)$^{-1}$).

Sulfate Release 2001					
Date	Weeks	0.02% S	0.20% S	0.39% S	0.67% S
4/25/01	4	2.95	13.8	6.10	15.5
5/10/01	2	4.83	11.1	4.81	14.2
5/23/01	2	3.67	9.73	5.88	18.3
6/15/01	3	3.00	14.7	9.62	19.4
8/02/01	7	0.858	4.38	2.92	4.31
8/20/01	2	2.29	20.1	7.26	12.9
9/12/01	3	0.430	5.02	1.39	2.63
10/11/01	4	3.01	10.5	9.95	17.7
10/30/01	2	2.11	7.45	12.7	23.2
11/28/01	4	1.12	3.45	5.71	10.8
Average rate		2.43	10.0	6.64	13.9
Lab[1]	60-100	4.46	66.6[2]	120[2]	98.3
Retardation Factor[4]		0.545	0.150	0.055	0.141
Sulfate Release 2002					
4/28/02	2	1.52	4.08	7.98	13.3
6/4/02	6	0.485	1.63	1.96	3.99
6/24/02	3	4.63	17.3	21.0	55.1
7/8/02	2	1.98	11.1	12.9	22.8
7/29/02	3	1.18	4.04	6.11	12.5
8/13/02	2	3.66	18.2	25.8	44.6
9/3/02	3	1.89	5.61	13.0	25.9
9/19/02	2	1.17	3.60	6.31	12.9
10/7/02	3	1.41	5.98	11.3	20.6
10/28/02	3	0.481	0.896	5.49	10.2
Average rate		1.84	7.24	11.2	22.2
Lab[1]	100-154	3.94	58.4[3]	111[3]	89.9
Retardation Factor[4]		0.467	0.124	0.101	0.247

[1] Data from laboratory reactors with 0.04%, 0.20%, 0.39% and 0.72% sulfur.

[2] Median pH values for 0.20% S and 0.39%S rock for weeks 60-100 were 4.15 and 3.97.

[3] Median pH values for 0.20% S and 0.39%S rock for weeks 100-154 were 4.09 and 3.91

[4] Field rate/lab rate.

Table 7. Page 2 of 5. Sulfate, calcium, magnesium, sodium, and potassium release rates from greenstone prediction field piles (μmol (kg rock week)$^{-1}$).

Calcium Release 2001					
Date	Weeks	0.02% S	0.20% S	0.39% S	0.67% S
4/25/01	4	13.8	46.3	31.7	28.7
5/10/01	2	21.9	36.4	24.9	23.7
5/23/01	2	8.71	28.7	23.2	27.6
6/15/01	3	6.95	37.0	29.1	29.4
8/02/01	7	2.12	11.8	5.07	5.68
8/20/01	2	5.64	49.7	12.8	14.9
9/12/01	3	1.07	11.3	2.38	3.01
10/11/01	4	7.30	24.5	14.9	20.2
10/30/01	2	5.02	14.7	17.3	25.7
11/28/01	4	2.43	7.06	7.49	11.9
Average rate		7.50	26.7	16.9	19.1
Lab[1]	60-100	6.79	3.32	16.1	16.8
Retardation Factor[2]		1.10	8.04	1.05	1.14
Calcium Release 2002					
4/28/02	2	3.84	7.87	10.8	13.9
6/4/02	6	1.41	2.67	2.63	4.33
6/24/02	3	10.4	34.8	26.6	57.1
7/8/02	2	3.88	17.1	14.2	23.4
7/29/02	3	2.85	8.15	7.90	13.5
8/13/02	2	8.19	29.2	26.6	44.5
9/3/02	3	3.90	10.2	14.0	26.6
9/19/02	2	2.97	6.31	6.39	13.0
10/7/02	3	3.22	10.4	12.2	20.4
10/28/02	3	1.38	1.34	5.86	10.2
Average rate		4.21	12.8	12.7	22.7
Lab[1]	100-154	4.98	2.51	12.7	20.5
Retardation Factor[2]		0.845	5.10	1.00	1.11

[1] Data from lab reactors with 0.04%, 0.20%, 0.39% and 0.72% sulfur.
[2] Field rate/lab rate.

Table 7. Page 3 of 5. Sulfate, calcium, magnesium, sodium, and potassium release rates from greenstone prediction field piles (μmol (kg rock week)$^{-1}$).

Magnesium Release 2001					
Date	Weeks	0.02% S	0.20% S	0.39% S	0.67% S
4/25/01	4	3.13	8.38	6.57	3.89
5/10/01	2	4.85	6.26	4.87	3.56
5/23/01	2	2.12	4.65	4.27	3.64
6/15/01	3	1.30	4.88	4.85	3.34
8/02/01	7	0.397	1.69	0.873	0.696
8/20/01	2	1.19	7.43	2.38	1.96
9/12/01	3	0.223	1.71	0.438	0.398
10/11/01	4	1.70	4.08	3.08	2.93
10/30/01	2	1.05	2.21	3.27	3.42
11/28/01	4	0.580	1.15	1.51	1.65
Average rate		1.67	4.24	3.21	2.55
Lab[1]	60-100	9.68	6.83	18.0	241
Retardation Factor[2]		0.173	0.621	0.178	0.011
Magnesium Release 2002					
4/28/02	2	0.769	1.16	1.76	1.74
6/4/02	6	0.285	0.455	0.451	0.529
6/24/02	3	1.96	4.99	4.26	6.45
7/8/02	2	0.726	2.59	2.33	2.72
7/29/02	3	0.499	1.08	1.21	1.45
8/13/02	2	1.43	3.90	4.09	4.90
9/3/02	3	0.668	1.31	2.18	2.87
9/19/02	2	0.491	0.831	0.952	1.41
10/7/02	3	0.568	1.40	1.95	2.24
10/28/02	3	0.248	0.230	0.938	1.06
Average rate		0.764	1.79	2.01	2.54
Lab[1]	100-154	7.73	7.72	13.9	321
Retardation Factor[2]		0.099	0.232	0.145	0.008

[1] Data from lab reactors with 0.04%, 0.20%, 0.39% and 0.72% sulfur.
[2] Field rate/lab rate.

Table 7. Page 4 of 5. Sulfate, calcium, magnesium, sodium, and potassium release rates from greenstone prediction field piles (μmol (kg rock week)$^{-1}$).

Sodium Release 2001					
Date	Weeks	0.02% S	0.20% S	0.39% S	0.67% S
4/25/01	4	32.0	30.9	33.8	22.6
5/10/01	2	66.6	29.5	32.6	16.9
5/23/01	2	42.1	30.3	32.2	22.0
6/15/01	3	38.9	32.1	26.0	17.0
8/02/01	7	8.31	7.01	3.44	2.30
8/20/01	2	19.0	31.4	8.46	5.12
9/12/01	3	3.17	6.93	1.37	0.863
10/11/01	4	18.1	13.4	6.81	4.72
10/30/01	2	13.9	7.55	6.55	4.38
11/28/01	4	5.96	3.43	2.64	1.97
Average rate		24.8	19.3	15.4	9.78
Lab[1]	60-100	0.728	0.837	0.887	0.622
Retardation Factor[2]		34.1	23.1	17.4	15.7
Sodium Release 2002					
4/28/02	2	9.94	3.53	3.83	2.51
6/4/02	6	3.20	1.24	0.964	0.703
6/24/02	3	29.5	18.8	10.3	10.4
7/8/02	2	14.7	10.3	5.53	3.67
7/29/02	3	7.70	4.12	2.62	1.91
8/13/02	2	24.4	14.7	8.04	5.85
9/3/02	3	10.5	4.92	3.47	2.63
9/19/02	2	6.38	2.58	1.35	1.47
10/7/02	3	6.28	3.90	2.23	1.66
10/28/02	3	2.88	0.554	1.10	0.792
Average rate		11.6	6.47	3.94	3.16
Lab[1]	100-154	0.583	0.857	0.894	0.915
Retardation Factor [2]		19.9	7.55	4.41	3.45

[1] Data from lab reactors with 0.04%, 0.20%, 0.39% and 0.72% sulfur.

[2] Field rate/lab rate.

Table 7. Page 5 of 5. Sulfate, calcium, magnesium, sodium, and potassium release rates from greenstone prediction field piles (μmol (kg rock week)$^{-1}$).

Potassium Release 2001					
Date	Weeks	0.02% S	0.20% S	0.39% S	0.67% S
4/25/01	4	2.42	3.81	4.53	2.36
5/10/01	2	3.89	3.78	3.82	2.98
5/23/01	2	3.35	4.50	4.64	4.85
6/15/01	3	3.18	5.10	5.10	5.30
8/02/01	7	0.933	1.42	0.956	1.09
8/20/01	2	1.83	5.73	2.24	2.44
9/12/01	3	0.341	1.58	0.397	0.457
10/11/01	4	1.89	2.55	2.06	2.32
10/30/01	2	1.25	1.59	2.02	2.37
11/28/01	4	0.826	0.694	0.824	0.997
Average rate		1.99	3.08	2.66	2.52
Lab[1]	60-100	12.4	3.35	4.51	3.67
Retardation Factor [2]		0.160	0.919	0.590	0.687
Potassium Release 2002					
4/28/02	2	0.902	0.920	1.13	1.12
6/4/02	6	0.325	0.335	0.319	0.349
6/24/02	3	3.58	4.82	4.06	5.97
7/8/02	2	2.15	3.13	2.60	3.22
7/29/02	3	1.42	1.34	1.35	1.70
8/13/02	2	3.05	4.77	4.07	5.00
9/3/02	3	1.59	1.84	2.17	2.68
9/19/02	2	1.36	1.10	1.46	1.37
10/7/02	3	0.955	1.33	1.26	1.51
10/28/02	3	0.802	0.194	0.597	0.706
Average rate		1.61	1.98	1.90	2.36
Lab[1]	100-154	8.31	2.25	3.19	2.81
Retardation Factor [2]		0.194	0.880	0.596	0.840

[1] Data from lab reactors with 0.04%, 0.20%, 0.39% and 0.72% sulfur.

[2] Field rate/lab rate.

Table 8. Ratio of drainage volumes[1] and chemical releases in 2002 to those in 2001. The drainage volume in 2002 approximated that in 2001. Rates of chemical release in 2002 were typically lower than those in 2001, reflecting decreasing concentrations in the drainage. Rates of sulfate release from the two piles of highest sulfur content increased from 2001 to 2002. This may indicate that acid conditions may be developing in some areas of these two piles.

Pile, Pct S	Drainage Volume[1]	SO_4	Ca	Mg	Na	K
0.02	0.99	0.76	0.56	0.46	0.47	0.81
0.20	1.01	0.72	0.48	0.42	0.34	0.64
0.39	0.96	1.69	0.75	0.63	0.26	0.71
0.67	1.06	1.60	1.18	1.00	0.32	0.94

[1] The drainage volume used for 2001 was that used to determine mass release rates. The actual drainage volume from the piles was approximately 20 to 30 percent higher than this value. This discrepancy will be rectified in the next report.

Table 9. Particle size distribution for rock and limestone used in field limestone addition tanks (percent passing). Analysis by Lerch Brothers, Inc.

FRACTION	Tank 1	Tank 2	Tank 3	Tank 4	Tank 5	Tank 6	Limestone
2"	100.0	100.0	100.0	100.0	100.0	100.0	100.0
1 ½"	97.1	97.3	91.3	90.1	88.9	95.3	100.0
1"	68.2	77.4	80.2	72.6	77.5	82.4	100.0
1/2"	43.3	40.5	53.2	52.4	61.7	54.8	100.0
1/4"	31.5	27.2	37.5	36.2	43.8	42.0	100.0
4M	27.1	22.9	32.4	31.1	37.8	37.8	100.0
10M	16.7	14.2	19.6	18.7	23.1	25.3	87.9
20M	12.6	10.8	14.4	13.6	16.9	19.5	57.1
28M	10.8	9.3	12.0	11.4	14.1	17.0	41.8
35M	9.5	8.2	10.2	9.9	12.1	15.3	30.2
48M	7.8	6.7	6.9	7.9	9.6	13.3	18.5
65M	6.2	5.5	5.1	6.3	7.6	11.6	9.0
100M	5.4	4.6	4.7	5.2	6.3	10.4	4.5
200M	3.4	2.8	2.5	3.1	3.7	8.2	1.4

NOTE: All samples were dry screened with the exception of Tank 6 which was wet screened for comparison.

Table 10. Whole rock chemistry (percent) of field limestone addition tanks. Analyses by ACTLABS, Inc.

Parameter	Tank 1	Tank 2	Tank 3	Tank 4	Tank 5	Tank 6	Limestone
S	0.56	0.40	0.50	0.46	0.55	0.48	0.01
S^{2-} [2]	0.54	0.34	0.48	0.44	0.53	0.46	0
SO_4^{2-} as S	0.016	0.06	0.02	0.016	0.02	0.02	0.016
CO_2	0.40	0.37	0.22	0.44	0.44	0.48	41.56[3]
Al_2O_3	13.86	13.09	12.53	13.12	13.28	13.00	0.47
CaO	0.41	0.37	0.26	0.46	0.32	0.43	27.63
Fe_2O_3	10.65	8.83	8.61	9.38	10.30	9.31	0.87
K_2O	1.86	2.03	1.95	1.91	1.85	2.09	0.29
MgO	3.59	2.96	2.91	3.26	3.41	2.89	18.82
MnO	0.068	0.065	0.053	0.089	0.065	0.059	0.081
Na_2O	0.36	0.38	0.30	0.39	0.34	0.38	<0.01
P_2O_5	0.15	0.12	0.10	0.12	0.13	0.21	0.03
SiO_2	64.70	68.40	69.30	65.96	65.38	67.32	9.68
TiO_2	0.587	0.458	0.452	0.522	0.519	0.514	0.026
LOI	4.19	3.73	3.62	3.89	4.02	3.72	41.95
TOTAL[2]	100.42	100.44	100.08	99.11	99.60	99.92	99.78

1 - Determined by difference. Less than values are assumed to be 0.
2 - For parameters from Al_2O_3 through LOI2.
3 - Analysis by Lerch Brothers Inc. and determined by LOI.

Table 11. 2001 and 2002 total input and output flow volumes in liters and yield coefficients for limestone tanks.

TANK	RATIO	INPUT FLOW VOLUME	OUTPUT FLOW VOLUME	YIELD COEFFICIENT
2001				
1	Control	649	635	98 %
6	Control	649	558	86 %
2	1:1	649	599	92 %
5	1:1	649	570	88 %
3	3:1	649	554	85 %
4	3:1	649	555	86 %
2002				
1	Control	737	371	50%
6	Control	737	387	53%
2	1:1	737	375	51%
5	1:1	737	363	49%
3	3:1	737	357	48%
4	3:1	737	363	49%

Note: Due to a heavy rainfall on 6/23/02, flow volumes for 6/24/02 were estimated based on the total flow and precipitation for the field season and the rainfall for the heavy rain event.

Table 12. Page 1 of 3. Sulfate, calcium and magnesium release rates (μmol(kg rock week)$^{-1}$) from limestone tanks.

Date	Week	Sulfate Release					
		Control Tank 1	Control Tank 6	1:1 Ratio Tank 2	1:1 Ratio Tank 5	3:1 Ratio Tank 3	3:1 ratio Tank 4
4/25/01	4	21.3	16.3	15.8	15.2	19.0	15.5
5/10/01	2	17.3	8.52	8.02	8.87	7.24	8.01
5/23/01	2	34.1	35.6	29.8	43.3	36.4	33.2
6/15/01	3	51.5	36.0	32.3	44.8	41.8	33.9
8/02/01	7	9.61	6.47	9.77	13.7	13.1	7.11
8/20/01	2	32.1	32.8	28.0	36.5	39.3	32.8
9/12/01	3	9.75	9.72	4.99	9.31	9.10	9.40
9/28/01	2	30.9	30.5	26.7	36.9	35.8	28.4
10/11/01	2	63.5	57.0	45.6	66.5	63.0	51.4
10/30/01	2	43.9	28.2	20.6	39.8	39.3	36.5
11/26/01	4	44.8	151	20.2	32.1	32.7	26.7
4/10/02	1	54.4	24.6	60.3	66.4	54.1	74.6
5/10/02	4	16.4	18.4	10.9	12.3	15.4	8.32
6/4/02	4	6.84	6.92	3.69	5.74	4.64	6.21
6/24/02	3	112	203	76.6	105	99.3	88.9
7/8/02	2	44.8	28.7	30.8	34.2	30.7	33.4
7/29/02	3	19.1	32.5	13.0	18.5	15.6	17.0
8/12/02	2	92.6	43.8	59.7	81.8	49.3	64.3
9/19/02	5	48.0	39.4	31.7	44.2	39.1	28.7
10/28/20	6	22.5	1.85	11.2	15.5	15.8	14.4
Average Rate		38.7	40.6	27.0	36.6	33.0	30.9

Table 12. Page 2 of 3. Sulfate, calcium and magnesium release rates (μmol(kg rock week)$^{-1}$) from limestone tanks.

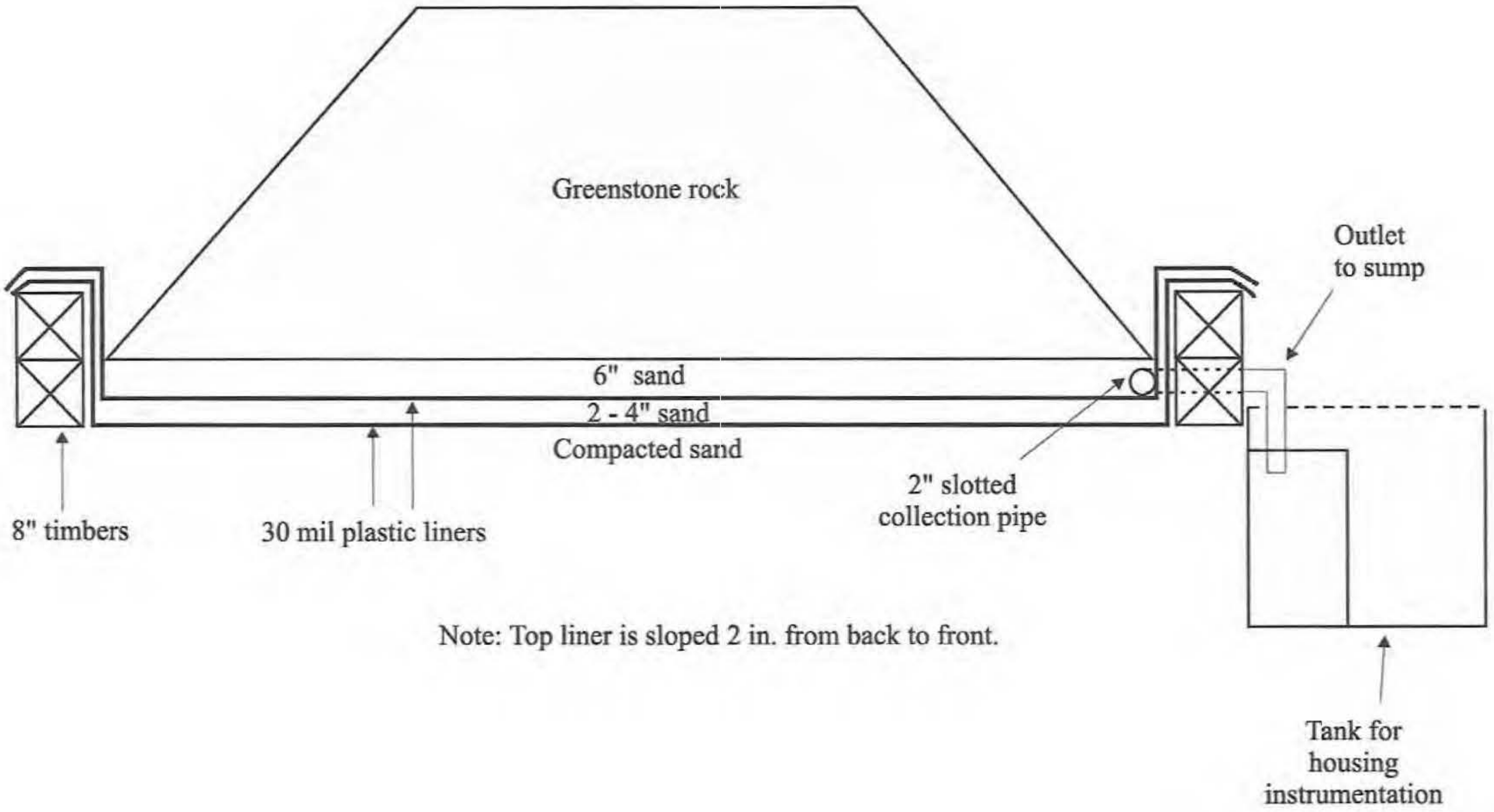
Date	Week	Calcium Release					
		Control Tank 1	Control Tank 6	1:1 Ratio Tank 2	1:1 Ratio Tank 5	3:1 Ratio Tank 3	3:1 ratio Tank 4
4/25/01	4	65.1	34.9	42.5	40.2	42.9	43.2
5/10/01	2	37.5	12.9	19.9	21.5	12.4	15.9
5/23/01	2	62.7	50.0	47.4	54.8	50.0	52.0
6/15/01	3	57.4	42.3	42.7	55.0	53.2	45.2
8/02/01	7	12.2	8.40	12.0	17.9	14.3	8.67
8/20/01	2	32.3	33.9	29.0	42.9	42.7	34.7
9/12/01	3	10.4	9.75	5.53	9.00	9.17	9.50
9/28/01	2	32.5	28.9	27.4	37.0	36.1	27.6
10/11/01	2	62.3	53.4	49.7	66.3	63.8	49.7
10/30/01	2	43.2	25.7	25.3	38.5	38.5	34.3
11/26/01	4	43.9	142	20.9	31.8	33.0	25.7
4/10/02	1	53.6	23.0	66.9	69.9	61.4	83.4
5/10/02	4	16.5	18.0	11.6	12.8	15.8	8.65
6/4/02	4	5.89	6.67	3.23	5.43	4.30	5.51
6/24/02	3	114	188	88.8	115	110	94.0
7/8/02	2	43.2	27.0	34.2	36.1	34.4	34.1
7/29/02	3	18.6	30.2	13.4	20.0	17.0	17.8
8/12/02	2	89.6	40.9	66.2	83.3	52.8	68.7
9/19/02	5	45.8	39.3	34.6	44.9	40.6	29.0
10/28/02	6	20.6	1.71	11.4	15.6	16.4	15.1
Average Rate		43.4	40.8	32.6	40.9	37.4	35.1

Table 12. Page 3 of 3. Sulfate, calcium and magnesium release rates (μmol(kg rock week)$^{-1}$) from limestone tanks.

Date	Week	Magnesium Release					
		Control Tank 1	Control Tank 6	1:1 Ratio Tank 2	1:1 Ratio Tank 5	3:1 Ratio Tank 3	3:1 ratio Tank 4
4/25/01	4	10.4	6.27	7.77	7.17	8.30	8.51
5/10/01	2	6.52	2.12	3.60	3.85	2.44	3.13
5/23/01	2	9.74	8.17	7.89	9.04	9.29	10.1
6/15/01	3	8.60	6.41	7.03	8.41	9.08	7.88
8/02/01	7	1.69	1.27	2.01	2.77	2.73	1.59
8/20/01	2	4.40	4.97	4.95	6.21	7.73	6.40
9/12/01	3	1.50	1.49	0.954	1.44	1.74	1.81
9/28/01	2	4.78	4.65	4.97	5.87	7.33	5.71
10/11/01	2	9.45	8.69	9.18	10.5	12.9	10.3
10/30/01	2	6.61	4.40	4.61	6.49	8.38	7.36
11/26/01	4	6.91	24.5	4.19	5.38	7.05	5.51
4/10/02	1	7.82	4.14	12.3	11.9	12.4	17.9
5/10/02	4	2.62	3.03	2.40	2.12	3.56	1.84
6/4/02	4	0.972	1.16	0.68	0.934	0.994	1.22
6/24/02	3	18.0	31.0	16.6	18.6	22.8	19.9
7/8/02	2	7.02	4.52	7.08	6.28	7.56	7.51
7/29/02	3	3.42	5.77	3.27	3.78	4.19	4.40
8/12/02	2	16.1	8.36	16.2	16.6	13.0	17.3
9/19/02	5	8.56	6.88	7.74	8.05	9.35	6.54
10/28/02	6	3.97	0.363	2.78	3.15	4.08	3.61
Average Rate		6.96	6.91	6.31	6.93	7.75	7.42

Figure 1. Bin construction.

Figure 2. Side view of bin construction for greenstone field experiment.



Note: Top liner is sloped 2 in. from back to front.

Figure 3. Leachate collection system for greenstone field experiment.

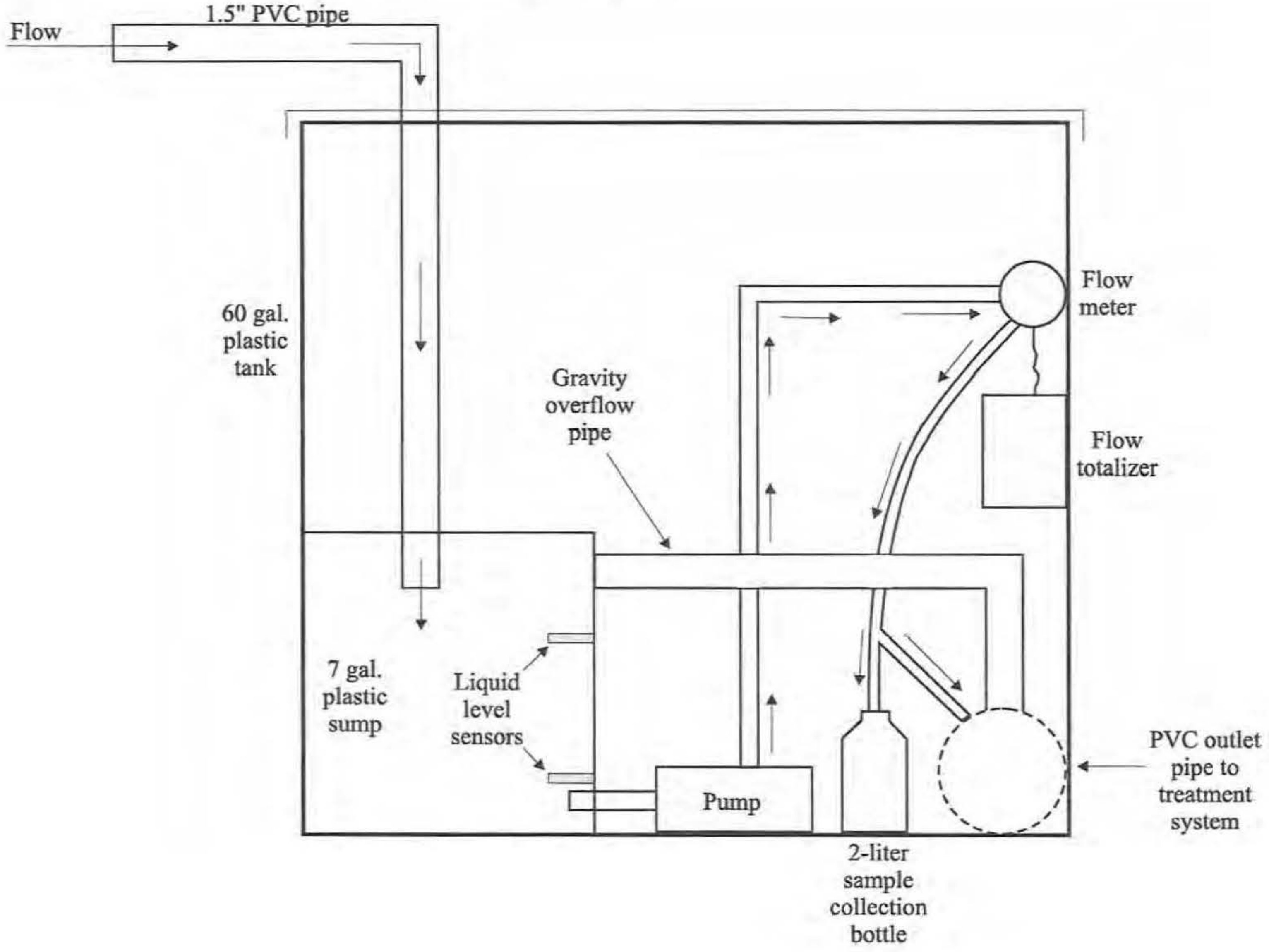


Figure 4. Leachate collection system.

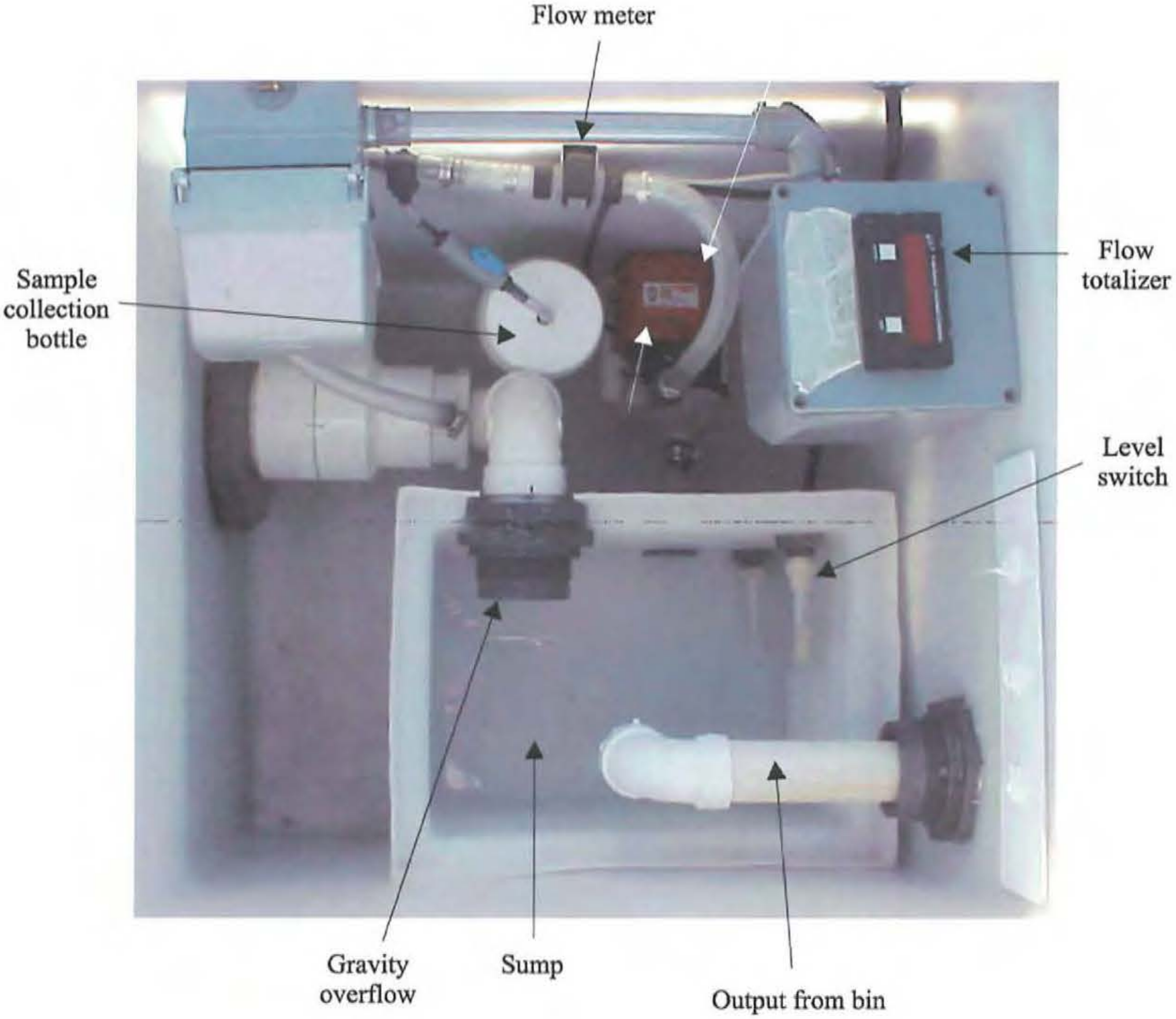
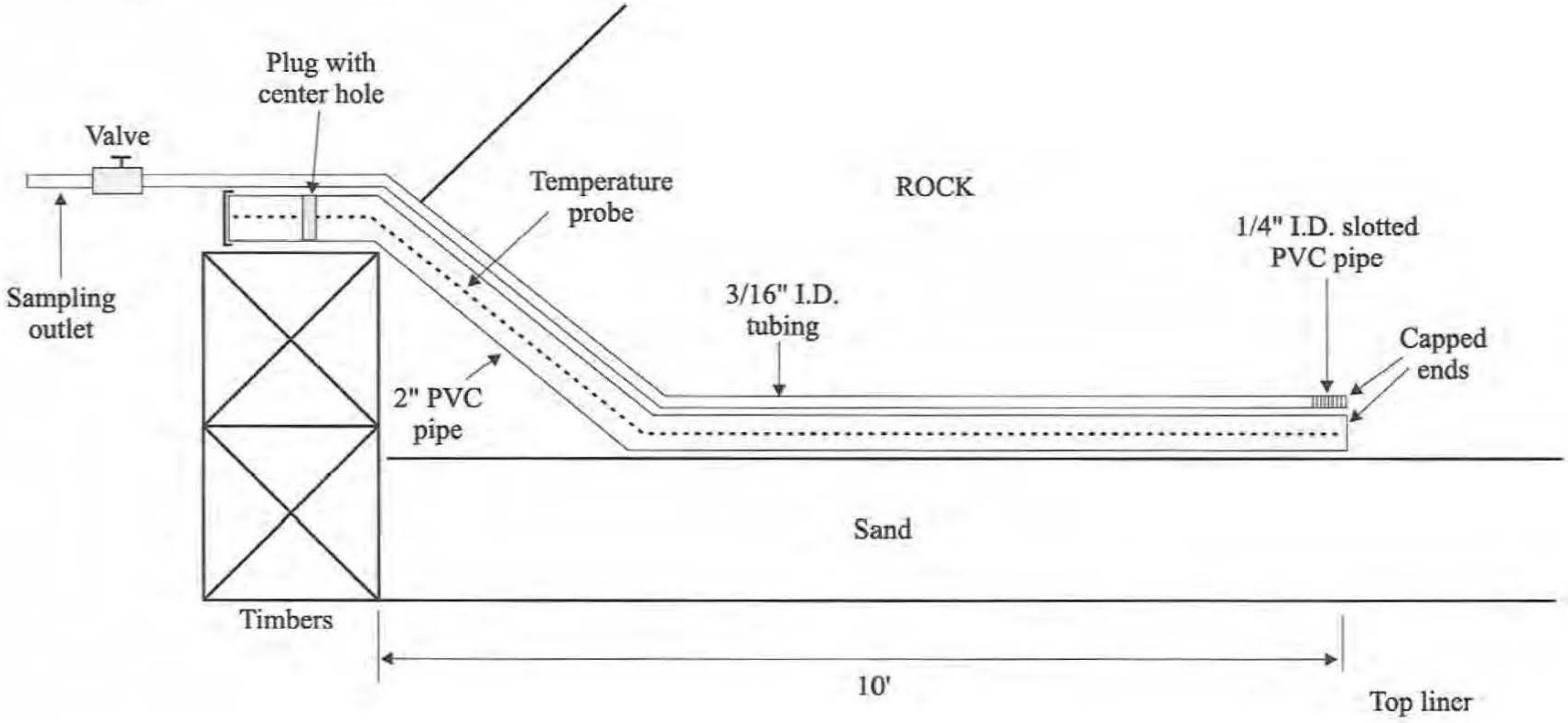


Figure 5. Temperature and oxygen sampling setup.



Note: Not illustrated is a second oxygen sampling port that extends 5 feet into the pile.

Figure 6. Initial rock addition and oxygen sampling setup.

Figure 7. Photograph of test pile construction.

Figure 8. Rock pile design for greenstone field experiment (not to scale).

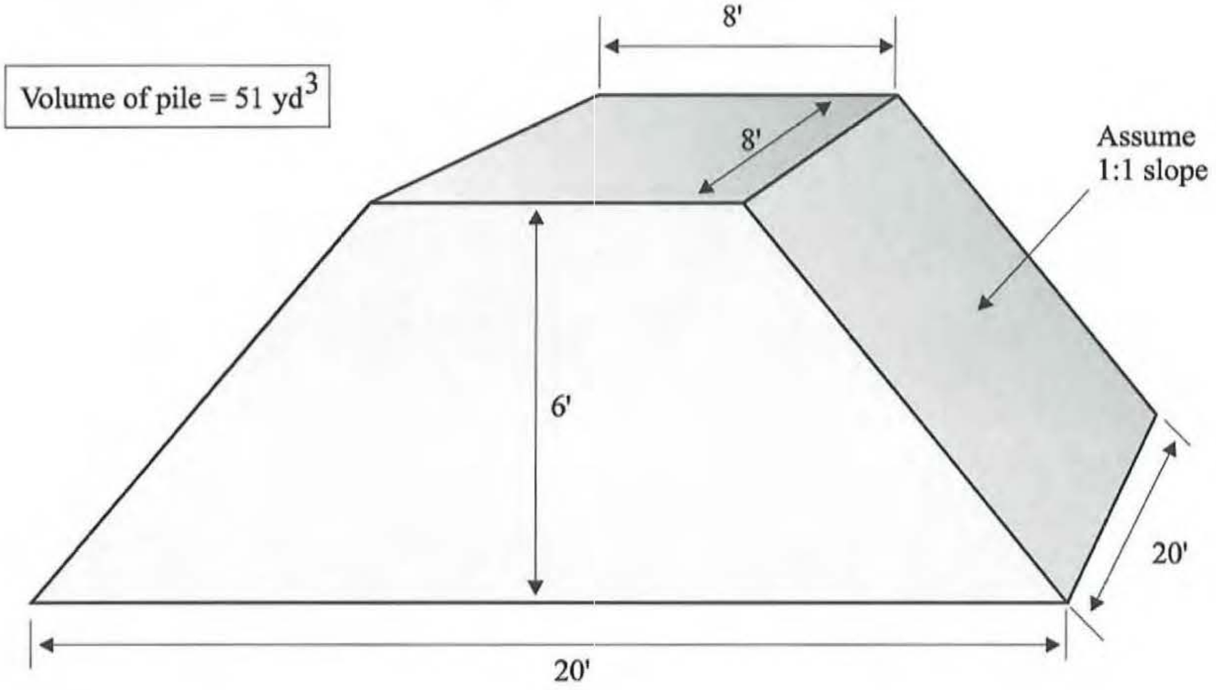


Figure 9. Temperature and oxygen sampling design (not to scale).

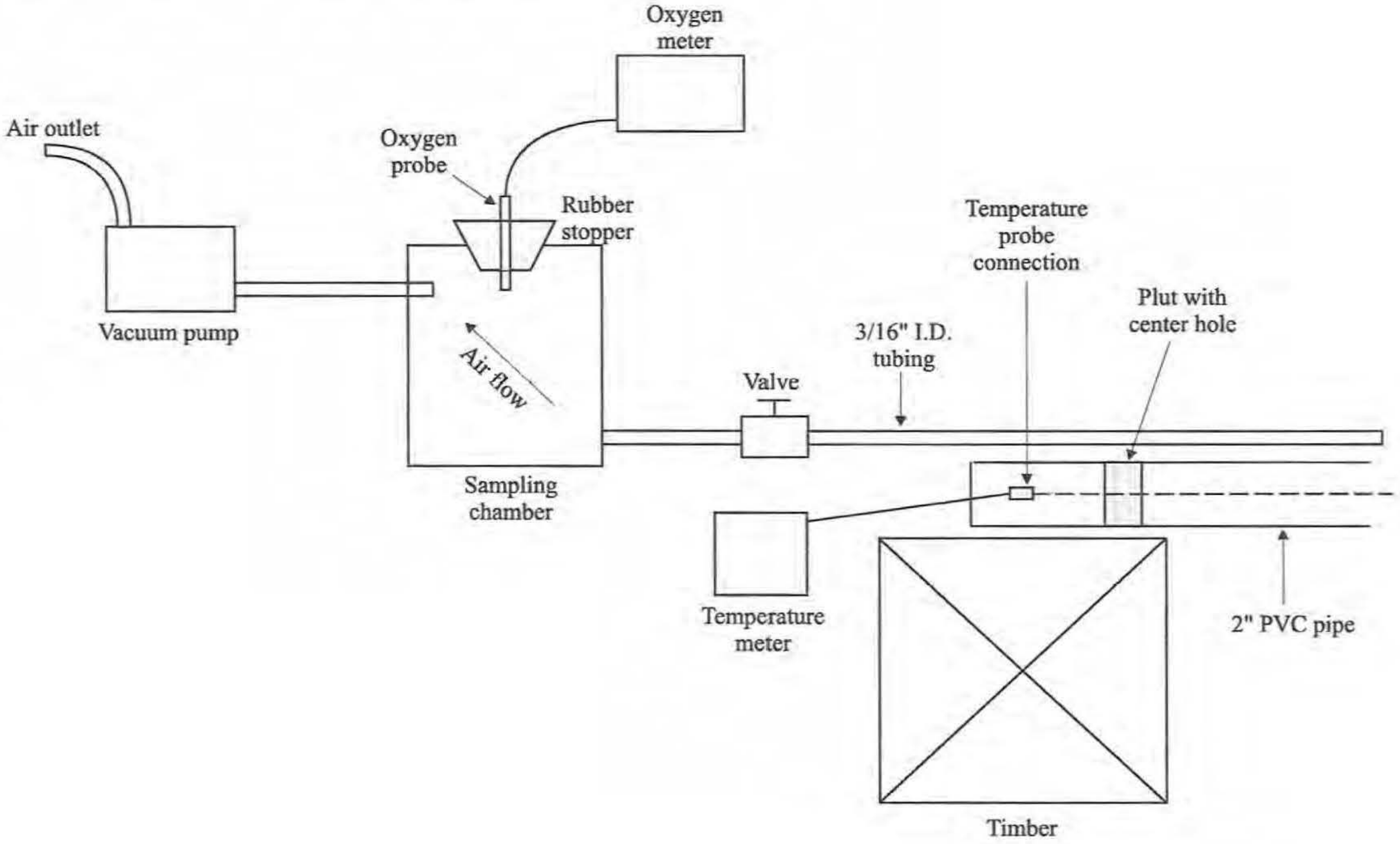


Figure 10. Greenstone rock used for the limestone addition experiment.

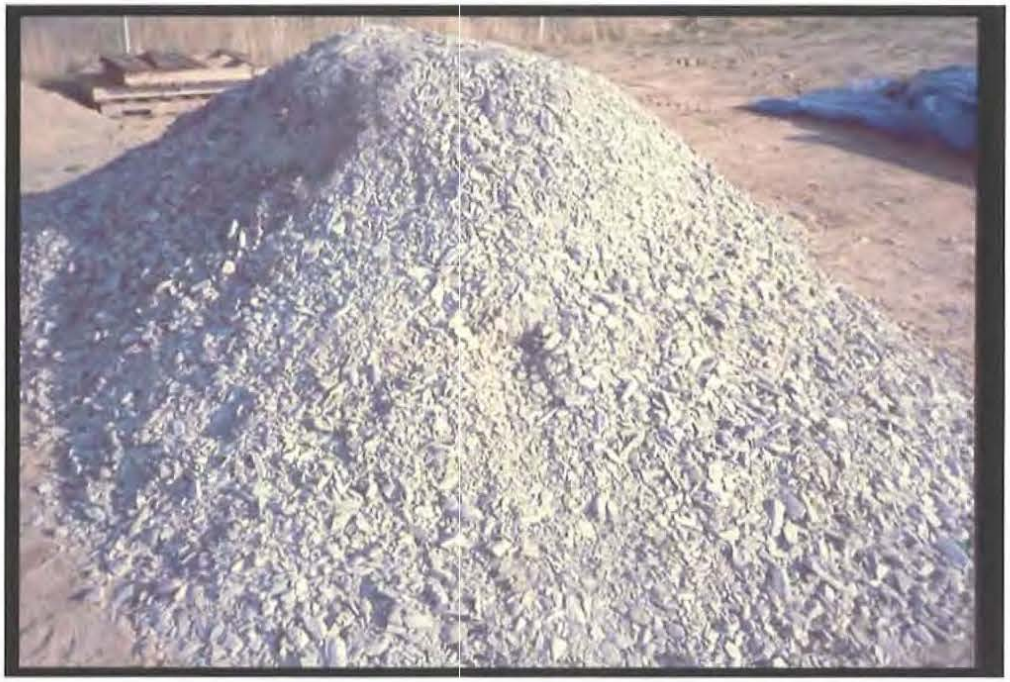


Figure 11. Rock in tank, 2001.

Figure 12. Adding rock and limestone to tanks. The material was scooped from the bucket into the tank.

Figure 13. Limestone used for the alkaline mixture experiment. The limestone product was purchased from Agrrerate Resources' Larson Mine on Grey Cloud Island and is called a manufactured sand

Figure 14. Adding limestone to the loader bucket. Limestone was added to the top and distributed over the material.

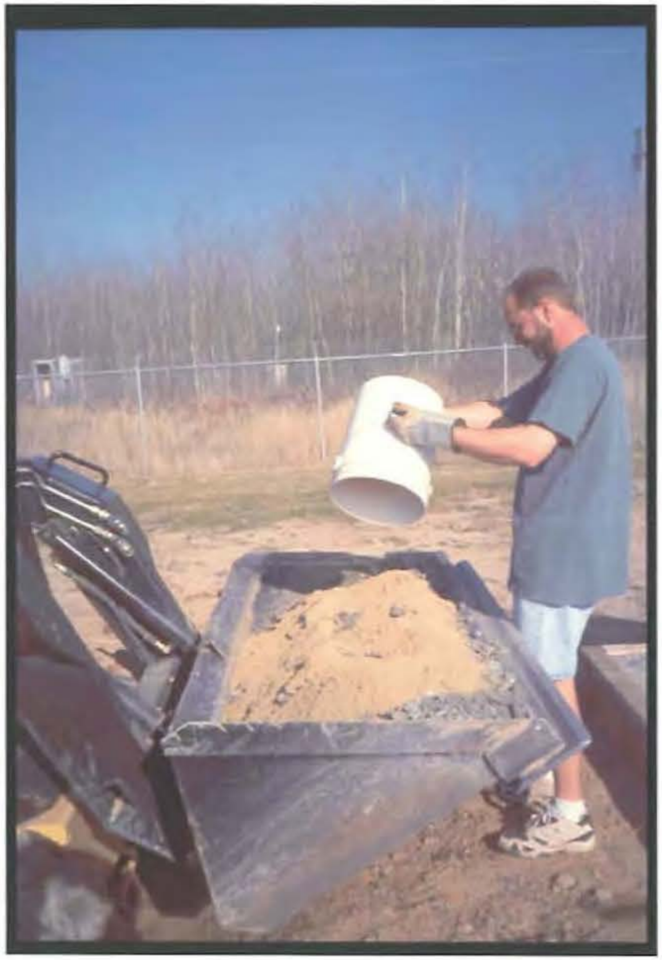


Figure 15. Limestone in loader after addition.

Figure 16. Limestone addition tanks set up with double containment.

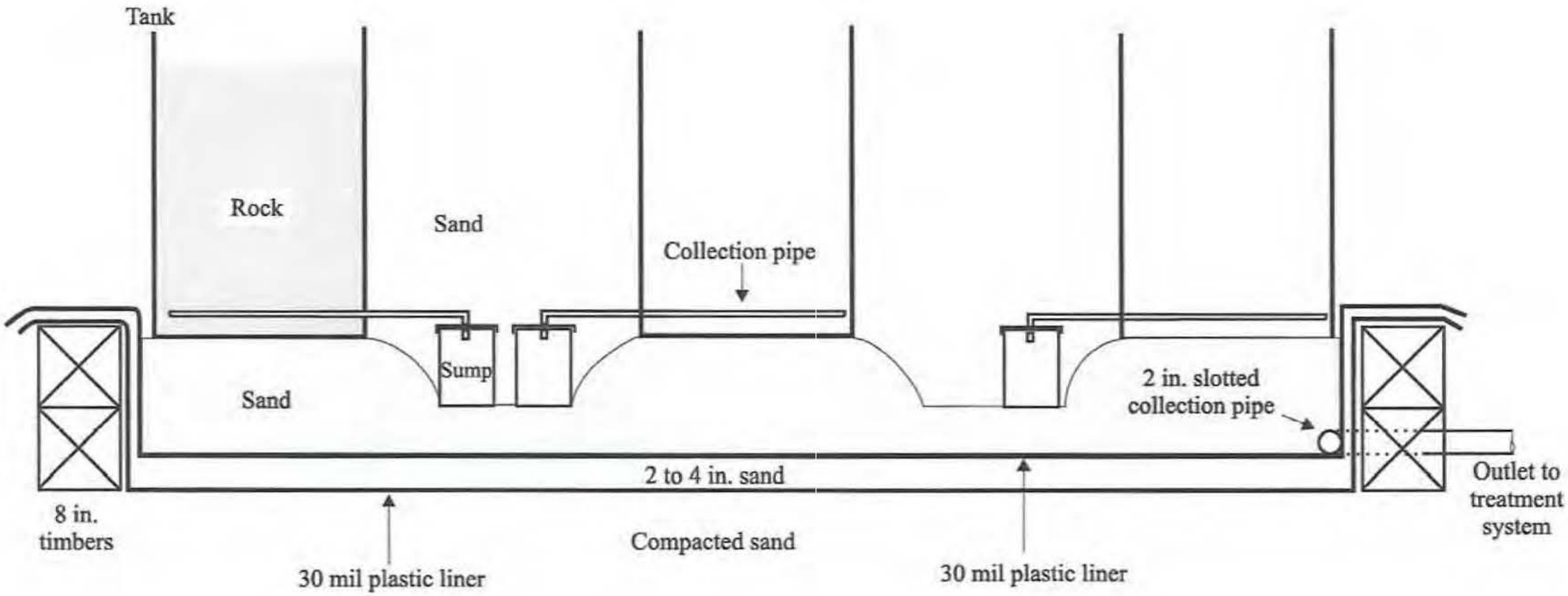


Figure 17. Alkaline mixture, experimental setup, 2001.

Figure 18. Details of limestone addition tank setup.

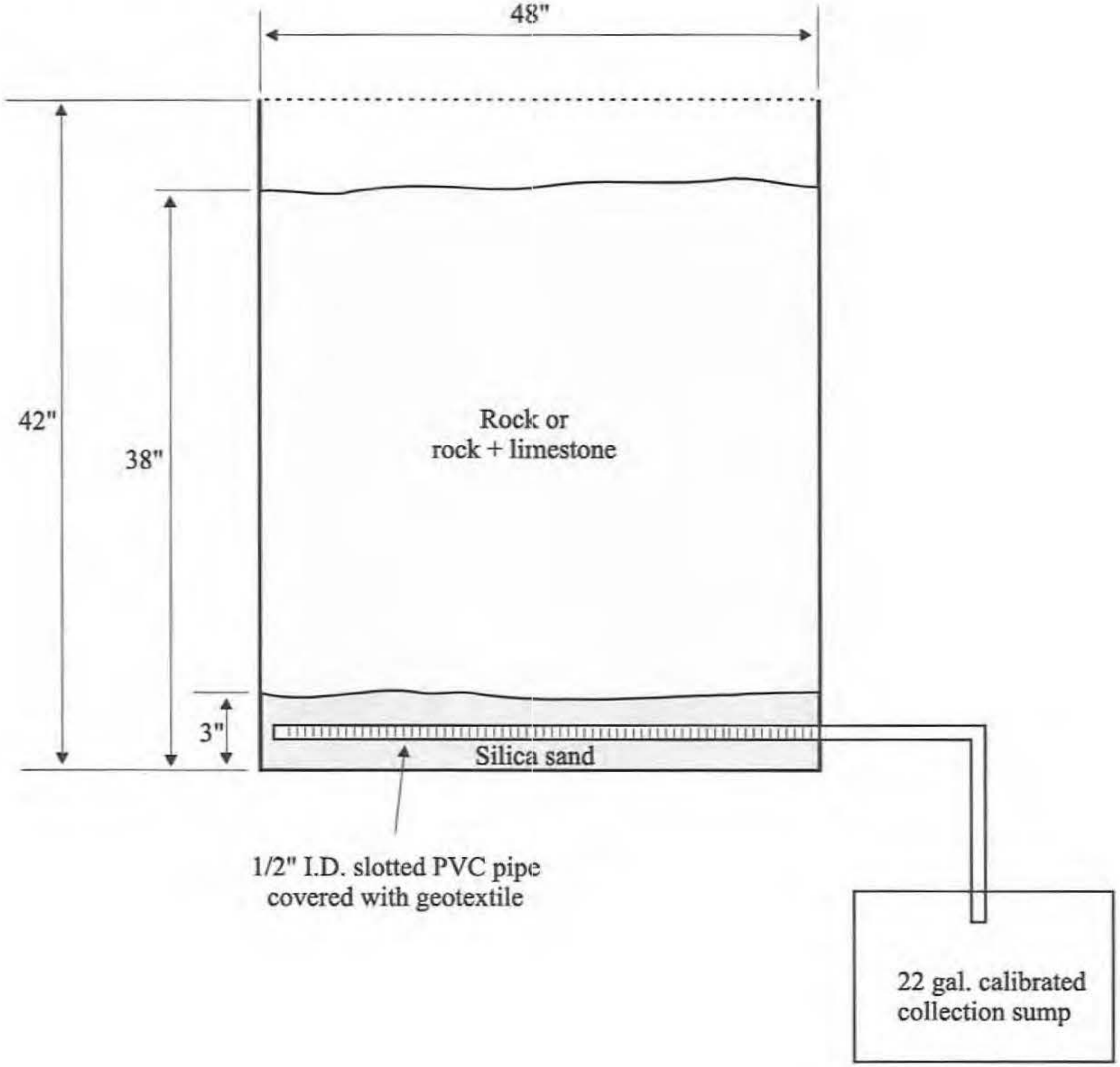


Figure 19. Drainage quality vs. time for the 0.02% S prediction field pile 1.

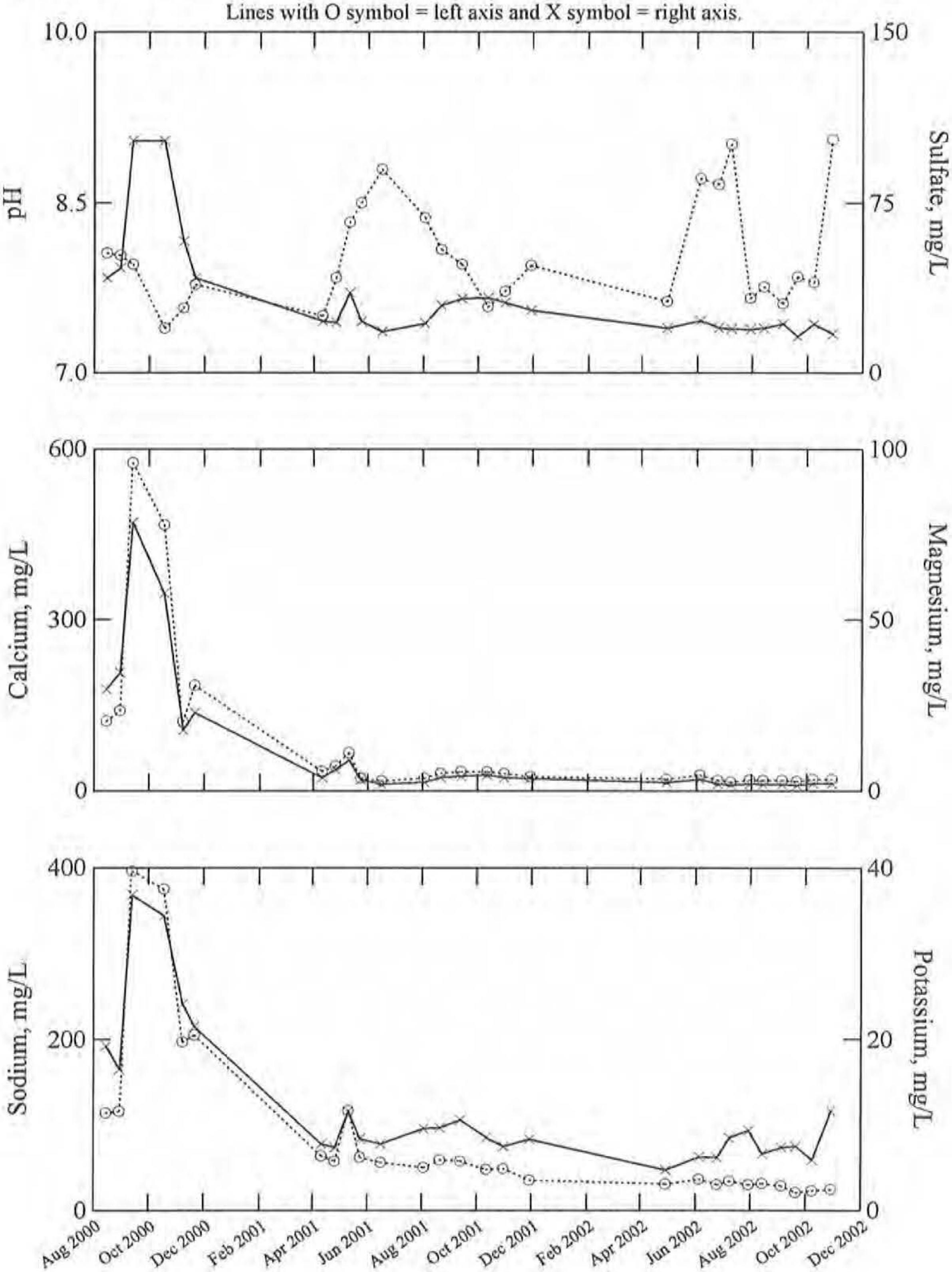


Figure 20. Drainage quality vs. time for the 0.20% S prediction field pile 2.

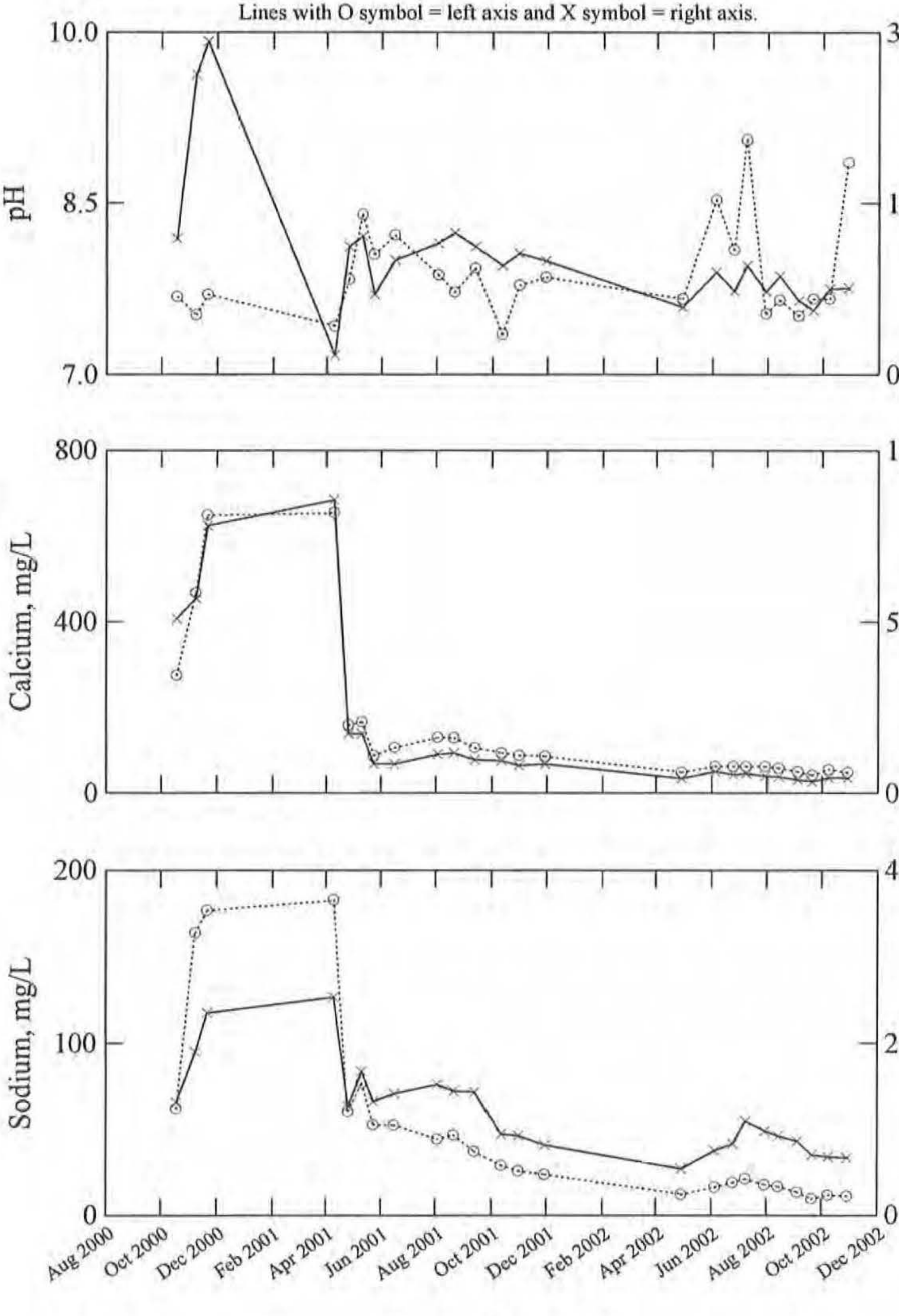


Figure 21. Drainage quality vs. time for the 0.39% S prediction field pile 3.

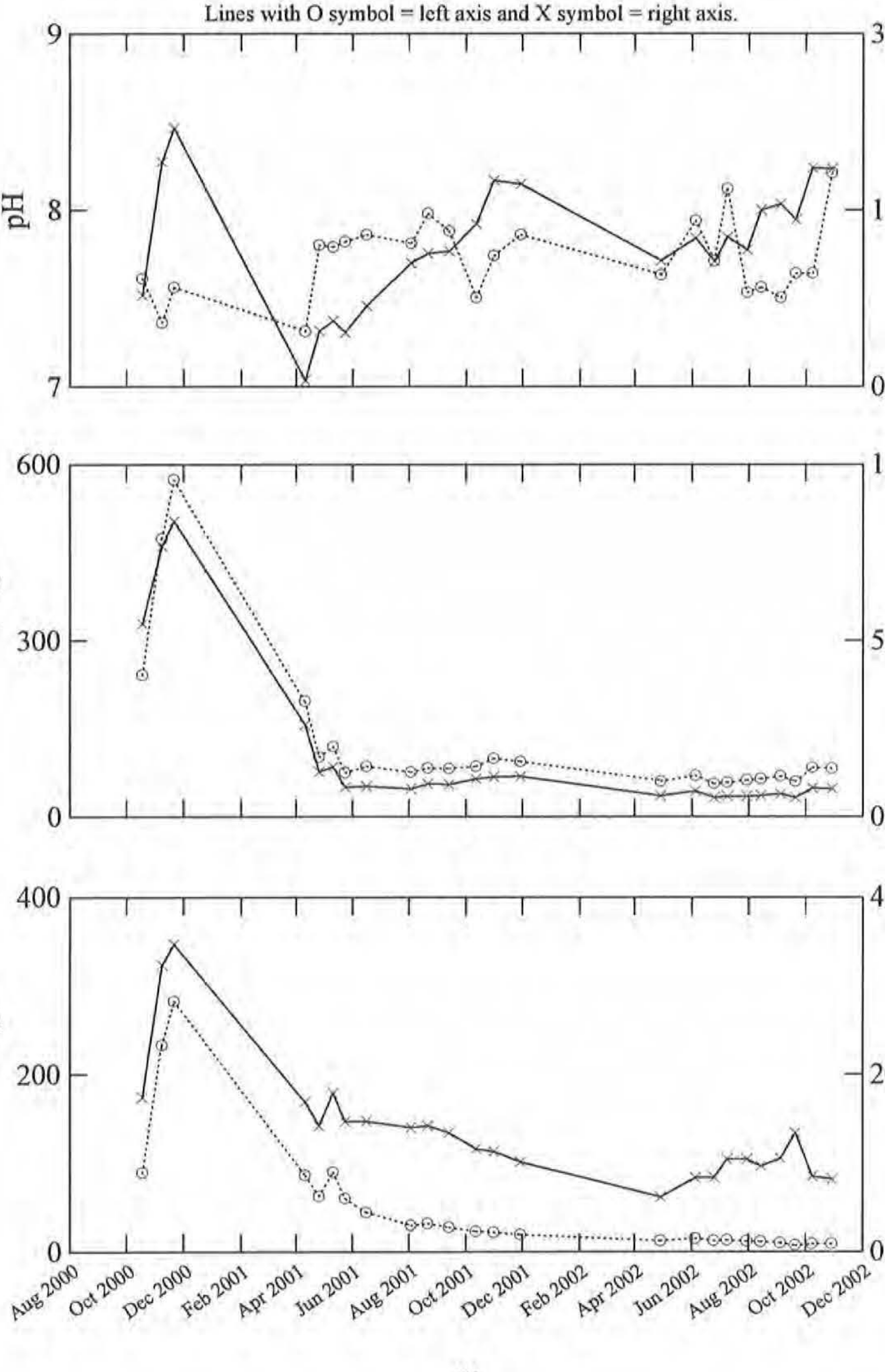


Figure 22. Drainage quality vs. time for the 0.67% S prediction field pile 4.

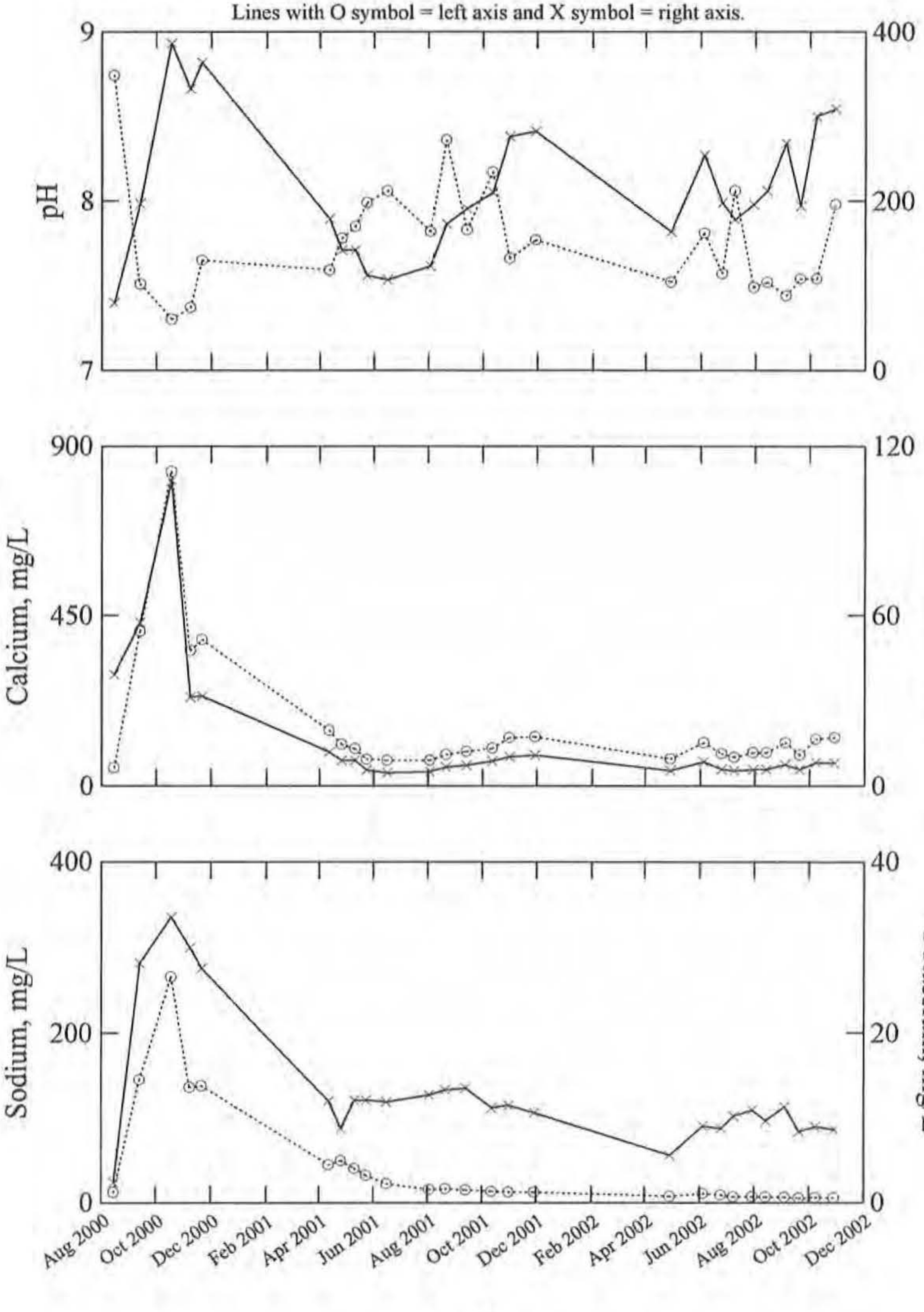


Figure 23. Average 2002 sulfate release rates increased linearly with solid phase sulfur content.

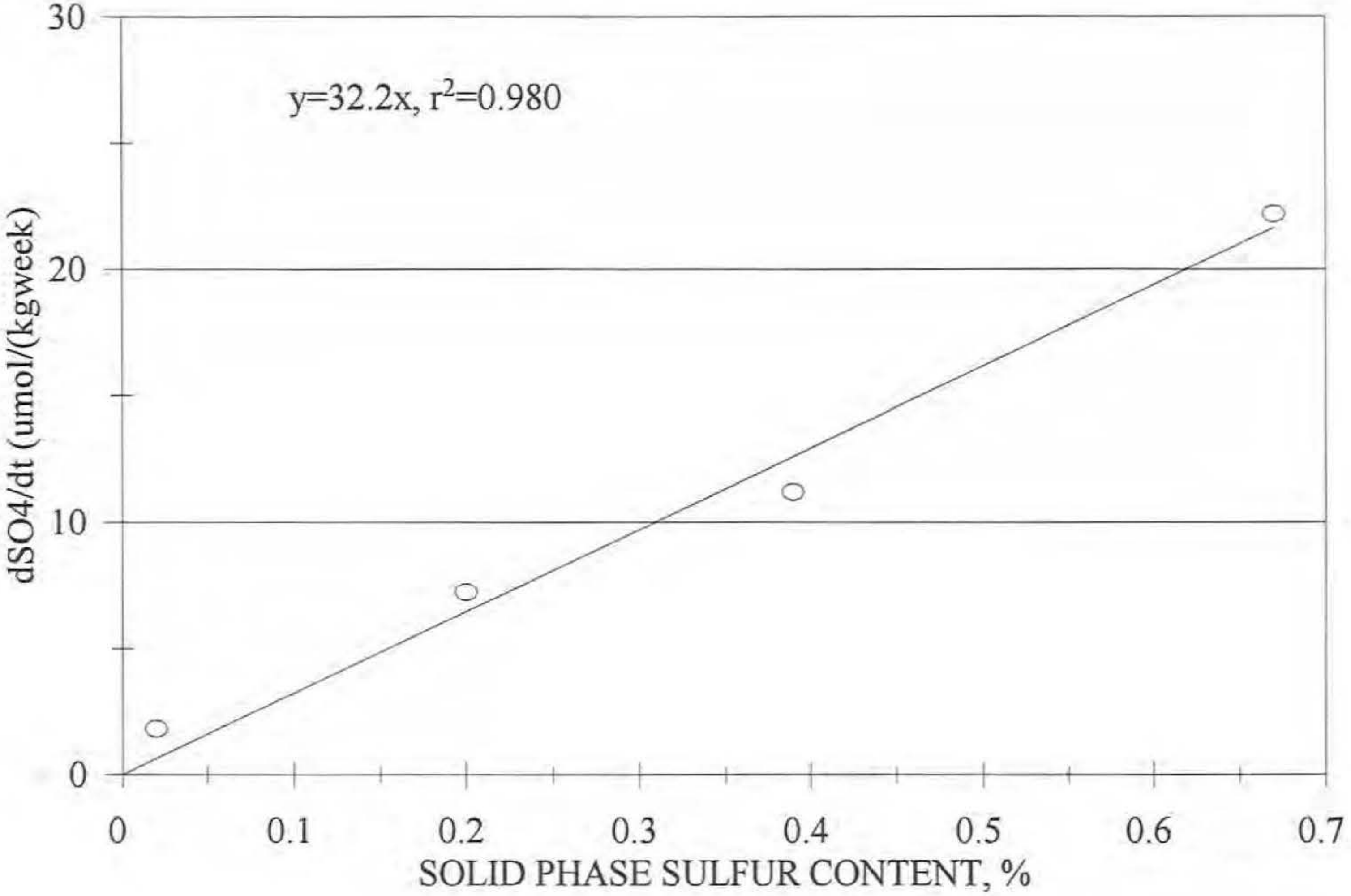


Figure 24. Drainage quality vs. time for the limestone addition control tank 1.

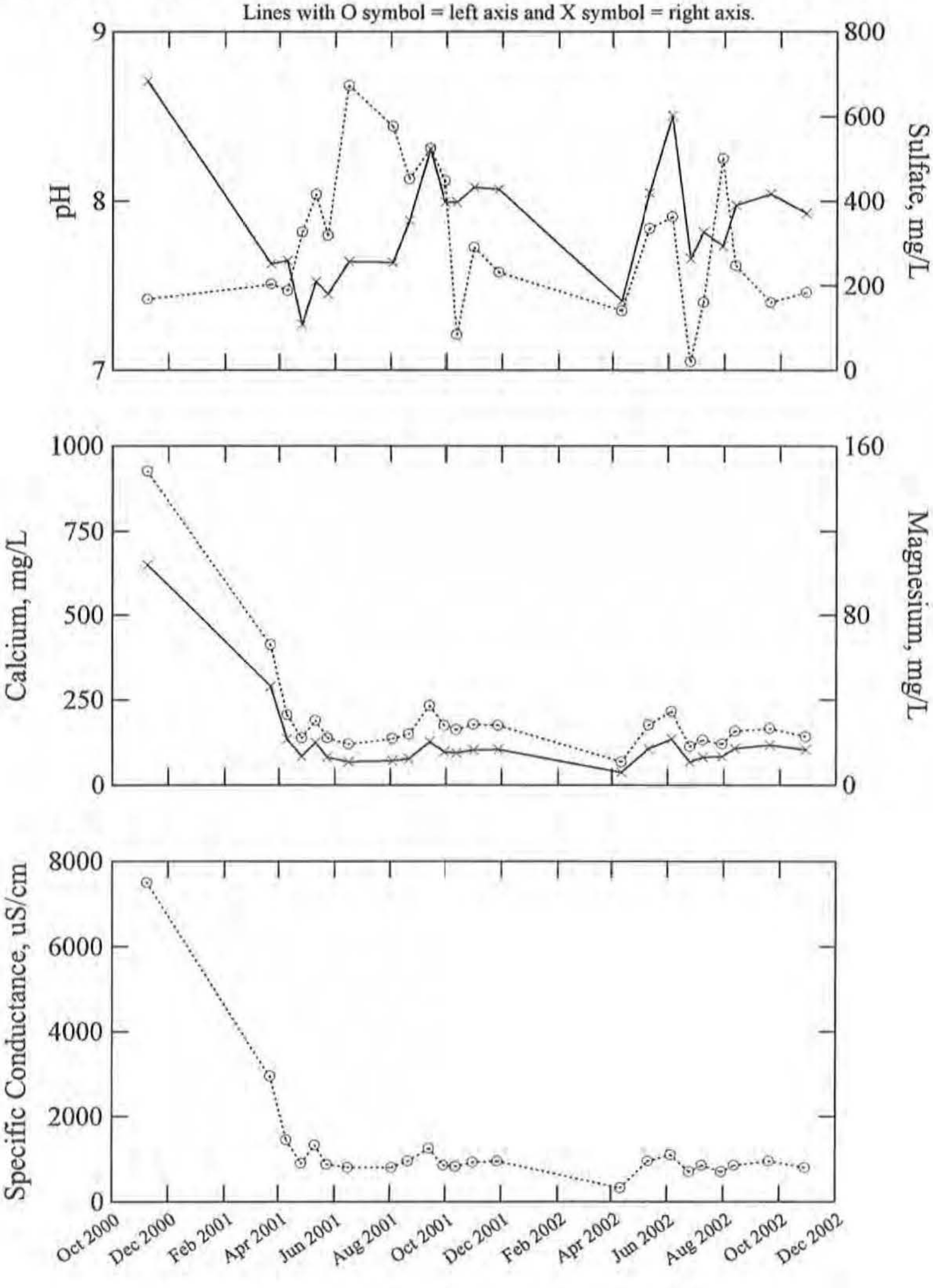


Figure 25. Drainage quality vs. time for the limestone addition control tank 6.

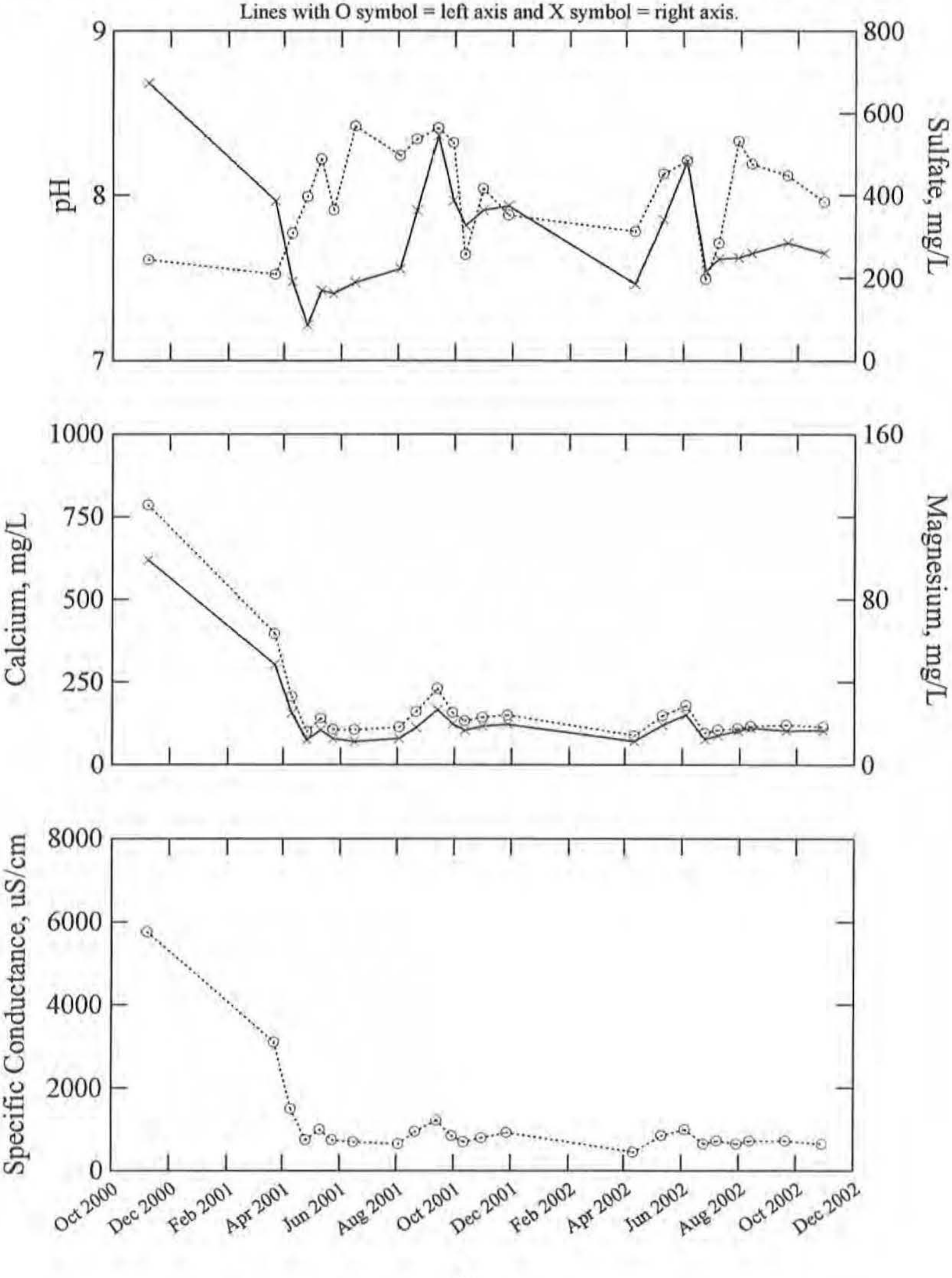


Figure 26. Drainage quality vs. time for the limestone addition 1:1 ratio tank 2.

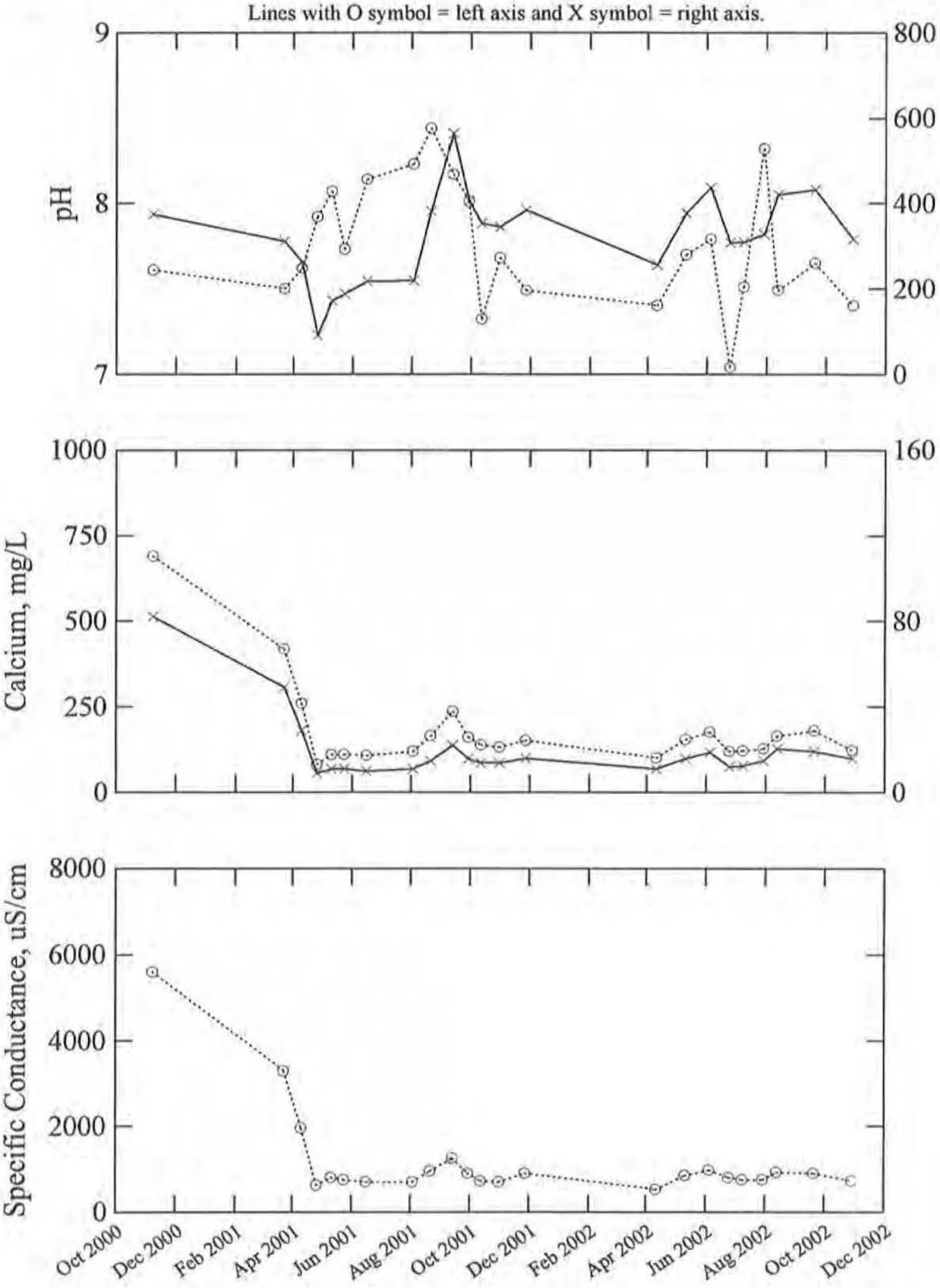


Figure 27. Drainage quality vs. time for the limestone addition 1:1 ratio tank 5.

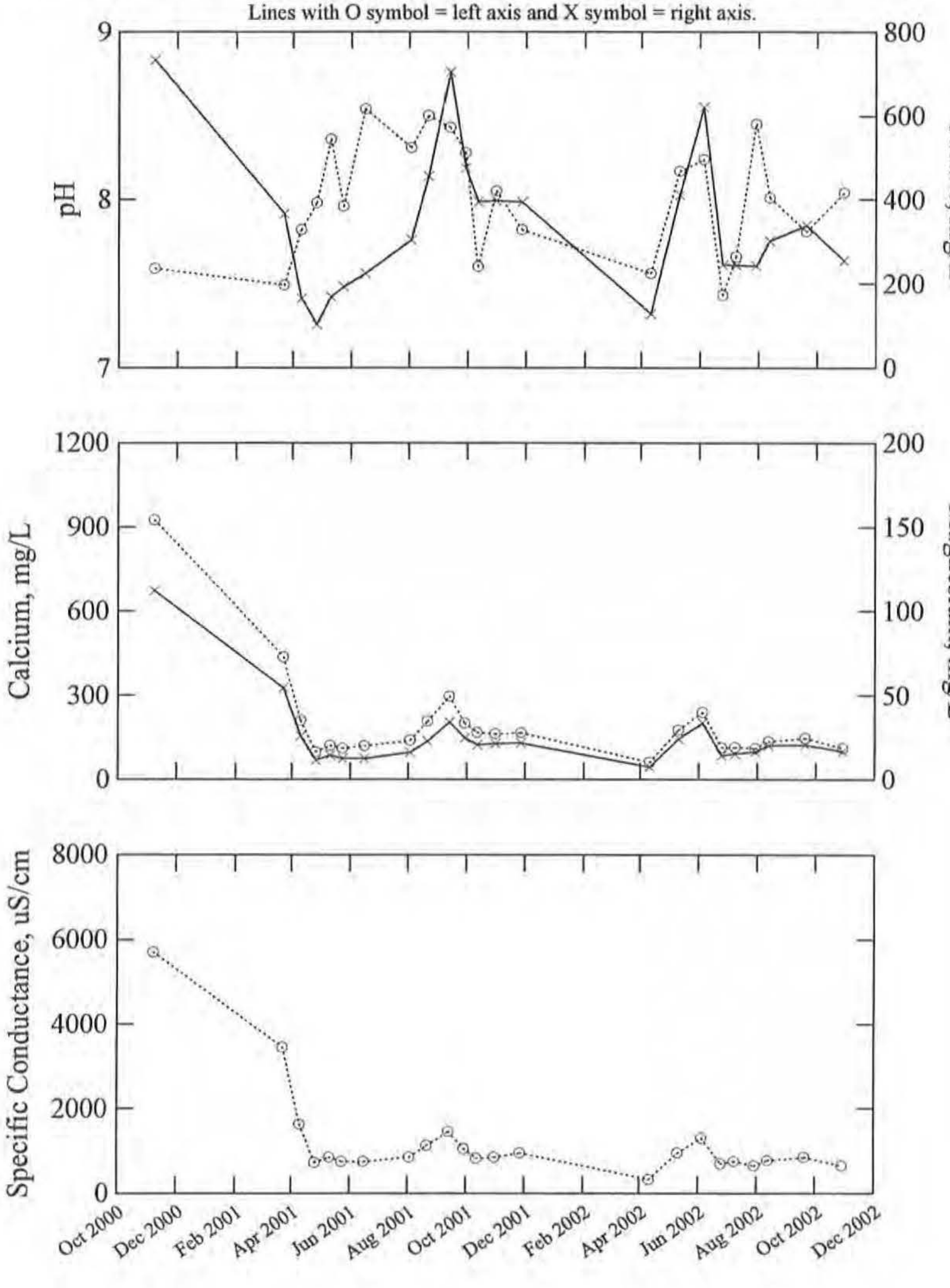


Figure 28. Drainage quality vs. time for the limestone addition 3:1 ratio tank 3.

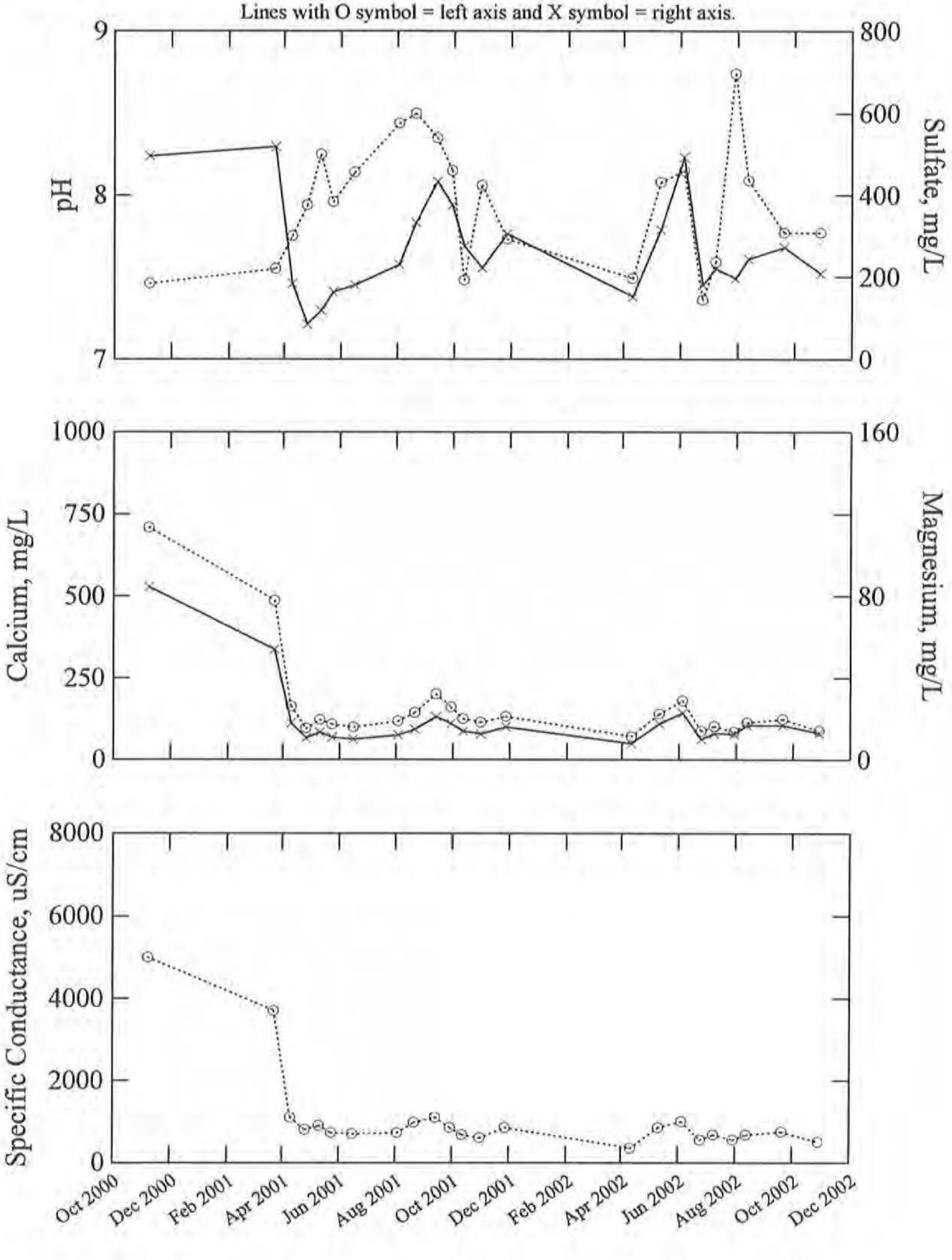
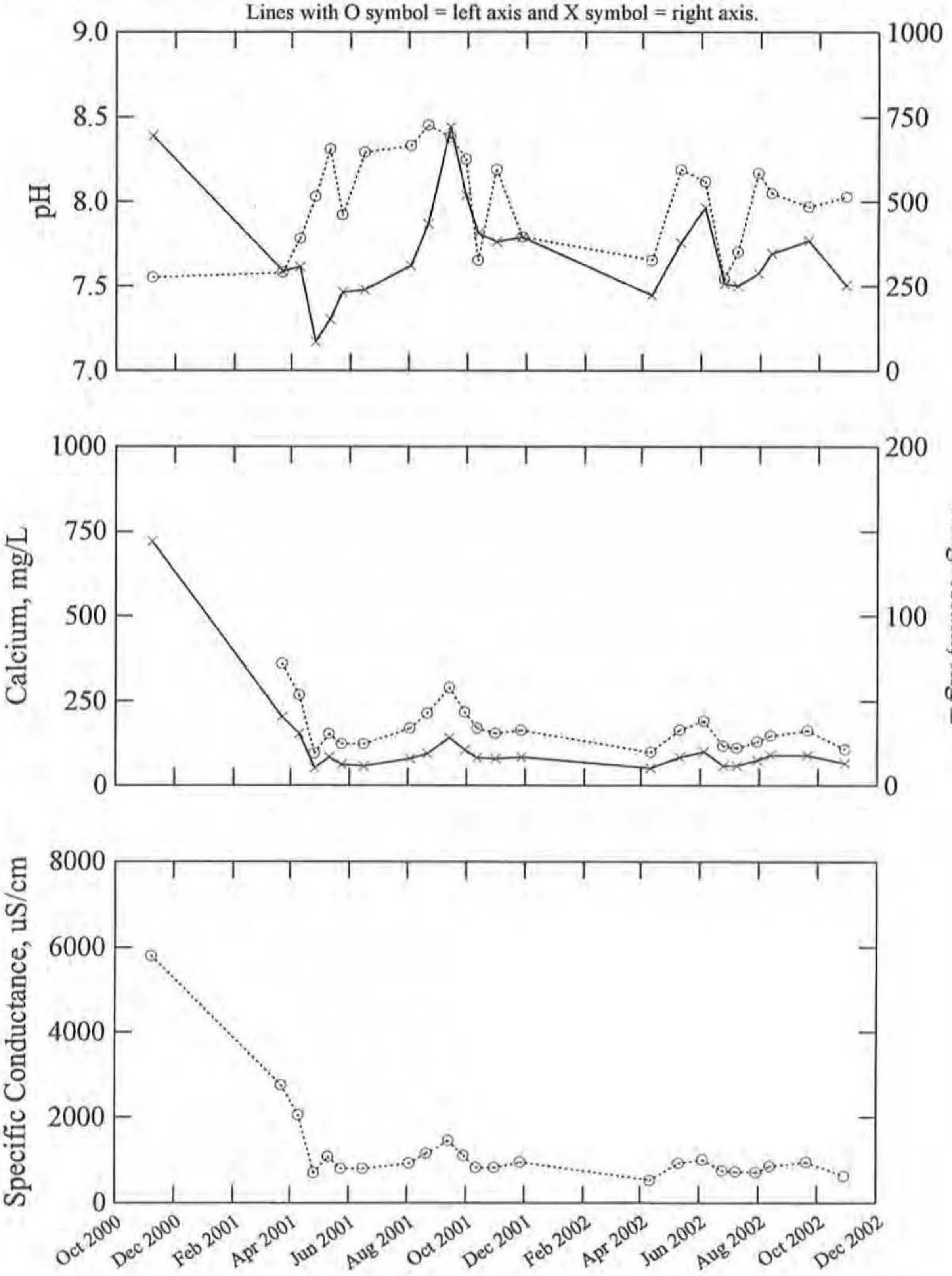


Figure 29. Drainage quality vs. time for the limestone addition 3:1 ratio tank 4.



APPENDIX 1

SOLIDS COMPOSITION OF ROCK FROM PREDICTION PILES AND LIMESTONE ADDITIION TANKS

Table A.1.1. Sulfur (piles 1 - 4) and CO_2 (pile 4) analyses from samples taken from the muck boxes. Analyses by Lerch Bros.

Sample #	Pile 1/ 0.02% Sulfur	Sample #	Pile 2/ 0.22% Sulfur	Sample #	Pile 3/ 0.39% Sulfur	Sample #	Pile 4/ 0.67% Sulfur	Pile 4/ CO_2
2-20	0.005	3-18	0.08	4-10	0.20	1-13	0.29	0.60
2-10	0.006	3-17	0.10	4-13	0.20	1-15	0.33	0.28
2-4	0.01	3-21	0.13	4-15	0.22	1-19	0.42	0.09
2-6	0.01	3-6	0.13	4-2	0.23	1-5	0.45	0.17
2-9	0.01	3-19	0.13	4-14	0.23	1-3	0.46	0.42
2-14	0.01	3-20	0.14	4-4	0.24	1-1	0.47	0.31
2-16	0.01	3-8	0.18	4-11	0.24	1-14	0.48	1.24
2-18	0.01	3-10	0.18	4-7	0.25	1-18	0.50	0.24
2-19	0.01	3-4	0.19	4-21	0.28	1-23	0.54	0.67
2-21	0.01	3-13	0.19	4-3	0.29	1-6	0.58	0.15
2-22	0.01	3-1	0.20	4-20	0.29	1-8	0.59	0.28
2-23	0.01	3-7	0.20	4-17	0.30	1-22	0.64	0.22
2-25	0.01	3-3	0.21	4-22	0.32	1-24	0.66	0.33
2-7	0.02	3-14	0.21	4-19	0.33	1-11	0.69	0.71
2-8	0.02	3-12	0.21	4-12	0.34	1-7	0.74	1.43
2-11	0.02	3-9	0.21	4-24	0.36	1-25	0.75	0.32
2-12	0.02	3-25	0.22	4-25	0.36	1-12	0.76	0.29
2-15	0.02	3-2	0.25	4-1	0.39	1-20	0.77	0.30
2-24	0.02	3-22	0.26	4-23	0.41	1-16	0.79	0.34
2-13	0.03	3-11	0.32	4-6	0.49	1-19	0.80	0.09
2-17	0.03	3-23	0.32	4-16	0.50	1-10	0.94	1.66
2-1	0.04	3-5	0.33	4-18	0.51	1-17	0.94	0.21
2-5	0.05	3-15	0.37	4-9	0.61	1-2	1.02	0.15
2-3	0.07	3-16	0.40	4-5	0.81	1-9	1.04	0.23
2-2	0.08	3-24	0.46	4-8	1.47	1-4	1.09	0.68

Table A.1.2. Summary statistics for percent sulfur on the 25 initial samples taken from the muck boxes.

Statistic	Pile 1/ 0.02% S	Pile 2/ 0.22% S	Pile 3/ 0.39%S	Pile 4/ 0.67% S
N of cases	25	25	25	25
Minimum	0.005	0.08	0.20	0.29
Maximum	0.08	0.46	1.47	1.09
Median	0.01	0.21	0.32	0.66
Mean	0.02	0.22	0.39	0.67
95% CI Upper	0.03	0.26	0.50	0.76
95% CI Lower	0.01	0.19	0.28	0.58
Standard Dev.	0.019	0.095	0.266	0.223

Table A1.3. Whole rock chemistry for the 0.02% S sample of greenstone. Analysis by ACTLABS.

Sample ID	S %	SO_4 %	CO_2 %	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	MnO %	MgO %	CaO %	Na_2O %	K_2O %	TiO_2 %	P_2O_5 %	LOI %	Total %
2-1	0.04	0.02	0.66	52.57	18.52	12.64	0.147	5.75	0.97	0.47	2.36	0.823	0.27	5.71	100.23
2-2	0.08		0.55	52.58	19.06	11.47	0.158	5.77	0.98	0.43	2.69	0.853	0.35	5.85	100.17
2-3	<0.01	0.02	0.11	54.49	18.99	10.26	0.126	5.62	0.71	0.44	2.75	0.826	0.39	5.39	99.99
2-4	<0.01		<0.05	54.00	19.59	10.27	0.118	5.69	0.75	0.39	2.77	0.868	0.45	5.48	100.37
2-5	0.04	0.02	0.51	53.37	19.24	10.99	0.167	5.67	0.79	0.40	2.83	0.861	0.32	5.69	100.33
2-6	0.02		0.15	50.85	20.56	11.19	0.125	6.09	0.80	0.42	2.98	0.924	0.49	5.81	100.25
2-7	<0.01	0.02	0.11	52.47	20.34	10.84	0.127	5.68	0.65	0.47	2.98	0.907	0.38	5.42	100.26
2-8	0.01		0.33	53.73	19.24	10.43	0.126	5.67	0.89	0.42	2.91	0.832	0.36	5.43	100.03
2-9	<0.01	0.02	0.07	53.58	19.08	10.62	0.124	5.88	0.87	0.38	2.67	0.838	0.60	5.45	100.09
2-10	<0.01		0.11	50.42	20.70	11.50	0.143	6.40	0.65	0.39	3.02	0.895	0.38	5.85	100.33
2-11	<0.01	0.02	0.11	55.14	18.60	10.27	0.125	5.40	1.14	0.39	2.59	0.800	0.72	5.18	100.34
2-12	<0.01		0.07	53.38	19.61	10.60	0.122	6.16	0.66	0.36	2.66	0.847	0.44	5.66	100.50
2-13	<0.01	0.02	0.07	50.42	20.54	11.71	0.130	6.87	0.58	0.39	2.39	0.913	0.35	6.09	100.39
2-14	<0.01		<0.05	50.82	20.20	11.52	0.125	6.75	0.55	0.40	2.50	0.908	0.37	5.95	100.09
2-15	<0.01	0.02	<0.05	52.46	19.88	11.07	0.122	6.16	0.63	0.45	2.64	0.877	0.41	5.63	100.33
2-16	0.02		0.07	52.50	19.55	11.18	0.128	6.64	0.63	0.38	2.32	0.868	0.40	5.83	100.42
2-17	<0.01	0.02	<0.05	50.25	20.47	11.71	0.140	6.81	0.62	0.42	2.62	0.911	0.39	5.97	100.31
2-18	<0.01		<0.05	51.46	20.09	11.39	0.129	6.57	0.65	0.34	2.60	0.878	0.40	5.87	100.38
2-19	<0.01	0.02	0.07	51.30	20.08	11.33	0.130	6.62	0.66	0.39	2.56	0.877	0.40	5.91	100.26
2-20	<0.01		<0.05	50.91	20.35	11.28	0.131	6.98	0.58	0.39	2.25	0.896	0.36	6.03	100.17
2-21	<0.01	0.02	0.48	52.07	19.18	11.42	0.160	6.49	0.88	0.33	2.58	0.834	0.29	6.17	100.41
2-22	<0.01		<0.05	49.91	20.58	12.06	0.136	7.36	0.50	0.40	2.06	0.913	0.30	6.28	100.50
2-23	<0.01	0.02	0.07	50.86	19.59	12.20	0.124	7.36	0.48	0.43	1.67	0.877	0.30	6.27	100.16
2-24	<0.01		<0.05	50.99	19.67	11.90	0.131	7.26	0.72	0.39	1.95	0.864	0.46	6.03	100.37
2-25	<0.01	0.02	<0.05	50.78	19.72	11.89	0.132	7.11	0.73	0.38	2.06	0.872	0.49	6.16	100.33

Table A1.4. Whole rock chemistry for the 0.20% S sample of greenstone. Analysis by ACTLABS.

Sample ID	**S %**	**SO_4 %**	**CO_2 %**	**SiO_2 %**	**Al_2O_3 %**	**Fe_2O_3 %**	**MnO %**	**MgO %**	**CaO %**	**Na_2O %**	**K_2O %**	**TiO_2 %**	**P_2O_5 %**	**LOI %**	**Total %**
3-1	0.20		0.59	60.07	14.83	10.84	0.111	5.07	1.29	0.39	1.75	0.663	0.27	5.05	100.34
3-2	0.24	0.09	0.18	59.62	14.26	11.26	0.099	6.10	0.93	0.25	1.33	0.644	0.43	5.28	100.21
3-3	0.19		0.22	59.90	15.21	10.38	0.099	5.81	0.72	0.27	1.67	0.688	0.35	5.28	100.37
3-4	0.18	0.02	0.18	65.68	13.23	9.41	0.081	3.68	0.52	0.39	1.68	0.550	0.19	4.11	99.53
3-5	0.33		<0.05	61.29	14.50	10.44	0.093	4.90	0.61	0.35	1.48	0.625	0.22	4.75	99.26
3-6	0.14	0.02	<0.05	61.48	14.25	10.17	0.095	6.02	0.70	0.26	1.26	0.645	0.42	4.93	100.23
3-7	0.20		<0.05	62.74	14.43	9.13	0.077	5.05	0.67	0.24	1.76	0.632	0.43	4.47	99.62
3-8	0.17	0.05	<0.05	58.05	15.55	11.11	0.099	6.12	0.83	0.20	1.67	0.745	0.54	5.14	100.05
3-9	0.19		<0.05	60.01	13.59	10.90	0.097	5.83	0.82	0.26	1.34	0.610	0.49	4.70	98.64
3-10	0.16	0.02	<0.05	59.56	15.23	10.71	0.096	5.92	0.64	0.25	1.53	0.691	0.44	4.92	99.98
3-11	0.22		<0.05	61.45	15.30	9.33	0.081	4.55	0.84	0.42	1.91	0.746	0.47	4.37	99.47
3-12	0.35	0.02	<0.05	61.06	13.88	9.90	0.078	6.30	0.93	0.27	1.33	0.615	0.44	5.04	99.84
3-13	0.19		0.18	60.76	14.86	10.19	0.095	5.09	1.07	0.28	1.86	0.685	0.48	4.75	100.13
3-14	0.19	0.02	0.11	60.43	15.07	10.02	0.091	5.02	0.90	0.32	1.99	0.724	0.46	4.55	99.57
3-15	0.36		0.29	62.05	14.23	9.23	0.078	4.93	0.89	0.27	1.86	0.623	0.39	4.56	99.11
3-16	0.40	0.02	0.18	60.16	14.61	10.56	0.084	5.94	0.72	0.24	1.63	0.619	0.45	5.22	100.23
3-17	0.11		0.18												
3-18	0.07	0.02	0.18	53.85	17.31	11.60	0.114	7.22	0.71	0.24	1.94	0.804	0.56	5.77	100.12
3-19	0.14		0.11	58.01	16.57	9.90	0.086	6.23	0.72	0.28	2.02	0.728	0.41	5.13	100.09
3-20	0.14	0.02	0.11	59.06	16.66	9.49	0.089	5.70	0.56	0.27	2.38	0.760	0.38	4.97	100.33
3-21	0.14		<0.05	66.11	13.56	9.11	0.084	3.51	0.44	0.23	1.99	0.532	0.29	3.73	99.58
3-22	0.24	0.02	2.70	59.39	16.84	10.26	0.104	4.56	0.50	0.24	2.64	0.703	0.28	4.91	100.43
3-23	0.32		0.29	62.11	15.85	9.36	0.092	4.03	0.56	0.31	2.52	0.672	0.31	4.53	100.33
3-24	0.44	0.02	0.15	62.47	14.80	9.39	0.089	4.24	0.61	0.28	2.22	0.688	0.40	4.36	99.54
3-25	0.23		0.29	62.85	14.32	9.49	0.099	4.36	1.18	0.29	2.07	0.611	0.76	4.26	100.29

Table A1.5. Whole rock chemistry for the 0.39% S sample of greenstone. Analysis by ACTLABS.

Sample ID	S %	SO_4 %	CO_2 %	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	MnO %	MgO %	CaO %	Na_2O %	K_2O %	TiO_2 %	P_2O_5 %	LOI %	Total %
4-1	0.40	0.02	0.18	68.63	12.49	8.04	0.076	4.10	0.38	0.42	1.28	0.504	0.21	4.05	100.18
4-2	0.21		0.11	67.38	12.89	8.76	0.078	3.70	0.34	0.36	1.41	0.520	0.21	3.65	99.29
4-3	0.27	0.02	<0.05	63.14	15.25	8.69	0.067	4.11	0.45	0.42	2.04	0.609	0.28	4.05	99.11
4-4	0.24		<0.05	64.84	14.43	8.37	0.068	3.98	0.60	0.35	2.01	0.569	0.48	3.85	99.55
4-5	0.75	0.02	<0.05	68.58	13.34	7.88	0.058	3.10	0.37	0.39	1.98	0.486	0.24	3.66	100.07
4-6	0.48		<0.05	66.88	14.90	7.57	0.057	2.88	0.35	0.38	2.60	0.596	0.24	3.53	99.96
4-7	0.23	0.02	<0.05	70.34	13.00	6.93	0.058	2.83	0.32	0.30	2.25	0.491	0.22	3.23	99.96
4-8	1.33		<0.05	76.72	8.06	8.49	0.052	2.17	0.25	0.27	0.85	0.335	0.18	3.01	100.39
4-9	0.57	0.10	<0.05	70.65	12.06	7.10	0.052	2.91	0.34	0.29	1.88	0.515	0.24	3.26	99.31
4-10	0.19		0.07	60.98	14.81	10.49	0.083	5.65	0.70	0.39	1.49	0.672	0.48	4.54	100.28
4-11	0.22	0.02	<0.05	65.39	14.92	8.21	0.067	3.24	0.41	0.42	2.32	0.628	0.27	3.67	99.55
4-12	0.32		<0.05	68.83	12.26	8.51	0.065	3.71	0.54	0.31	1.57	0.509	0.39	3.49	100.18
4-13	0.20	0.02	<0.05	69.19	13.24	8.27	0.072	2.47	0.30	0.29	2.10	0.507	0.21	3.20	99.85
4-14	0.22		<0.05	65.93	14.66	8.66	0.071	3.37	0.45	0.41	1.95	0.647	0.29	3.65	100.08
4-15	0.22	0.04	<0.05	65.13	14.54	8.90	0.079	3.23	0.36	0.44	2.08	0.626	0.22	3.68	99.28
4-16	0.50		0.11	68.32	12.46	9.33	0.077	3.09	0.32	0.33	1.52	0.535	0.23	3.44	99.67
4-17	0.28	0.02	<0.05	66.12	13.06	9.45	0.079	3.76	0.43	0.34	1.59	0.599	0.30	3.71	99.43
4-18	0.46		<0.05	66.59	13.38	9.50	0.078	3.08	0.36	0.38	1.65	0.562	0.27	3.62	99.47
4-19	0.34	0.06	<0.05	71.54	12.15	7.81	0.071	2.59	0.25	0.34	1.79	0.439	0.16	3.04	100.18
4-20	0.28		<0.05	67.10	12.71	10.72	0.094	2.95	0.30	0.34	1.29	0.613	0.21	3.38	99.72
4-21	0.26	0.02	0.11	68.82	13.08	8.49	0.073	2.95	0.41	0.52	1.47	0.557	0.20	3.40	99.97
4-22	0.30		0.07	68.55	13.45	8.28	0.065	2.72	0.27	0.53	1.63	0.517	0.18	3.24	99.43
4-23	0.36	0.02	0.15	70.08	12.39	8.59	0.073	2.72	0.30	0.56	1.45	0.501	0.21	3.20	100.06
4-24	0.35		0.15	71.77	10.95	8.85	0.084	2.89	0.29	0.45	1.03	0.435	0.27	3.09	100.13
4-25	0.36	0.02	0.37	66.38	12.73	10.66	0.131	2.93	0.35	0.63	1.31	0.486	0.23	3.63	99.47

Table A1.6. Whole rock chemistry for the 0.67% S sample of greenstone. Analysis by ACTLABS.

Sample ID	S %	SO_4 %	CO_2 %	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	MnO %	MgO %	CaO %	Na_2O %	K_2O %	TiO_2 %	P_2O_5 %	LOI %	Total %
1-1	0.48		0.29	66.52	12.80	9.92	0.066	3.43	0.33	0.28	1.96	0.494	0.09	3.61	99.51
1-2	0.91	0.02	0.07	66.15	12.76	10.61	0.059	3.62	0.21	0.25	1.78	0.538	0.11	3.85	99.95
1-3	0.44		0.48	63.13	13.79	11.67	0.100	3.73	0.30	0.17	1.86	0.696	0.18	4.11	99.74
1-4	1.02	0.02	0.66	64.30	12.71	11.14	0.118	3.38	0.25	0.25	1.85	0.519	0.69	4.53	99.75
1-5	0.45		0.07	64.74	13.64	10.28	0.058	4.07	0.25	0.19	1.87	0.598	0.11	3.88	99.68
1-6	0.55	0.02	0.18	62.83	14.08	10.88	0.062	4.06	0.33	0.21	2.00	0.651	0.15	4.16	99.42
1-7	0.76		1.55	62.31	12.85	12.90	0.197	3.80	0.34	0.18	1.71	0.560	0.14	5.02	100.00
1-8	0.59	0.13	0.26	61.20	14.28	11.44	0.101	4.36	0.24	0.21	1.99	0.613	0.11	4.61	99.16
1-9	0.98		0.18	66.30	12.30	10.91	0.074	3.65	0.27	0.17	1.67	0.581	0.11	4.15	100.17
1-10	0.86	0.02	1.75	65.43	11.12	12.92	0.223	3.20	0.24	0.20	1.47	0.419	0.14	4.87	100.22
1-11	0.67		0.70	65.42	12.55	11.47	0.119	3.47	0.22	0.16	1.70	0.525	0.15	4.22	100.02
1-12	0.74	0.02	0.29	68.67	12.35	9.54	0.060	2.99	0.14	0.15	1.96	0.488	0.09	3.81	100.25
1-13	0.29		0.62	62.65	13.91	11.64	0.113	3.66	0.27	0.18	1.96	0.577	0.17	4.24	99.35
1-14	0.42	0.02	1.25	63.85	13.50	11.44	0.164	3.20	0.34	0.26	2.15	0.480	0.10	4.64	100.13
1-15	0.30		0.55	68.68	12.72	8.64	0.057	2.83	0.17	0.19	2.16	0.439	0.08	3.39	99.36
1-16	0.72	0.02	0.33	69.98	11.98	9.14	0.068	2.73	0.15	0.21	1.91	0.414	0.07	3.63	100.29
1-17	0.89		0.15	67.72	12.55	9.84	0.055	3.20	0.16	0.17	1.90	0.472	0.10	3.85	100.02
1-18	0.50	0.04	0.15	67.07	13.08	9.40	0.054	3.20	0.16	0.20	2.08	0.486	0.09	3.61	99.43
1-19	0.40		-0.05	71.00	12.06	7.79	0.038	2.76	0.08	0.22	1.99	0.394	0.06	3.16	99.56
1-20	0.71	0.03	0.29	68.39	12.76	9.64	0.065	2.67	0.18	0.22	2.12	0.396	0.07	3.68	100.18
1-21	0.75		0.18	67.95	12.88	9.46	0.054	2.70	0.15	0.21	2.21	0.436	0.09	3.66	99.80
1-22	0.60	0.02	0.22	66.62	12.78	10.28	0.062	3.46	0.14	0.21	1.81	0.496	0.08	3.87	99.81
1-23	0.50		0.73	68.77	11.19	9.90	0.108	3.18	0.19	0.17	1.51	0.464	0.12	3.81	99.40
1-24	0.63	0.05	0.29	66.22	13.03	10.58	0.075	3.51	0.31	0.22	1.86	0.581	0.12	3.96	100.46
1-25	0.68		0.37	63.79	13.47	10.55	0.076	3.59	0.24	0.20	2.04	0.554	0.09	4.19	98.79

Table A1.7. Trace metal chemistry for the 0.02% S sample of greenstone (values in ppm unless otherwise noted). Analysis by ACTLABS.

Sample ID	Au ppb	As	Br	Co	Cr	Cs	Hf	Ir ppb	Mo	Rb	Sb	Sc	Se	Ta	Th	U	W	La	Ce
2-1	<5	2	<1	38	275	4.9	2.7	<5	<5	74	<0.2	28.5	<3	<1	1.4	0.7	<3	15.3	34
2-3	<5	<2	<1	37	304	5.6	2.7	<5	<5	89	0.3	29.6	<3	<1	1.3	0.8	<3	13.8	32
2-5	<5	<2	<1	38	292	5.3	2.4	<5	<5	68	<0.2	28.4	<3	<1	1.2	<0.5	<3	13.6	32
2-7	<5	<2	1	38	293	4.8	2.6	<5	<5	84	<0.2	30.6	<3	<1	1.1	<0.5	<3	15.1	35
2-9	<5	<2	<1	38	290	4.9	2.5	<5	<5	78	<0.2	29.2	<3	<1	1	<0.5	<3	13.8	31
2-11	<5	<2	<1	34	282	5.5	2.3	<5	<5	77	<0.2	28.3	<3	<1	1.1	0.6	<3	16.9	37
2-13	<5	<2	<1	41	304	4.7	2.5	<5	<5	61	0.2	30.8	<3	<1	1.4	<0.5	<3	13.8	32
2-15	<5	<2	<1	39	310	4.6	2.5	<5	<5	61	0.3	29.8	<3	<1	1	0.6	<3	12.5	30
2-17	<5	<2	<1	41	294	5.3	2.3	<5	<5	81	0.2	32.1	<3	<1	1.3	<0.5	<3	13.4	32
2-19	<5	<2	<1	40	290	5.1	2.7	<5	<5	97	<0.2	30.7	<3	<1	1.2	<0.5	<3	13	28
2-21	<5	<2	<1	37	283	5.1	2.3	<5	<5	89	<0.2	29	<3	<1	1.1	<0.5	<3	9.6	22
2-23	<5	<2	<1	46	303	3.8	2.1	<5	<5	55	<0.2	30.9	<3	<1	1	<0.5	<3	11.4	27
2-25	<5	<2	<1	41	292	4.2	2.5	<5	<5	75	0.3	30.5	<3	<1	1.1	<0.5	<3	12	29

Sample ID	Nd	Sm	Eu	Tb	Yb	Lu	Ag	Cd	Cu	Ni	Pb	Zn	Bi
2-1	15	3.7	0.9	<0.5	1.9	0.28	<0.3	<0.3	23	140	<3	92	<2
2-3	18	3.5	1	<0.5	1.9	0.28	<0.3	<0.3	16	137	<3	89	<2
2-5	16	3.5	1.1	0.5	2	0.32	<0.3	<0.3	32	136	<3	88	<2
2-7	17	3.7	1.1	<0.5	2.1	0.34	<0.3	<0.3	20	142	<3	88	<2
2-9	15	3.7	1.2	0.5	2.2	0.34	<0.3	<0.3	17	141	<3	91	<2
2-11	18	4.3	1.5	0.7	3.1	0.46	<0.3	<0.3	54	137	<3	91	<2
2-13	15	3.7	1.1	<0.5	1.8	0.28	<0.3	<0.3	13	166	<3	98	<2
2-15	13	3.3	1.1	<0.5	2.1	0.33	<0.3	<0.3	23	143	<3	85	<2
2-17	14	3.5	1	<0.5	1.9	0.29	<0.3	<0.3	17	153	<3	99	<2
2-19	12	3.4	0.9	0.6	1.8	0.28	<0.3	<0.3	12	145	<3	95	<2
2-21	12	2.7	0.8	<0.5	1.4	0.25	<0.3	<0.3	19	138	<3	99	<2
2-23	15	3.1	0.9	<0.5	1.6	0.25	<0.3	0.6	12	172	<3	103	<2
2-25	15	3.7	1.4	<0.5	4	0.6	<0.3	<0.3	11	156	<3	97	<2

Table A1.8. Trace metal chemistry for the 0.20% S sample of greenstone (values in ppm unless otherwise noted).

Analysis by ACTLABS.

Sample ID	Au ppb	As	Br	Co	Cr	Cs	Hf	Ir ppb	Mo	Rb	Sb	Sc	Se	Ta	Th	U	W	La	Ce
3-2	<5	8	<1	47	470	2.5	2.5	<5	<5	43	0.2	20.6	<3	<1	2.7	0.8	<3	23.1	49
3-4	<5	5	<1	29	334	2.4	4.2	<5	<5	37	<0.2	16.7	<3	1	3.5	0.7	<3	21.8	51
3-6	<5	7	<1	38	413	2.9	2.5	<5	<5	48	<0.2	20.7	<3	<1	2.6	1	<3	16.5	37
3-8	<5	7	<1	38	470	2.7	3.1	<5	<5	57	0.3	21.8	<3	<1	3.9	1	<3	23.8	54
3-10	<5	8	<1	38	394	2.9	3.2	<5	<5	58	<0.2	20.7	<3	<1	3.2	0.8	<3	20.9	46
3-12	<5	8	<1	38	462	2.1	2.7	<5	<5	35	0.2	19.4	<3	<1	3.2	0.8	<3	27.7	62
3-14	<5	8	<1	34	403	2.7	4.5	<5	<5	64	<0.2	19.5	<3	<1	3.8	1	<3	31.5	72
3-16	<5	9	<1	35	400	2.6	3.2	<5	<5	55	<0.2	19.6	<3	<1	3	0.9	<3	21.2	48
3-18	<5	4	<1	40	428	3.8	2.7	<5	<5	60	<0.2	25.9	<3	<1	2.2	<0.5	<3	14.8	33
3-20	6	7	<1	37	335	3.3	2.8	<5	<5	66	0.2	23.4	<3	<1	3.1	1.1	<3	18.7	41
3-22	<5	11	<1	36	350	3	3.7	<5	<5	79	<0.2	22.2	<3	<1	4.1	1	<3	22.7	50
3-24	<5	17	<1	44	362	2.6	2.5	<5	<5	68	0.2	19.9	<3	<1	2.6	0.6	<3	17.2	38

Sample ID	Nd	Sm	Eu	Tb	Yb	Lu	Ag	Cd	Cu	Ni	Pb	Zn	Bi
3-2	22	5.1	1.1	0.7	2.1	0.32	<0.3	<0.3	123	188	<3	114	<2
3-4	21	5.4	1.1	<0.5	4.3	0.64	<0.3	<0.3	70	146	6	106	<2
3-6	18	4.2	1.1	<0.5	2	0.3	<0.3	<0.3	175	201	<3	110	<2
3-8	25	5.5	1.3	<0.5	2	0.31	<0.3	<0.3	97	205	<3	131	<2
3-10	23	4.9	1.1	<0.5	2.2	0.34	<0.3	0.8	151	233	<3	121	<2
3-12	31	6.2	1.4	<0.5	1.7	0.26	<0.3	<0.3	122	179	<3	94	<2
3-14	35	7.3	1.7	0.7	2.1	0.31	<0.3	<0.3	83	221	7	108	<2
3-16	23	4.8	1.2	0.7	3.5	0.56	<0.3	<0.3	91	180	<3	106	<2
3-18	16	3.8	0.9	<0.5	1.6	0.23	<0.3	<0.3	65	216	<3	106	<2
3-20	21	4.4	1.1	<0.5	1.8	0.27	<0.3	<0.3	96	192	<3	104	<2
3-22	24	5	1.1	0.6	2.3	0.33	<0.3	<0.3	110	156	13	114	<2
3-24	19	4	1	<0.5	1.7	0.25	<0.3	<0.3	131	171	<3	97	<2

Table A1.9. Trace metal chemistry for the 0.39% S sample of greenstone (values in ppm unless otherwise noted). Analysis by ACTLABS.

Sample ID	Au ppb	As	Br	Co	Cr	Cs	Hf	Ir ppb	Mo	Rb	Sb	Sc	Se	Ta	Th	U	W	La	Ce ppm
4-1	<5	7	<1	28	311	2.4	3.5	<5	<5	36	0.2	15.4	<3	<1	3	0.7	<3	17.5	39
4-3	7	14	1	30	326	2.6	3.6	<5	<5	65	<0.2	19.3	<3	<1	3.5	1.1	<3	22.4	48
4-5	<5	15	<1	27	288	1.8	4.2	<5	<5	54	<0.2	13.8	<3	1	4.3	1.4	<3	26.6	58
4-7	7	20	<1	30	282	2.5	3.7	<5	<5	72	<0.2	14.5	<3	<1	3.6	0.9	<3	24.2	54
4-9	<5	20	<1	30	343	2.5	3.3	<5	<5	64	<0.2	14.9	<3	<1	2.7	<0.5	<3	19.6	45
4-11	5	18	<1	35	341	2.5	4.1	<5	<5	79	<0.2	18.8	<3	<1	3.8	<0.5	<3	25.2	54
4-13	<5	37	<1	37	306	2.4	4.1	<5	<5	61	<0.2	16.3	<3	<1	3.2	<0.5	<3	18.9	42
4-15	<5	11	<1	35	328	2.7	4.7	<5	<5	63	<0.2	19.3	<3	<1	3.9	1.1	<3	23.8	53
4-17	<5	15	<1	41	352	2.2	3.7	<5	<5	52	0.3	19.0	<3	<1	4.0	1.3	<3	24.4	54
4-19	<5	16	<1	33	256	2.3	5.4	<5	<5	59	0.3	13.6	<3	<1	4.8	1.5	<3	29.0	62
4-21	<5	9	<1	35	289	1.8	4.0	<5	<5	51	<0.2	16.6	<3	<1	3.5	0.8	<3	20.5	46
4-23	<5	12	<1	29	297	1.6	4.5	<5	<5	55	0.3	15.4	<3	<1	3.5	1.0	<3	22.4	52
4-25	<5	27	<1	28	305	1.6	4.2	<5	<5	58	0.2	15.3	<3	<1	4.1	1.1	<3	23.1	49

Sample ID	Nd	Sm	Eu	Tb	Yb	Lu	Ag	Cd	Cu	Ni	Pb	Zn	Bi
4-1	19	4.3	0.9	<0.5	2.7	0.41	<0.3	<0.3	51	127	<3	81	<2
4-3	24	5.0	1.1	<0.5	2.3	0.35	<0.3	<0.3	60	133	<3	82	<2
4-5	27	5.4	1.1	<0.5	2.4	0.39	<0.3	<0.3	72	118	<3	78	<2
4-7	24	5.3	1.0	<0.5	2.5	0.38	<0.3	<0.3	68	110	<3	74	<2
4-9	22	4.2	0.9	<0.5	2.4	0.37	<0.3	<0.3	84	115	<3	71	<2
4-11	26	5.6	1.1	0.7	2.4	0.37	<0.3	<0.3	65	155	<3	85	<2
4-13	21	4.3	0.9	0.5	2.8	0.45	<0.3	<0.3	72	153	<3	86	<2
4-15	25	5.6	1.1	0.7	3.3	0.49	<0.3	<0.3	53	163	<3	87	<2
4-17	26	5.7	1.1	0.7	2.8	0.41	<0.3	<0.3	124	187	<3	95	<2
4-19	31	6.3	1.2	0.6	3.3	0.50	<0.3	<0.3	57	121	<3	88	<2
4-21	21	5.0	1.0	0.7	3.2	0.48	<0.3	<0.3	66	160	<3	88	<2
4-23	23	5.4	1.1	<0.5	2.7	0.42	<0.3	<0.3	67	142	<3	84	<2
4-25	25	5.4	1.0	0.6	3.0	0.44	<0.3	<0.3	71	136	<3	83	<2

Table A1.10. Trace metal chemistry for the 0.67% S sample of greenstone (values in ppm unless otherwise noted). Analysis by ACTLABS.

Sample ID	Au ppb	As	Br	Co	Cr	Cs	Hf	Ir ppb	Mo	Rb	Sb	Sc	Se	Ta	Th	U	W	La	Ce
1-2	<5	6	<1	24	95	2.2	5.7	<5	<5	63	0.3	16.1	<3	1	5	1	<3	33.5	72
1-4	<5	6	<1	23	120	2.2	6	<5	<5	66	0.3	15.3	<3	<1	4.8	1.4	<3	30.3	67
1-6	<5	5	<1	31	123	1.9	5.3	<5	<5	72	0.3	20.7	<3	<1	3.8	1.1	<3	23.3	54
1-8	5	5	<1	27	179	2.8	4.5	<5	<5	60	0.3	18.8	<3	<1	3.3	0.8	<3	22.8	52
1-10	<5	4	<1	21	127	1.6	5.2	<5	<5	51	0.3	12.8	<3	<1	4.4	0.8	<3	27.4	62
1-12	<5	3	<1	21	97	2	6.6	<5	<5	69	0.3	13.7	<3	1	5.2	1.3	<3	36.3	80
1-14	<5	2	<1	20	129	3	7.5	<5	<5	75	0.2	13.6	<3	1	5.6	1.5	<3	35.5	79
1-16	<5	4	<1	18	123	2.2	6.6	<5	<5	66	0.3	11.4	<3	1	5.2	1.5	<3	35.1	78
1-18	<5	4	<1	19	97	2.6	7.6	<5	<5	69	0.2	14.6	<3	<1	5.7	1.2	<3	34.9	77
1-20	<5	3	<1	18	106	2.3	7.8	<5	<5	67	0.3	11.2	<3	1	6	1.4	<3	41.4	91
1-22	<5	4	<1	21	90	1.9	7.1	<5	<5	71	-0.2	15	<3	1	5.5	1.3	<3	34.3	76
1-24	<5	5	<1	25	131	2.3	5.8	<5	<5	60	0.3	17.1	<3	<1	4.3	0.8	<3	26	59

Sample ID	Nd	Sm	Eu	Tb	Yb	Lu	Ag	Cd	Cu	Ni	Pb	Zn	Bi
1-2	35	7.5	1.4	0.9	4.4	0.66	<0.3	0.4	100	67	<3	158	<2
1-4	32	7	1.2	1.1	5.3	0.81	<0.3	<0.3	77	68	3	158	<2
1-6	25	5.7	1	0.8	4	0.62	<0.3	<0.3	88	92	5	163	<2
1-8	23	5.4	0.9	0.6	3	0.47	<0.3	<0.3	52	119	<3	189	<2
1-10	30	6.6	1.1	0.8	4.2	0.63	<0.3	<0.3	57	67	<3	174	<2
1-12	39	8.7	1.6	1.2	6.1	0.95	<0.3	<0.3	69	60	4	145	<2
1-14	39	8.8	1.5	1.5	8.1	1.22	<0.3	<0.3	47	73	<3	143	<2
1-16	36	8.4	1.5	1.3	6.7	1.01	<0.3	<0.3	46	63	7	140	<2
1-18	37	8.6	1.5	1.3	6.4	0.97	<0.3	<0.3	55	59	<3	157	<2
1-20	44	10	1.7	1.2	6.7	1.01	<0.3	<0.3	46	62	<3	147	<2
1-22	37	8.2	1.4	1	5.3	0.79	<0.3	<0.3	58	61	<3	169	<2
1-24	28	6.3	1	0.8	4.4	0.65	<0.3	<0.3	62	78	<3	144	<2

Table A1.11. Particle size distribution of field bins (percent).

SIZE FRACTION	PILE 1 PERCENT	PILE 2 PERCENT	PILE 3 PERCENT	PILE 4 PERCENT
+ 12"	12.2	8.7	3.0	7.9
- 12" / +6"	10.6	8.9	6.2	6.1
- 6" / +21/2"	21.4	23.3	12.5	11.1
- 21/2" / +3/4"	33.0	32.7	30.9	32.7
-3/4" / +1/4"	11.4	10.5	22.1	19.3
-1/4" / +10	5.2	6.2	12.1	11.7
-10 / +35	3.8	6.2	8.4	6.9
-35 / +100	1.5	2.3	3.1	2.4
-100 / +200	0.3	0.4	0.7	0.7
-200	0.5	0.8	1.0	1.2

Table A1.12. Whole rock analyses as a function of particle size for the greenstone prediction piles. Analysis by ACTLABS.

Mesh Size	S %	S^{2-} [1] %	SO_4 %	CO_2 %	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	MnO %	MgO %	CaO %	Na_2O %	K_2O %	TiO_2 %	P_2O_5 %	LOI %	TOTAL[2] %
0.02% Sulfur Bin																
+2½	0.04	0.00	0.05	<0.05	50.43	21.22	11.59	0.114	6.61	0.42	0.67	1.85	0.942	0.29	6.45	100.59
+3/4	0.04	0.00	0.05	<0.05	52.18	19.34	11.66	0.129	6.74	0.43	0.34	2.42	0.783	0.28	5.96	100.27
+1/4	0.06	0.06	<0.05	<0.05	51.41	19.08	12.20	0.180	6.09	0.90	0.46	2.53	0.878	0.35	5.63	99.72
+10	0.04	0.04	<0.05	0.37	53.89	18.33	11.38	0.117	5.67	1.00	0.53	2.55	0.829	0.36	5.79	100.44
+35	0.05	0.05	<0.05	0.81	54.41	18.33	10.83	0.115	5.44	1.06	0.62	2.52	0.763	0.38	5.73	100.20
+100	0.05	0.05	<0.05	1.00	54.17	17.66	10.31	0.108	5.10	1.25	0.82	2.50	0.751	0.33	6.04	99.04
+200	0.07	0.07	<0.05	1.03	53.34	17.88	10.60	0.114	5.21	1.61	0.95	2.41	0.799	0.33	6.38	99.63
<200	0.08	0.00	0.05	1.50	50.68	18.64	11.15	0.122	5.49	1.83	0.84	2.56	0.679	0.33	7.33	99.65
0.20% Sulfur Bin																
+2½	0.80	0.80	<0.05	<0.05	75.83	8.18	9.48	0.081	2.60	0.10	0.09	0.76	0.133	0.06	2.89	100.20
+3/4	0.41	0.41	<0.05	0.07	66.39	14.99	8.31	0.075	2.38	0.33	0.20	2.89	0.594	0.21	3.51	99.88
+1/4	0.73	0.73	<0.05	<0.05	60.94	14.56	11.01	0.095	5.32	0.64	0.30	1.61	0.617	0.30	4.98	100.37
+10	0.27	0.27	<0.05	0.81	61.05	14.68	9.61	0.086	4.39	1.65	0.49	2.15	0.660	0.33	5.05	100.14
+35	0.25	0.25	<0.05	0.92	60.06	15.55	9.16	0.079	4.27	1.52	0.42	2.28	0.657	0.33	5.04	99.37
+100	0.26	0.26	<0.05	1.72	57.30	16.74	9.42	0.081	4.30	2.12	0.56	2.50	0.743	0.29	6.07	100.12
+200	0.28	0.28	<0.05	2.13	52.49	17.78	10.23	0.087	4.83	3.16	0.60	2.61	0.836	0.33	7.15	100.09
<200	0.25	0.23	0.05	2.90	50.39	18.29	10.15	0.088	4.67	3.85	0.57	2.78	0.776	0.33	8.25	100.14
0.39% Sulfur Bin																
+2½	0.25	0.25	<0.05	<0.05	47.89	14.90	17.03	0.097	9.74	1.31	0.03	0.18	0.857	0.95	6.41	99.39
+3/4	0.50	0.50	<0.05	<0.05	78.51	8.12	6.51	0.047	2.61	0.14	0.13	1.04	0.267	0.10	2.56	100.05
+1/4	0.28	0.28	<0.05	<0.05	68.71	12.55	8.42	0.062	3.29	0.52	0.34	1.64	0.555	0.32	3.50	99.92
+10	0.29	0.29	<0.05	0.37	67.56	13.47	8.12	0.063	3.24	0.77	0.44	1.85	0.529	0.26	3.94	100.24
+35	0.33	0.33	<0.05	0.33	64.82	15.22	8.41	0.064	3.20	0.61	0.59	2.26	0.593	0.26	4.11	100.13
+100	0.31	0.26	0.15	0.40	60.77	17.37	8.74	0.070	3.36	0.79	0.80	2.59	0.699	0.25	5.00	100.45
+200	0.60	0.60	<0.05	1.10	56.80	17.52	11.49	0.096	3.45	0.93	0.55	3.12	0.769	0.12	5.66	100.49
<200	0.55	0.52	0.10	1.10	54.76	18.48	11.46	0.100	3.48	1.05	0.49	3.37	0.718	0.12	6.14	100.17
0.67% Sulfur Bin																
+2½	0.90	0.90	<0.05	<0.05	75.36	11.59	5.99	0.018	1.65	0.06	0.20	2.54	0.222	0.04	2.78	100.45
+3/4	0.25	0.25	<0.05	<0.05	73.04	10.62	7.57	0.045	3.27	0.18	0.18	1.25	0.480	0.13	3.04	99.81
+1/4	0.75	0.70	0.15	<0.05	68.43	12.28	9.38	0.043	3.33	0.16	0.22	1.82	0.460	0.08	3.64	99.85
+10	0.63	0.58	0.15	0.29	66.98	12.90	10.14	0.065	3.18	0.32	0.25	2.01	0.489	0.09	4.00	100.41
+35	0.46	0.46	<0.05	0.22	64.55	14.28	9.97	0.066	3.28	0.29	0.29	2.33	0.524	0.09	4.05	99.71
+100	0.43	0.43	<0.05	0.48	60.06	16.54	10.51	0.073	3.43	0.54	0.46	2.90	0.650	0.10	4.82	100.08
+200	0.34	0.29	0.15	0.66	57.49	18.31	9.26	0.075	3.64	1.14	0.89	2.80	0.783	0.27	5.78	100.43
<200	0.32	0.32	<0.05	0.66	57.32	18.26	9.17	0.076	3.47	1.23	0.84	2.94	0.746	0.26	5.93	100.24

[1] Determined by difference. Less than values are assumed to be 0.

[2] Total for parameters SiO_2 through LOI.

Table A1.13. Page 1 of 2. Trace metal analyses as a function of particle size for the greenstone prediction piles. Analysis by ACTLAB Concentrations in mg/L unless indicated otherwise.

Mesh Size	Ag	Au ppb	As	Bi	Br	Co	Cr	Cd	Cu	Cs	Hf	Ir ppb	Mo	Ni	Pb	Rb
0.02% Sulfur pile																
+2½	<0.3	<5	<2	<2	<1	44	338	<0.3	19	3.8	3.4	<5	<5	191	<3	68
+3/4	<0.3	<5	<2	<2	<1	44	256	0.4	34	5.3	2.9	<5	<5	144	<3	86
+1/4	<0.3	5	<2	<2	<1	50	309	<0.3	291	5.3	2.9	<5	<5	199	<3	90
+10	0.3	5	<2	<2	<1	40	326	<0.3	24	3.8	4.0	<5	<5	140	<3	90
+35	0.4	<5	<2	<2	<1	38	272	<0.3	30	5.1	2.9	<5	<5	138	<3	76
+100	0.5	<5	4	<2	2	39	313	<0.3	51	4.9	4.0	<5	<5	136	19	108
+200	0.4	<5	5	<2	3	39	234	<0.3	43	5.6	3.7	<5	<5	133	17	97
<200	0.4	13	5	<2	4	41	254	<0.3	55	8.0	4.1	<5	<5	143	11	84
0.20% Sulfur pile																
+2½	0.9	<5	4	<2	<1	6	120	<0.3	59	1.7	7.4	<5	<5	30	<3	30
+3/4	0.6	6	8	<2	<1	31	289	<0.3	112	4.2	5.0	<5	<5	95	<3	107
+1/4	0.6	<5	11	<2	<1	47	354	<0.3	177	3.3	4.1	<5	<5	210	<3	79
+10	0.5	<5	12	<2	<1	38	408	<0.3	77	3.0	4.9	<5	<5	156	<3	94
+35	0.6	<5	12	<2	<1	38	362	<0.3	110	3.3	4.6	<5	<5	161	<3	79
+100	0.8	<5	14	<2	2	40	412	<0.3	142	3.9	5.2	<5	<5	166	15	93
+200	0.9	6	18	<2	3	41	360	<0.3	187	4.8	6.0	<5	<5	178	12	86
<200	1.0	11	21	<2	2	39	335	<0.3	170	4.9	6.2	<5	<5	181	17	94
0.39% sulfur pile																
+2½	0.3	5	26	<2	<1	62	699	<0.3	27	2.0	3.9	<5	<5	251	<3	<20
+3/4	0.6	6	12	<2	<1	25	155	<0.3	121	<0.5	3.3	<5	<5	79	4	<20
+1/4	0.5	5	12	<2	<1	32	314	<0.3	156	2.0	3.9	<5	<5	189	<3	61
+10	0.6	<5	13	<2	<1	33	325	<0.3	82	2.9	4.9	<5	<5	150	<3	77
+35	0.7	<5	15	<2	<1	35	302	<0.3	107	3.8	5.8	<5	<5	157	14	93
+100	0.9	6	17	<2	<1	37	368	<0.3	165	3.7	7.2	<5	<5	161	10	76
+200	1.6	<5	8	<2	4	30	93	<0.3	85	4.3	12.5	<5	<5	91	27	121
<200	1.8	<5	10	<2	5	29	104	0.3	98	4.7	13.4	<5	<5	90	28	124
0.67% Sulfur pile																
+2½	1.2	<5	<2	<2	<1	9	93	<0.3	63	2.8	11.1	<5	<5	33	<3	113
+3/4	0.5	5	<2	<2	<1	19	154	<0.3	54	1.9	4.9	<5	<5	60	7	43
+1/4	0.9	10	4	<2	<1	26	116	<0.3	105	2.0	7.2	<5	<5	67	<3	76
+10	0.8	<5	4	<2	<1	25	159	<0.3	75	2.1	7.5	<5	<5	63	<3	85
+35	1.1	<5	3	<2	<1	23	110	<0.3	67	2.7	8.8	<5	<5	64	4	103
+100	1.2	<5	5	<2	4	25	153	<0.3	75	3.6	10.5	<5	<5	71	<3	108
+200	0.8	52	27	<2	1	41	305	<0.3	149	4.2	8.5	<5	<5	149	25	100
<200	0.8	72	29	<2	<1	40	291	<0.3	171	4.5	8.1	<5	<5	142	20	108

Table A1.13. Page 2 of 2. Trace metal analyses as a function of particle size for the greenstone prediction piles. Analysis by ACTLAB Concentrations in mg/L unless indicated otherwise.

Mesh Size	Sb	Sc	Se	Ta	Th	U	W	Zn	La	Ce	Nd	Sm	Eu	Tb	Yb	Lu
0.02% Sulfur pile																
+2½	0.2	29.2	<3	<1	1.0	<0.5	<3	87	17.4	42	22	4.7	1.8	0.7	3.2	0.47
+3/4	0.2	30.4	<3	<1	1.3	1.0	<3	96	11.5	29	16	3.4	1	<0.5	1.8	0.26
+1/4	0.3	30.6	<3	<1	1.5	<0.5	<3	97	12.7	29	13	3.3	1.1	0.6	1.8	0.27
+10	0.3	28.6	<3	<1	2.0	<0.5	<3	84	14.6	33	18	3.7	1.2	<0.5	2.2	0.35
+35	0.5	27.4	<3	<1	1.8	0.9	<3	91	15.0	36	16	3.7	1.2	<0.5	2.2	0.34
+100	0.9	26.1	<3	<1	2.2	0.9	3	101	16.2	36	21	3.8	1.2	0.6	2.3	0.35
+200	1.3	25.7	<3	<1	3.2	<0.5	<3	92	17.6	39	20	3.9	1.1	0.6	2.3	0.35
<200	1.9	26.0	<3	1	2.9	0.7	<3	104	17.5	40	21	3.8	1.2	0.6	2.5	0.37
0.20% Sulfur pile																
+2½	<0.2	3.6	<3	<1	6.9	1.8	<3	97	46.6	101	45	9.3	1.7	1.5	8	1.31
+3/4	<0.2	20.7	<3	<1	2.9	0.9	<3	86	14.3	33	13	3.5	1.1	<0.5	2.8	0.41
+1/4	0.4	21.0	<3	<1	4.0	1.8	<3	104	18.3	43	16	4.3	1.3	0.7	2.9	0.44
+10	0.4	21.2	<3	<1	4.0	1.8	<3	98	20.4	46	21	4.6	1.4	<0.5	2.5	0.37
+35	<0.2	22.1	<3	<1	3.8	1.4	10	95	23.2	54	24	5.1	1.3	0.6	2.6	0.41
+100	0.8	22.9	<3	1	4.4	2.0	4	116	28.5	60	29	5.6	1.5	0.8	2.9	0.45
+200	1	25.1	<3	<1	5.6	1.1	6	115	34.9	79	33	6.8	1.7	0.9	3.3	0.49
<200	1.4	25.1	<3	<1	5.0	1.4	<3	131	32.4	68	27	6.3	1.6	<0.5	3.2	0.50
0.39% sulfur pile																
+2½	0.3	25.9	<3	<1	6.3	2.0	<3	136	19.9	48	27	6.5	1.9	<0.5	1.9	0.28
+3/4	0.3	10.0	<3	<1	2.8	0.8	<3	78	18.9	43	20	3.7	1.3	<0.5	2.1	0.33
+1/4	<0.2	16.8	<3	<1	4.9	1.6	4	93	28.7	63	24	5.5	1.5	<0.5	2.8	0.43
+10	0.3	17.3	<3	<1	4.7	1.9	<3	93	23.5	50	26	4.9	1.3	0.7	2.9	0.43
+35	0.2	18.9	<3	<1	5.6	1.5	<3	93	30.4	68	29	6.2	1.5	0.8	3.3	0.50
+100	0.9	21.5	<3	1	7.1	1.7	<3	124	38.7	86	44	7.5	1.8	0.9	3.8	0.57
+200	1.5	22.0	<3	2	8.7	2.2	<3	194	49.2	111	39	10.5	2.2	1.7	8.2	1.25
<200	1.8	23.6	<3	2	10.1	2.4	<3	190	45.8	98	45	10.0	2.1	1.6	8.8	1.37
0.67% Sulfur pile																
+2½	<0.2	4.2	<3	3	8.1	1.6	<3	88	62.8	126	66	13.0	2.5	2.1	8.3	1.35
+3/4	<0.2	15.7	<3	<1	3.3	<0.5	<3	121	26.2	58	30	5.4	1.1	0.8	4	0.60
+1/4	0.4	14.7	<3	1	5.2	1.8	<3	132	31.4	70	33	6.9	1.4	0.9	5	0.78
+10	0.3	16.0	<3	1	5.6	1.4	<3	132	30.8	71	34	6.8	1.5	1.0	4.9	0.75
+35	0.2	17.1	<3	2	6.7	1.4	<3	139	37.7	85	39	8.3	1.8	1.5	6.9	1.09
+100	0.6	20.7	<3	2	8.2	1.9	<3	157	44.5	100	40	9.6	2	1.6	7.6	1.16
+200	1.5	23.2	<3	<1	7.5	2.4	<3	107	46.3	99	42	8.8	2	1.0	4.3	0.66
<200	1.5	22.3	<3	<1	7.3	1.4	4	114	45.9	98	50	8.7	2.1	1.1	4.2	0.65

Table A1.14. Sobek method for determination of neutralization potentials from the 0.67% total sulfur (pile 4) muck box samples.

Sample	pH after HCL	mLs to pH 7.0	pH 7.0 NP	mLs to pH 8.3	pH 8.3 NP	mLs back to pH 8.3	pH 8.3 Final NP[1]
1-1	2.09	16.4	9.0	ns	ns	ns	7.2 est.
1-2	2.04	16.9	7.75	ns	ns	ns	5.95 est.
1-3	2.10	16.9	7.75	ns	ns	ns	5.95 est.
1-4	2.19	16.7	8.25	ns	ns	ns	6.45 est.
1-5	2.06	16.5	8.75	ns	ns	ns	6.95 est.
1-6	ns	16.35	9.125	ns	ns	ns	7.3 est.
1-7	2.02	15.9	10.25	ns	ns	ns	8.45 est.
1-8	2.02	16.8	8.0	ns	ns	ns	6.2 est.
1-9	2.01	16.0	10.0	ns	ns	ns	8.2 est.
1-10	1.93	16.5	8.75	17.3	6.75	17.4	6.5
1-11	1.93	16.6	8.5	17.3	6.75	17.4	6.5
1-12	2.11	17.3	6.75	18.0	5.0	18.1	4.75
1-13	2.01	17.0	7.5	17.5	6.25	17.6	6.0
1-14	1.96	15.7	10.75	16.3	9.25	16.4	9.0
1-15	1.96	17.3	6.75	17.7	5.75	17.8	5.5
1-16	1.98	16.7	8.25	17.2	7.0	17.3	6.75
1-17	1.96	16.9	7.75	17.3	6.75	17.4	6.5
1-18	1.99	18.1	4.75	18.6	3.5	18.7	3.25
1-19	2.00	16.6	8.5	18.2	4.5	18.3	4.25
1-20	1.96	16.8	8.0	17.4	6.5	17.5	6.25
1-21	1.96	17.2	7.0	17.8	5.5	17.9	5.25
1-22	1.97	16.6	8.5	17.2	7.0	17.3	6.75
1-23	1.92	17.2	7.0	17.8	5.5	17.9	5.25
1-24	1.96	16.9	7.75	17.4	6.5	17.5	6.25
1-25	1.95	16.2	9.5	17.3	6.75	17.4	6.5
Blank	1.64	20.1	0	ns	ns	ns	ns

Mean NP at pH 7.0 = 8.2 kg $CaCO_3$/t
Mean NP at pH 8.3 = 5.95 kg $CaCO_3$/t
Mean NP at pH 8.3 using est. values = 6.32 kg $CaCO_3$/t
Calculated NP = 10.5 kg $CaCO_3$/t
1-NP after 24 hours

Table A1.15. Particle size distribution for rock used in field limestone addition tanks and limestone (percent passing). Analysis by Lerch Brothers, Inc.

FRACTION	Tank 1	Tank 2	Tank 3	Tank 4	Tank 5	Tank 6	Limestone
2"	100.0	100.0	100.0	100.0	100.0	100.0	100.0
1 ½"	97.1	97.3	91.3	90.1	88.9	95.3	100.0
1"	68.2	77.4	80.2	72.6	77.5	82.4	100.0
1/2"	43.3	40.5	53.2	52.4	61.7	54.8	100.0
1/4"	31.5	27.2	37.5	36.2	43.8	42.0	100.0
4M	27.1	22.9	32.4	31.1	37.8	37.8	100.0
10M	16.7	14.2	19.6	18.7	23.1	25.3	87.9
20M	12.6	10.8	14.4	13.6	16.9	19.5	57.1
28M	10.8	9.3	12.0	11.4	14.1	17.0	41.8
35M	9.5	8.2	10.2	9.9	12.1	15.3	30.2
48M	7.8	6.7	6.9	7.9	9.6	13.3	18.5
65M	6.2	5.5	5.1	6.3	7.6	11.6	9.0
100M	5.4	4.6	4.7	5.2	6.3	10.4	4.5
200M	3.4	2.8	2.5	3.1	3.7	8.2	1.4

NOTE: All samples were dry screened with the exception of Tank 6 which was wet screened for comparison.

Table A1.16. Whole rock chemistry (percent) of field limestone addition tanks. Analyses by ACTLABS, Inc.

Parameter	Tank 1	Tank 2	Tank 3	Tank 4	Tank 5	Tank 6	Limestone
S	0.56	0.40	0.50	0.46	0.55	0.48	0.01
S^{2-} [2]	0.54	0.34	0.48	0.44	0.53	0.46	0
SO_4^{2-} as S	0.016	0.06	0.02	0.016	0.02	0.02	0.016
CO_2	0.40	0.37	0.22	0.44	0.44	0.48	41.56[3]
Al_2O_3	13.86	13.09	12.53	13.12	13.28	13.00	0.47
CaO	0.41	0.37	0.26	0.46	0.32	0.43	27.63
Fe_2O_3	10.65	8.83	8.61	9.38	10.30	9.31	0.87
K_2O	1.86	2.03	1.95	1.91	1.85	2.09	0.29
MgO	3.59	2.96	2.91	3.26	3.41	2.89	18.82
MnO	0.068	0.065	0.053	0.089	0.065	0.059	0.081
Na_2O	0.36	0.38	0.30	0.39	0.34	0.38	<0.01
P_2O_5	0.15	0.12	0.10	0.12	0.13	0.21	0.03
SiO_2	64.70	68.40	69.30	65.96	65.38	67.32	9.68
TiO_2	0.587	0.458	0.452	0.522	0.519	0.514	0.026
LOI	4.19	3.73	3.62	3.89	4.02	3.72	41.95
TOTAL[2]	100.42	100.44	100.08	99.11	99.60	99.92	99.78

1 - Determined by difference. Less than values are assumed to be 0.

2 - For parameters from Al_2O_3 through LOI2.

3 - Analysis by Lerch Brothers Inc. and determined by LOI.

Table A1.17. Page 1 of 2. Trace metal analysis of field limestone addition tanks. Analysis by ACTLABS, Inc.; concentrations in ppm.

Parameter	Tank 1	Tank 2	Tank 3	Tank 4	Tank 5	Tank 6	Limestone
Ag	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3
As	5	3	6	3	5	4	2
Au	<5	<5	<5	<5	6	5	<5
Bi	<2	<2	<2	<2	<2	<2	<2
Br	<1	<1	<1	<1	<1	<1	7
Cd	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3
Co	26	20	21	22	24	22	2
Cr	105	75	65	75	71	76	5
Cs	2.2	2.7	2.0	2.4	2.1	2.5	<0.5
Cu	67	36	44	45	44	45	7
Hf	5.5	6.6	6.3	5.9	6.6	6.5	<0.5
Mo	<5	<5	<5	<5	<5	<5	<5
Ni	97	69	69	82	76	75	3
Pb	<3	<3	6	<3	5	<3	<3
Rb	68	67	63	57	54	71	<20
Sb	0.3	0.3	0.3	0.3	0.3	0.3	<0.2
Se	<3	<3	<3	<3	<3	<3	<3
Ir	<5	<5	<5	<5	<5	<5	<5
Ta	<1	<1	<1	1	1	<1	<1
W	<3	<3	<3	<3	<3	<3	<3
Zn	158	132	132	147	149	135	17

Table A1.17. Page 2 of 2. Trace metal analysis of field limestone addition tanks. Analysis by ACTLABS, Inc.; concentrations in ppm.

Parameter	Tank 1	Tank 2	Tank 3	Tank 4	Tank 5	Tank 6	Limestone
Ce	57	76	74	63	72	73	6
La	26.9	34.8	33.2	28.1	32.7	33.3	2.4
Lu	0.66	0.85	0.76	0.84	0.82	0.86	<0.05
Nd	27	33	35	31	34	36	<5
Sc	18.1	13.6	13.6	16.2	16.3	15.4	0.5
Tb	0.8	1.1	0.9	0.9	1.0	1.2	<0.5
Th	4.0	5.6	4.9	4.7	4.7	4.9	<0.5
U	1.1	1.3	1.7	0.9	1.1	1.2	0.9
Yb	4.3	5.5	4.9	5.4	5.2	5.5	0.2
Sm	6.0	7.5	7.3	6.2	7.2	7.4	0.4
Eu	1.2	1.5	1.4	1.2	1.3	1.5	0.1

Attachment A.1.1. Mineralogy of original greenstone sample used in the limestone addition experiment. Analyses by Mineralogical Consulting Service, Pengilly, MN.

MINERALOGICAL CONSULTING SERVICE

29022 West Shore Lane ~ Pengilly, MN 55775-2222
Phone 218/885-2358 ~ Email cygnus@cpinternet.com

February 7, 2003

Mr. Kim Lapakko
Minnesota Department of Natural Resources
Division of Lands and Minerals
500 Lafayette Road
St. Paul, MN 55155-4045

Re: Greenstone "Tank" Samples

Ref: Greenstone Sample Mineralogy by L.A. Mattson to Kim Lapakko, January 31, 2003

Dear Mr. Lapakko:

The mineralogical composition and pyrite liberation were determined for the two (2) greenstone "tank" samples submitted. These samples are labeled #11920 (Limestone Addition Experiment Control 1, Tank 1) and #11925 (Limestone Addition Experiment Control 2, Tank 6). The sample material received had been crushed and screened/sieved into fourteen (14) size fractions from -2+1 1/2 inch to -200 mesh.

Macroscopic and low power microscopic examination of the crushed fragments indicates that both samples are very fine grained quartz-chlorite-sericite schist. No coarser grained quartz or carbonate mineral segregations were observed in either sample. However, both samples are contaminated with what appears to be typical glacial drift sand and gravel. The contamination in the size fractions received may constitute up to 5 to 8 percent (very rough estimate) of the overall combined samples. It occurs as scattered rock fragment in the coarser size fractions and as abundant rounded sand sized grains in the finer size fractions.

Minerals and Mineral Chemistry

Minerals present in the greenstone are listed in Table 1. No attempt was made to account for the minerals in the sample contamination. Table 2 is a summary of Scanning Electron Microscope/Energy Dispersive Spectroscopy (SEM/EDS) mineral chemistry determined for sericite, chlorite, and siderite at Macalester College. The average chemistry for the minerals listed in Table 2 was used in this study. See reference report for a discussion of the greenstone minerals and mineral chemistry.

Mineralogical Composition

Table 3 lists the mineral compositions for these two (2) samples. Two (2) compositions are listed for each sample; one based on the Macalester College average SEM/EDS chemistry for sericite, chlorite, and siderite, and the other using the average values for sericite and siderite along with a calculated chemistry for chlorite. See reference report for discussion of methods. As in the reference report, the mineralogical compositions using the calculated chlorite chemistry yield better mineral summations.

The contamination noted previously is SiO_2 rich and if present in the assay pulp used for the whole rock chemical assays would yield mineralogical compositions with relatively high quartz contents compared to the greenstone alone. Other whole rock chemical assays would probably not be greatly increased by the contamination, but would all be relatively lower due to dilution of the greenstone material. Minerals other than quartz in the mineralogical compositions would also be relatively lower compared to the composition of the greenstone alone.

Pyrite Liberation

Pyrite liberation was determined by microscopic examination of the fragments and particles in each of the fourteen (14) screen/sieve fractions (-2+1 1/2 inch to -200 mesh) received. A general examination was conducted of the coarser fractions where no pyrite is liberated and loose grain counts were completed for the finer particle size fractions. The overall pyrite liberation is the weighted (by sieve fraction weight percent) average of pyrite liberation in each size fraction and assumes that each size fraction has the same pyrite content as the total sample.

The pyrite is all fine grained and liberated pyrite was observed only in the size fractions finer than 28 mesh/600 microns. Most of the liberated pyrite is minus 48 mesh/300 microns. Observations indicate that seven (7) percent of the pyrite is liberated in sample #11920 and twelve (12) percent is liberated in sample #11925. However, sample #11920 was received as "dust covered" dry screened fractions and sample #11925 was received as "clean" wet screened fractions. Each size fraction of sample #11920 required washing with water to remove the "dust" and allow observation of any pyrite present. The washed-off "dust" is mostly minus 200 mesh and normally contains a high percent of liberated pyrite. Particle size distribution data provided indicates that dry screened sample #11920 has 3.4 percent minus 200 mesh compared to 8.2 percent in wet screened sample #11925. Considering the difference in sample preparation, both samples probably have very similar pyrite liberation of about twelve (12) percent.

Sincerely,

L.A. Mattson, Geologist/Process Mineralogist

Table I: Minerals -- Greenstone Samples

Mineral	Formula	Other Metals (see SEM assays)
Pyrite**	FeS_2	
Melanterite***	$FeSO_4.7H_2O$	
Siderite*	$FeCO_3$	Mg, Mn, Ca, Na?
Quartz*	SiO_2	REE****
Chlorite*	Complex Mg,Al,Fe Silicate	Cr, Ni, V, Ti
Sericite*	$KAl_3Si_3O_{10}(OH)_2$	Na, Ba, Cs, Rb, Ti
Na-Feldspar**	$NaAlSi_3O_8$	Sr
Ilmenite***	$FeTiO_3$	V
Hematite**	Fe_2O_3	
Magnetite**	Fe_3O_4	
Apatite**	$Ca_5(PO_4)_3$	As, REE, Th, U
Zircon**	$ZrSiO_4$	Hf
Tourmaline**	Na,Mg,Fe,Mn,Al,B Silicate	F, Li

*Minerals identified in X-ray Diffraction studies at Macalester College.

**Minerals identified visually in this and/or previous studies.

***Minerals not positively identified but assumed present based on overall mineralogy and chemistry

****REE group elements are not known to occur as replacements in quartz. In these samples higher REE values are generally associated with high silica/high quartz contents. Exact nature of REE occurance is unknown.

ORIGINAL #

Table 2

Summary of SEM Analysis of Greenstone Samples

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	
	102	106	103	058	105	100	057	066	068	063	060	067	064	062	Overall
Sericite	GR004R1	GR005R2	GR005R3	GR010R45	GR012R6	GR016R7	GR016R89	GR020R10	GR026R11	GR039R12	GR050R13	GR059R1415	GR072R16	GR122R1718	Average
Na_2O	1.26	0.67	0.70	1.02	0.62	2.19	1.34	0.96	1.81	1.15	0.93	1.95	0.75	0.74	1.15
MgO	0.58	1.02	1.23	1.11	0.97	0.23	0.71	0.71	0.84	0.67	1.00	0.76	0.80	0.88	0.82
Al_2O_3	33.76	34.02	34.04	35.19	31.36	35.86	34.80	33.02	32.39	35.12	32.66	34.19	32.91	35.18	33.89
SiO_2	45.99	48.93	47.92	49.38	47.75	46.94	46.95	46.77	44.05	49.04	43.48	46.68	47.67	45.32	46.92
P_2O_5	0.02	na[1]	na	na	0.01	0.28	0.05	0.04	0.04	0.06	0.03	0.04	0.00	0.21	0.07
K_2O	9.16	10.73	10.37	9.88	9.90	6.92	8.87	9.29	7.62	9.78	8.70	7.73	10.14	9.02	9.15
CaO	0.00	0.01	0.00	0.01	0.00	0.04	0.10	0.01	0.00	0.15	0.02	0.00	0.13	0.11	0.04
TiO_2	0.21	0.39	0.31	0.21	0.32	0.35	0.15	0.11	0.12	0.16	0.09	0.10	0.26	0.21	0.21
Cr_2O_3	0.03	0.01	0.05	0.06	0.04	0.13	0.01	0.01	0.01	0.02	0.02	0.00	0.03	0.02	0.03
MnO	0.00	0.00	0.02	0.01	0.00	0.11	na	0.01	0.01	na	0.01	0.01	na	0.09	0.02
FeO	1.87	2.75	3.46	1.81	2.96	3.32	2.25	1.54	1.23	1.79	2.46	0.90	2.70	3.72	2.34
NiO	0.01	0.02	0.04	0.03	0.04	0.06	0.04	0.01	0.00	0.03	0.01	0.02	0.04	0.03	0.03
H_2O	7.11	1.47	2.22	1.30	6.01	3.58	4.73	7.52	11.88	2.50	10.60	7.62	5.17	4.46	5.44
CO_2	na	na	na	na	na	na	na	na	na	na	na	na	na	na	na
F	na	na	na	na	na	na	na	na	na	na	na	na	na	na	na
Total	100.00	100.02	100.35	100.00	100.00	100.00	100.00	100.00	100.00	100.48	100.00	100.00	100.60	100.00	100.12
Mg0/FeO	0,31	0,37	0,36	0,61	0,33	0,07	0,32	0,46	0,68	0,37	0,41	0,84	0,30	0,24	0,35

Chlorite	GR004R1	GR005R2	GR005R3	GR010R45	GR012R6	GR016R7	GR016R89	GR020R10	GR026R11	GR039R12	GR050R13	GR059R1415	GR072R16	GR122R1718	Overall Average
Na_2O	0.36	0.36	0.46	0.54	na	0.00	na	0.32	0.42	na	0.42	0.36	na	0.00	0.32
MgO	14.81	5.17	14.30	12.78	7.08	7.13	7.21	15.53	13.14	10.78	8.37	11.43	5.83	11.83	10.39
Al_2O_3	21.29	21.60	21.87	24.50	21.14	21.18	21.13	21.54	22.02	21.14	21.29	22.09	19.37	21.29	21.53
SiO_2	25.46	23.64	24.99	26.57	23.14	23.16	22.57	25.67	25.39	24.25	24.20	24.35	22.66	24.00	24.29
P_2O_5	na	na	na	na	0.04	0.17	0.03	na	na	0.19	na	na	0.03	0.08	0.09
K_2O	0.00	0.54	0.03	0.03	0.02	0.02	0.08	0.02	0.03	0.11	0.19	0.03	0.26	0.01	0.10
CaO	0.00	0.03	0.08	0.00	0.02	0.05	0.01	0.04	0.02	0.40	0.02	0.04	0.02	0.02	0.05
TiO_2	0.06	0.04	0.05	0.01	0.05	0.11	0.16	0.01	0.02	0.08	0.02	0.03	0.09	0.03	0.05
Cr_2O_3	0.05	0.01	0.05	0.00	0.02	0.05	0.03	0.02	0.04	0.03	0.01	0.07	0.05	0.02	0.03
MnO	0.07	0.06	0.04	0.08	na	0.45	na	0.06	0.10	na	0.07	0.10	na	0.22	0.12
FeO	25.54	33.27	25.29	20.87	36.01	36.58	34.29	23.96	27.86	29.77	33.66	29.53	36.06	26.66	29.95
NiO	0.07	0.05	0.08	0.11	0.08	0.31	0.08	0.07	0.05	0.06	0.06	0.16	0.09	0.07	0.09
H_2O	12.30	15.23	12.78	14.51	12.39	10.80	14.42	12.75	10.91	13.18	11.70	11.82	15.55	15.77	13.15
CO_2	na	na	na	na	na	na	na	na	na	na	na	na	na	na	na
F	na	na	na	na	na	na	na	na	na	na	na	na	na	na	na
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.18
Mg0/FeO	0.58	0.16	0.57	0.61	0.20	0.19	0.21	0.65	0.47	0.36	0.25	0.39	0.16	0.44	0,35

Siderite	GR004R1	GR005R2	GR005R3	GR010R45	GR012R6	GR016R7	GR016R89	GR020R10	GR026R11	GR039R12	GR050R13	GR059R1415	GR072R16	GR122R1718	Overall Average
Na_2O	no siderite in sample							NO ANALYSIS			0.88	no siderite in sample	0.94	no siderite in sample	0.91
MgO											3.11		2.28		2.69
Al_2O_3											0.12		0.11		0.12
SiO_2											0.09		0.09		0.09
P_2O_5											0.01		0.05		0.03
K_2O											0.02		0.01		0.01
CaO											0.09		0.07		0.08
TiO_2											0.00		0.00		0.00
Cr_2O_3											na		na		na
FeO											54.21		55.33		54.77
NiO											na		na		na
MnO											3.91		4.64		4.27
H_2O											na		na		na
CO_2											37.56		36.49		37.02
F											na		na		na
Total											100.00		100.00		100.00
								0.05 CO_2			1.76 CO_2		6.85 CO_2		

Sample includes 2 phases of siderite. Average shown excludes 2 "minor" phase spots with high MnO, high MgO, and Low FeO.

SEM analysis at Macalester College

Table 3, Greenstone "Tank" Samples - Mineralogical Composition

	Sample #11920		Sample #11925	
Minerals	1*	2*	1*	2*
Pyrite	1.02	1.02	0.86	0.86
Melanterite	0.14	0.14	0.17	0.17
Siderite	1.08	1.08	1.30	1.30
Quartz	47	48	50	50
Chlorite	33	27	26	22
Sericite	20	20	23	23
Na-Feldspar	0.98	0.98	0.90	0.90
Ilmenite	1.11	1.11	0.98	0.98
Apatite	0.32	0.32	0.46	0.46
Fe-Oxides	--	--	--	--
Zircon	Tr	Tr	Tr	Tr
Tourmaline	Tr	Tr	Tr	Tr
Summation	**104.65**	**99.65**	**103.67**	**99.67**
Al_2O_3 Excess	-0.25	0.92	-0.44	0.28
Add'l Fe Used	1.92	0.18	1.23	0.20
Chlorite MgO/FeO	0.35	0.45	0.35	0.42

1* values are based on average SEM/EDS chemistry for sericite, chlorite, and siderite.
2* values are based on average SEM/EDS chemistry for sericite & siderite and calculated chemistry for chlorite.

Calculated Chlorite Chemistry (Wt. %)

Sample	SiO_2	Al_2O_3	FeO	MgO
11920	24.98	21.50	27.70	12.46
11925	24.77	21.50	28.45	11.92

APPENDIX 2

FIELD TEST PILES: PRECIPITATION, FLOW, REACTION CONDITIONS

Table A2.1. Daily precipitation data for 2000. Precipitation data from the DNR Hibbing Research Site. Rain data is in inches.

Day	Month											
	Jan.	Feb.	Mar.	Apr.	May	June	July	Aug.	Sept	Oct.	Nov.	Dec.
1					0.16			0.62	0.03		0.07	
2										0.15	0.70	
3											0.06	
4												
5									0.82	0.06		
6									0.63		0.15	
7								0.38			0.96	
8					1.45						0.04	
9								0.28				
10							1.84					
11									0.24			
12					0.29	2.22	0.82					
13						0.40	0.15				0.15	
14								0.25				
15					0.12							
16						0.90				1.52		
17								0.02				
18								0.20	0.01			
19						0.46	0.02					
20									0.08			
21						0.94		0.13				
22					0.18							
23						0.72						
24												
25									0.25			
26					0.11	0.25	0.38					
27										1.29	0.40	
28								0.48				
29								0.09				
30						0.15				0.10		
31							0.12	1.56				
Total	0.56	0.45	0.64	0.75	2.31	6.04	3.33	4.01	2.06	3.12	2.53	

Annual total = 22.39, Annual average for Hibbing = 26.93

Table A2.2. Daily precipitation data for 2001. Precipitation data from the DNR Hibbing Research Site. Rain data is in inches.

Day	Month											
	Jan.	Feb.	Mar.	Apr.	May	June	July	Aug.	Sept	Oct.	Nov.	Dec.
1					0.18	0.33						0.02
2							0.11					
3							0.20					
4												
5												0.38
6				0.19	0.69						0.04	
7					0.03	0.07		0.15			0.25	
8								0.46		0.18		
9				1.93	0.24				0.55			
10												
11										1.15		
12				0.84		0.96				0.67	0.09	
13					0.26							
14						1.38		0.51				
15					0.01	0.06	0.03	0.20		0.48		0.02
16				0.56			0.36				0.01	
17						0.16	0.02	0.33				
18						0.01	0.54		0.08	0.06	0.09	
19							0.03					
20					1.19				0.01			
21					0.78							
22					0.33					0.31		
23				1.24	0.02				0.93	0.22		0.45
24				0.01		0.01				0.04	1.05	
25												
26												
27												
28					0.92					0.21		
29							0.65	0.39				
30				0.31			0.02	0.01			0.15	
31				0.23			0.96					
Total	0.31	0.23	0.19	5.31	4.65	2.98	2.90	2.05	1.57	3.32	1.68	0.87

Annual total = 26.06, Annual average for Hibbing = 26.93

Table A2.3. Daily precipitation data for 2002. Precipitation data from the DNR Hibbing Research Site. Rain data is in inches.

Day	Month											
	Jan.	Feb.	Mar.	Apr.	May	June	July	Aug.	Sept	Oct.	Nov.	Dec.
1								0.11				
2					0.04				0.28			
3												
4		0.01						0.05				
5				0.02	0.26				0.04			
6					0.02			0.19		1.39		
7					0.08		1.77	0.02				
8					0.22					0.03		
9					0.06	0.04						
10	0.04			0.18		0.06	0.01	0.67	0.58			
11			0.27									
12	0.01				0.38	0.01		0.76			0.02	
13	0.01			0.01	0.03	0.23						
14	0.04									0.45		
15								1.18	0.01			
16	0.02			0.11								
17				0.15				0.56				0.05
18				0.08		0.41						
19		0.07				0.18			1.11			
20										0.37		
21	0.02		0.02				0.36	0.01				0.02
22							0.04		0.07			
23						8.81			0.08		0.01	
24												
25	0.06	0.07			0.05		0.30					
26				0.03								
27										0.11		
28	0.04		0.22	0.22	0.03		1.07	0.19				
29	0.03			0.08	0.36			0.01	0.07			
30			0.01						0.01			
31							1.61					
Total	0.27	0.15	0.52	0.88	1.53	9.74	5.16	3.75	2.25	2.35	0.03	0.07

Annual total = 27.21, Annual average for Hibbing = 26.93

Attachment A2.1. Page 1 of 10. Field notes on waste characterization piles.

2000

7/18/00 - Started filling bin 1 (low sulfur, 0.02) and finished on 7/21.

7/21/00 - Started filling bin 4 (high sulfur, 0.67) and finished on 7/25.

8/1/00 - After a downpour of 0.67" of rain pile 4 received no flow and pile 1 about 200-300 mLs.

8/2/00- Attempted to measure O_2 and temperature. O_2 meter was giving some strange readings, unsure if the pump was not strong enough or O_2 meter not working.

8/8/00 - .38" of rain: pile 4 had about 4 ½" of leachate in the sump, pile 1 had about ¾" of leachate in the sump, no water dripping. The flow totalizers and meters are not working, there appears to be a problem with the electrical wiring. Installed new flow totalizers in piles 1 and 4.

8/9/00 - AM: pile 4 had no change in water volume since 8/8, the leachate in pile 1 was up to the bottom of the second float switch (3" from top of the sump).

8/14/00 - AM rain gage = .25". No flow to pile 4, still had only about 4" of water in sump and about 60 mLs in collection bottle. Pile 1 had about 4 ½" of water in sump (meter read 60) and approximately 200 mL in collecting sample bottle. The flow totalizer in pile 1 appears to be working and was calibrated. Collected baseline grab samples from the sumps of piles 1 and 4 for nutrients (500 mL) and metals (250 mL). Both piles had some algal growth in the sump and on the pipe fixtures. Pile 4 pH = 8.74, SC = 550, Pile 1 pH = 8.06, SC = 1450.

8/15/00 - 0.30" rain from PM on 8/14. Pile 4 water level the same in sump, SC = 500. Pile1 water nearing the trip switch (8:20 AM) and water dripping from inlet pipe at about 15 to 20mL/30 sec. SC = 1200. More water in the collection bottle than 8/14 which indicates that there was flow but the meter read zero after being reset during calibration.
8/17/00 - 0.02" rain

8/18/00 - 0.20" rain in AM. Pile 4 water level is up to the gravity overflow pipe. Pile 1 has very little water in sump, and water dripping in from stockpile which indicates that the pump had tripped. Water in collecting bottle was 3/4" higher than previous date. Neither flow totalizer is working, still having electrical problems. Meters read 0. Used YSI probe to compare the CG 502 oxygen probe. Some trials produced the same numbers and other trials did not. A more detailed comparison will be performed at a later date.

8/21/00 - Piles 1 and 4 both received flow but flow instrumentation was still out of order. Cleaned the level sensors in both sumps to remove organic film that may affect the sensor. This will have to part of the routine maintenance check.

8/22/00 - 0.13" of rain, flow instrumentation is still out of order and will have to be shipped to the company for repair.

8/25/00 - Pile 4: the water level is ½ way up on the bottom sump switch. Pile 1: the water level is ½ inch above the bottom sump switch, some water in collection bottle.

8/28/00 - Heavy downpour Friday evening (8/25) resulting in 0.48" rain. Pile 1: (1435) sump water 3/4" above bottom sump switch and pile is flowing at a slow drip. Sample collection bottle was full, replaced with new bottle. Brown slime covering bottom of sump, with clumps of green algae present. Meter reads 0. Pile 4: 1 1/4" water in the sump with about 100 mL in the collection bottle. No flow. Slight oil sheen on surface of water. Meter not functioning yet.

8/29/00 - 0.09" rain from previous evening. Pile 1: Water in the sump measured 1" more than on 8/28 and the collection bottle had about mLs of water which indicates that the pump had been triggered. Pile 4: Replaced collection bottle to measure pH and SC. Sampled metals and nutrients from pile 1, not enough water to sample pile 4.

8/31/00 - 1.56" rain between 8/30 PM and 8/31 AM, also heavy mist off and on all day. Replaced sample bottle in pile 1.

9/1/00 - Pile 4: Water is ½ way between sump switches, water dripping into sump. Sample bottle is 3/4 full which indicates that the pump had triggered. Meter not working yet. Pile 1: Water is ½ way between switches, water dripping into sump. Sample bottle is approximately 1/5 full which indicates that the pump had triggered, meter still reads zero.

9/5/00 - 0.82" rain over the weekend. Pile 4: There is about 2" of water in the sump and the collection bottle is full (2000 mLs). Flow rate at 1247 was 31 mL/min. Pile 1: There is 2 ½" water in the sump and the collection bottle is full (2000 mLs). Flow rate was 15.5 mL/min. Bin 3: Started loading with the 0.39% S rock. Loaded 2 truck loads into the pile and took 4 samples from each load once they were placed in the pile. One random sample from the east side of the pile, one from the west, one from the north, and one from the south.

9/7/00 - 0.63" rain. Pile 4: There is about 6" of water in the sump and the collection bottle almost ½ full, Flow rate is at 155 mLs/ 30 sec. Pile 1: There is a little over 6" of water in sump and the collection bottle is 1/4 full, flow rate is at 140 mLs/30 sec. Pile 3: Pile is complete. Took rock samples as described on 9/5.

9/11/00 - 0.24" rain over weekend. Pile 4: There is about 2 ½" of water in the sump and the collection bottle is full (exchanged for new bottle). Flow rate is at 14 mLs/min. Pile 1: There is about 4" of water in the sump and the collection bottle is 3/4 full (exchanged for new bottle). Flow rate is at 9 mLs/min. There is a considerable amount of algae present in the sump. Pile 3: Received some flow (water in the sump is to the top of the bottom sump switch), Pile is not currently flowing. Sump needs cleaning due to the presence of algae.

Attachment A2.1. Page 3 of 10. Field notes on waste characterization piles.

9/12/00 - Pile 4: There is 7" water in the sump and the collection bottle is full (exchanged for new bottle) Flow is at 1.5 mLs/min. Pile 1: There is about 6" of water in sump and the collection bottle is full (exchanged for new bottle). Pile is not currently flowing. Still having problems troubling shooting the electrical problem. Pile 3: No flow.

9/15/00 - Pile 4: 3" of water in the sump, no flow. Pile 1: About 1" of water in the sump, no flow.

9/18/00 - Started filling bin 2. Took additional rock samples using the same procedure as pile 3.

9/19/00 - Pile 4: Water level in the sump is unchanged from 9/15, no water in the collection bottle, and the pile is not flowing. There is some green algae starting appear in the sump. Pile 1: Water level in the sump is unchanged from 9/15, no water in the collection bottle, and the pile is not flowing. There is brown/green algae present in the sump. Pile 2: Pile is complete.

9/26/00- Pile 4: 2 ½ to 2 3/4" water in sump (0911), meter read 98, accidentally hit sump switch, now 1 1/4" (water half way up to sump inlet pipe). Grey plastic shavings from meter box in sump, collection bottle about 1" water. Pile 3- dry with grey shavings. Pile 2 - 1 3/4" water in sump, collection bottle empty. Pile 1 - 5 1/4" water in sump, collection bottle a <1" with grey shavings.

10/6/00 - Pile 4 - 1" water in sump, and 1" water in collecting bottle, Pile 3 - empty, Pile 2 - 1 5/8" water in sump, collection bottle empty, Pile 1 - 6 3/4" water in sump, >1 " water in collecting bottle.

10/11/00 - Same as 9/26, switched collection bottles (1126) on piles1 (100 mL) and 4 (250 mL) to sample SC, and pH. Power to pumps off and collection bottles disconnected.

10/17/00 - Power still off so water is not collecting in bottles. Rained (1.52") over weekend, so collected grab samples of sumps from the 4 piles. All still flowing (1020) Pile 4 - 33 mL/min, Pile 3 - 25 mL/min, Pile 2 - 15 to 17 mL/min, Pile 1 - 25 mL/min. Piles 1 and 4 had a greenish tint to water and some algae on sump fixtures, and Piles 1, 3, and 4 had grey shavings in sump from drilling of electrical boxes. Pile 4 also had algae in sump.

10/27/00 - 1.29" rain on 10/26. All piles flowing. At 0930 pile 4 - 87 mL/min, Pile 3 - 93 mL/min, Pile 2 - 78 mL/min, Pile 1 - 76 mL/min. At 1430 Pile 4 - 71 mL/min, Pile 3 - 71 mL/min, Pile 2 - 72 mL/min, and Pile 1 - 67 mL/min.

11/1/00 - 0.7" rain. No evidence of erosion. Pile 4 - 150 mL/10 sec (960 mL/min), Pile 3 - 160mL/ 13 sec (688 mL/min) , Pile 2 - 160 mL/13 sec (688 mL/min), Pile 1 - 160 mL/ 10 sec (900 mL/min). Meters working.

Attachment A2.1. Page 4 of 10. Field notes on waste characterization piles.

11/6/00 - more rain last week, 0.15". Strong flow, all pumps about ½ to3/4 full, 100 to 200 mL in collection bottles (except #2, valve to bottle was closed). Flow meter reading Pile 4 - 11, Pile 3 - 171, Pile 2 - 170, Pile 1 - 156.

11/7/00 - 0.96"rain. Tripped each pile sump so that could get an instantaneous sample from inlet pipe (water in collection bottle had been diluted as a result of sump calibration). Changed number on meters from 1200 to 1150 (1415), noted that sump screens needed cleaning from plastic chips and algae.

11/8/00 - 0.4"rain. Flow and volume measurements taken. Cleaned sumps and flow meter screens.

11/9/00 - Freezing rain/snow overnight. Meter and flow measurements (1545). Changed collection bottle. Still flowing 15 to 20 mL/min.

11/13/00 - Rain snow mix over the weekend. Meter and flow measurements (1345). Still flowing 12 to 14 mL/min.

11/14/00 - Meter and flow measurements (1050). Piles still flowing 2 to 22 mL/min.

11/16/00 - Meter and flow measurements (1300). Piles flowing 5 to 6 mL/min.

11/20/00 - No flow, piles starting to slush and freeze. Switched collection bottles for spring.

11/22/00 - Removed remaining water and cleaned piles for winter, also drained sump pumps.

11/22 to 12/4 - Water flowing at some point in piles 2 through 4.

12/4/00 - Meter readings Pile 3 -1296, Pile 4 - 635.

2001

3/22/01 - Warm temperatures led to flow in some tanks (Pile 2,3). Snow left only on north side of piles.

3/23/01 - Flow in Piles 2 - 4, ranged from 16 - 4 mL/min. Weather turning cool again.

3/27/01 - Electrical disturbance in Pile 2. Reset meter to zero. Sump was nearly full prior to trip for reset test. No flow in piles.

4/2/01 - Slow (~1mL/min) flow in Piles 2 and 3. Snow previous night and AM. Snow remains on north side of rock piles.

Attachment A2.1. Page 5 of 10. Field notes on waste characterization piles.

4/4/01 - Pile 2 - ¾" from tripping, tripped sump to see if meter was working. Meter read 13 after trip.

4/6/01 - There is standing water in pile 3 with a film on the surface. The outlet pipe must be frozen.

4/9/01 - Pile 4 - standing water in pile (ice in pipe), exchanged bottles from Piles 2 & 3 because they were full.

4/10/01 - Switched collection bottle in Pile 1 .

4/11/01 - Pile 4 output pipe still frozen, pumped about 1/2 gal of hot water in to thaw pipe (at 1100). Pipe began to flow, after a few minutes flow was 250 mL/9 sec. 1 trip resulted in a meter reading of 654. At 1300 the meter read 908, at 1520 meter read 1531.

4/12/01 - Pile 4 changed collection bottle.

4/19/01 - Pile 2 changed collection bottle.

4/20/01 - Piles 1, 3, and 4 changed collection bottles.

4/25/01 - Collected samples for analysis.

5/4/01 - Checked calibration of meter to sump. Power outage caused erroneous readings.

5/7/01 - Checked calibration of meter/sump again, cleaned all screens (clogged with algae, insects).

5/10/01 - Collected samples for analysis. All outlets are still dripping.

5/23/01 - Collected samples for analysis.

6/4/01 - Cleaned algae from sumps, forgot to disconnect sample bottle on Piles 2- 4 when cleaning.

6/8/01 - Put new collection jars in piles due to contamination of algae when cleaning on 6/4.

6/15/01 - Collected samples for analysis.

6/21/01 - Measured oxygen and temperature of rock piles.

7/11/01 - Measured oxygen and temperature of rock piles.

Attachment A2.1. Page 6 of 10. Field notes on waste characterization piles.

7/13/01 - Cleaned sumps and pumped out water.

7/19/01 - The cover on Pile 2 flew open during a storm the previous evening. Noticed that there is algae (red) &/or possible mold (black spots) in most of the tubing from pump to overflow pipe, also a little green algae tubing to collection jars.

7/24/01 - Measured oxygen and temperature of rock piles.

7/30/01 - Rain AM, mist and fog.

7/31/01 - Cleaned algae from pump screens, overcast, humid.

8/1/01 - Thunderstorms previous evening, 0.96 in rain. Wind may have blown tiny rock particles around, also slight erosion activity between piles. Humidity 100% (all week). Steady flow still at 1045. Sumps in piles 1 and 4 stained brown with a little algae, pile 2 very green, pile 3 lots of filamentous-like algae. Changed collection jars on piles 1, 2, and 4 (in refrigerator for analysis). Pile 3 collection jar was only half full.

8/2/01 - Collected flow from overnight and added to yesterdays collection jars for analysis (piles 1, 2, and 4). Collected sample jar from pile 3 for analysis. Measured oxygen of piles with YSI. Calibrated YSI (@ 25C in lab while probe in chamber).

8/8/01 - Thunderstorms 4:30 - 5AM, heavy winds, rain = 0.15 in. Visual inspection of piles: Pile 1 - sump brown with green filaments of algae, Pile 2 - sump thick green filaments of algae, Pile 3 - sump green algae, not as thick as sump 2, Pile 4 - sump stained ark brown with some green algae. All sumps flowing at a rapid drip. Thunderstorms again at 4:30 PM.

8/9/01 - Thunderstorms at 12:30 AM, heavy winds, rain= 0.45 in (includes rain from 8/8/01 afternoon storm).

8/20/01 - Collected samples for analysis. Sumps full of algae. Cleaned screens.

8/23/01 - Measured oxygen content of the rock piles using YSI. Possible errors when the temperature reaches 35 C in the measuring chamber.

8/29/01 - Cleaned sumps on the prediction piles. Measured water volume and removed water while cleaning.

8/30/01 - Pile 1 pump was not working and resulted in lost flow. The lower sump switch was stuck in the off position, and water exited the sump via the outflow pipe.

9/12/01 - Collected samples for analysis and disposed of remaining sample.
Cleaned algae from screens.

Attachment A2.1. Page 7 of 10. Field notes on waste characterization piles.

9/24/01 - 0.93" rain on 9/22/01, heavy frost over night. Adjusted valve on pile #2 as there was very little water in the sample jar (AM). Cleaned algae from the valves and tubing around the valves that connect to the collection jars (PM). This algae may have been restricting flow to the sample collection jars.

9/25/01 - Oxygen measurements of the prediction piles ranged from 20 to 21%. Temps averaged 13.1 C for the top of the piles and 14.8 C for the bottom of the piles.

9/28/01 - Cleaned sumps with bleach solution, rinsed and pumped out remaining water. Will need to add 3L to the next meter reading for compensation of the water below the sump switch. Sumps switches on piles 1 and 2 were sticking in the on position. The problem with pile 1 cleared up after the sump was cleaned. The sump in pile 2 was tilted causing the switch to remain in the on position.

10/9/01 - No flow in sumps. Lost power at 10:30, power on at 11:30.

10/10/01 - All piles are flowing.

10/11/01 - Slow drip in all sumps. Collected samples for analysis. Flow rate for input water was a slow drip. The connection to flow meter was loose and leaked into the plastic pile (approximately 4" of water).

10/17/01 - Oxygen measurements of the prediction piles ranged from 21.6 to 23.8%. Temps averaged 11.7 C for the top of the piles and 8.9 C for the bottom of the piles. Prediction piles dripping in late AM, leftover from earlier rain or frost thawing.

10/19/01 - Placed heat lamps near sumps to prevent the pumps from freezing in cold weather.

10/23/01 - Rain evening of 10/22. Cleaned all screens on flow meters. Crud from inside tubing and small insects clogged the screens. Pile 3 had white fibrous material (hair-like) clogging the screen. Piles all flowing with fast drip to a trickle.

10/30/01 - Collected water samples for analysis.

11/8/01 - Rain PM of 11/7. Sponged water out of sumps.

11/26/01 - Rain over weekend (Sat.), snow today with more predicted overnight. Temps to drop by end of week. Heat lamps were out in Pile's 3 and 4, replaced bulbs. All sumps had slow, steady flow into them.

11/28/01 - Tripped sump switches so that all water emptied out of them. Switched sample jars on all piles and collected water for analysis. Pile's 2 through 4 still flowing, Pile #1 stopped flowing. All sumps had some green and brown, Pile #1 had fuzzy green algae (most likely from heat lamp).

12/7/01 - Slow flow

12/11/01 - Slow flow

12/13/01 - All piles flowing except Pile #1. Temperature of the rock piles range from 0.9 to1.8C for the top of the piles and 2.0 - 4.3C for the bottom of the piles. All rock piles have settled over time due to erosion activity.

12/17/01 - Slow flow in all piles.

12/19/01 - No flow in all piles.

12/20/01 - Changed collection jars in all piles.

2002

4/10/02 – No flow in piles yet. Read flow meters for baseline numbers.

4/14/02 – AM sprinkles, sunny, light wind. Flow began sometime between 4/10 and 4/14. Temps on 4/12 and 4/13 in low 60's. Temperature readings of rock piles ranged from –0.3 to 0°C. Some slumping of rock piles, need to do some landscaping/re-build berms to prevent runoff from mixing between piles in heavy rains.

4/28/02 – Collected samples for metals, sulfate, silica and nutrient analysis. Some algae in Piles 3 & 4 sumps (probably came from sump inlet pipe, and heat lamps).

5/15/02 – Measured oxygen ant temperature of piles. Raged from 14.7 (one low value) to 20.6%.

6/4/02 - Collected samples for metals, and sulfate analysis. All sumps had some algae, and water in sumps was cloudy. Cleaned out sumps.

6/24/02 – Severe thunderstorms and heavy rain June 22 to 24 totaling 8.81" of precipitation. Collected samples for metals, sulfate, silica and nutrient analysis.

7/8/02 – 1.77" rain previous evening to early AM. In AM collected samples from inlet pipe directly for SC, pH and Alkalinity comparison to water in sample collection jars. Collected sample jar water in PM to sample for metals and sulfate. Sumps had thick brown, brown and green, or green algae in them.

7/11/02 – Tripped sumps, recorded meter readings (volume), and cleaned sumps (problems with algae growth). Measured oxygen and temperature of piles. Oxygen ranged from 16.7 to 19.4%, and temperatures ranged from 16.8 to 20.7 C.

Attachment A2.1. Page 9 of 10. Field notes on waste characterization piles.

7/26/02 – Painted covers of sump housing black to reduce sunlight.

7/29/02 – Tripped all sumps, collected water samples for metals and sulfate analyses.

8/1/02 – 1.61" rain previous evening. Water in Pile 1 sump was cloudy. Tubing came loose from sample collection jar in Pile 2 so very little sample was collected. Collected water samples directly from the inlet pipe for SC, pH.

8/2/02 - Cleaned algae from tubing and valve connected to sample collection jars in all piles. Collection jars from Piles 1 and 4 were nearly full, dumped approximately 2/3 of the sample.

8/6/02 – DR cleaned sumps, and checked calibration of sumps (± 1 liter). Add 3 liters to the next meter reading to accommodate for the volume from the sump bottom to the first switch.

8/12/02 – 0.74" rain early AM. All sumps had a strong, steady flow. Removed a small volume of water from the collection jars in Piles 1, 2, and 4 to make room for the rest of the flow. Will wait one day before getting a sample for analysis. Pump for Pile 2 sump stuck on. Sump was not level in the pile.

8/12/02 - Collected water for metals, sulfate and nutrient analyses, and discarded the rest of the sample. Tripped all sumps prior to sample collection.

8/15/02 – Rain PM of 8/14 and all day of 8/15, 0.86". Opened valve from pump to sample collection jar on pile 2 (left closed after last sampling).

8/16/02 – 0.32" more rain in late afternoon on 8/15. Sample collection jar on Pile 4 full, removed some water.

8/19/02 – Rain over weekend, 0.56". Sample collection jars on Piles 1 and 4 were full, emptied about 1/4th of sample.

8/26/02 - Measured oxygen and temperature of piles. Oxygen ranged from 15.6 to 19 %, and temperatures ranged from 15.6 to 19.1C.

9/10/02 – 0.58"rain early AM. Collected water samples directly from the inlet pipe for SC and pH comparison with sample collection jar. Lower sump switch on Pile 3 stuck, resulting in lost flow into collection jar. Pile 1 some brown particulate accumulating on bottom of sump, Pile2 brown clumps with little green, Pile 3 small green speckled sump, Pile 4 dark green on inlet spout and lower sump switch.

9/19/02 – 1.1" rain early AM. All sumps had a strong, steady flow. Collection jars on piles 1, 2 and 4 were full and replaced with new bottles. Water in Pile 1 sump was cloudy. Collected water samples directly from the inlet pipe for SC and pH comparison with sample collection jar

Attachment A2.1. Page 10 of 10. Field notes on waste characterization piles.
water.

9/20/02 – Collected water sample from Pile 3 collection jar. All sumps had various green and brown vegetation growths in them.

10/7/02 - 1.39" rain early over weekend. Collected water sample for analysis.

10/28/02 – 0.11" rain. Collected water sample for analysis.

11/7/02 – Cleaned sumps for excess vegetation growth and slight sediment or precipitate.

11/15/02 – Pumped sumps dry and added second light to prevent freezing.

Attachment A2.2. Page 1 of 3. Estimation of unmeasured flow from greenstone prediction piles in 2000.

The sampling instrumentation for the field piles includes an electronic flow meter and flow totalizer. When this instrumentation was installed there were problems with the electrical wiring to the meters. As a result all meters and totalizers were damaged and were sent back to the distributor for repair. Due to these problems flow was not recorded for pile 1 from July 21st to November 1st, for pile 2 from September 18th to November 1st, for pile 3 from September 5th to November 1st, and for pile 4 from August 14th to November 9th, 2000.

Flow estimates for these periods, as well as periods in the future when flow recording problems are encountered, will be based on the yield coefficient for each pile and precipitation during the period of unmeasured flow. The yield coefficient is the output from the pile over a given period of time divided by the input from rainfall during that same period. The following calculation is for the input volume to the piles.

20 ft. x 20 ft. x (ft/12 in) x P = 33.3 P ft^3 or 940 P Liters, where P is precipitation in inches.

After the meters were installed there were one to three week periods in 2000 for which yield coefficients could be calculated. The yield coefficients for these periods ranged from 0.50 to 0.63 (table I). These yields are in good agreement with a range of 0.44 to 0.58 reported for an earlier field study conducted by the MN DNR (Eger et al., 1985). Since these yield coefficients represent fairly short periods, 2001 data was used to estimate lost flow.

2001 yield coefficients for two periods were calculated (tables II and III). Yield coefficients for the approximate period of lost flow in 2000 (August to November) and for the entire field season (April to December) are presented in tables II and III, respectively. The yield coefficients for the August to November period were in fairly good agreement with the 2000 results with the exception of Pile 2 which was slightly higher. The yield coefficients for the entire field season (0.73 – 0.85) with the exception of pile 2 were much higher than either the 2000 or 2001 August to November results as well as data collected from an earlier field study conducted by the MN DNR.

At this time it is not clear why the yield coefficients for the entire field season seem higher than would be expected. Since the data from August to November for the 2001 field season is in fairly good agreement with the 2000 data as well as the earlier field study it was determined that those yield coefficients would be the most accurate to use when determining the 2000 lost flow (tables IV - VII).

Attachment A2.2. Page 2 of 3. Estimation of unmeasured flow from greenstone prediction piles in 2000.

Table I. 2000 yield coefficients for greenstone prediction piles (Piles 1-3: 1-20 November; Pile 4: 13-20 November).

PILE	RAIN (in.)	INPUT (L)	OUTPUT (L)	YIELD COEFFICIENT
1	2.13	2002	1114	0.56
2	2.13	2002	1174	0.59
3	2.13	2002	1260	0.63
4	0.15	141	71	0.50

Table II. Yield coefficients for the period 2 August through 28 November, 2001.

PILE	RAIN (in.)	INPUT (L)	OUTPUT (L)	YIELD COEFFICIENT
1	11.55	10857	6864	0.63
2	11.55	10857	8781	0.81
3	11.55	10857	5622	0.52
4	11.55	10857	6641	0.61

Table III. Yield coefficients for the entire 2001 field season (1 January – 22 December).

PILE	RAIN (in.)	INPUT (L)	OUTPUT (L)	YIELD COEFFICIENT
1	25.61	24,073	20383	0.85
2	25.61	24,073	20029	0.83
3	25.61	24,073	17486	0.73
4	25.61	24,073	19606	0.81

Table IV. 2000 lost flow estimations based on yield coefficients from table II for pile 1.

SAMPLE DATE	RAIN (in.)	INPUT FLOW (L)	PILE 1 LOST FLOW (L)
8/14	2.03	1908	1202
8/29	1.22	1147	722
9/12	3.28	3083	1942
10/17	2.07	1946	1226
11/07	1.39	1306	823
Total	9.99	9390	5915

Attachment A2.2. Page 3 of 3. Estimation of unmeasured flow from greenstone prediction piles in 2000.

Table V. 2000 lost flow estimations based on yield coefficients from table II for pile 2.

SAMPLE DATE	RAIN (in.)	INPUT FLOW (L)	PILE 2 LOST FLOW (L)
10/17	2.07	1946	1576
11/07	1.39	1306	1058
Total	3.46	3252	2634

Table VI. 2000 lost flow estimations based on yield coefficients from table II for pile 3.

SAMPLE DATE	RAIN (in.)	INPUT FLOW (L)	PILE 3 LOST FLOW (L)
9/12	1.45	1363	709
10/17	2.07	1946	1012
11/07	1.39	1306	679
Total	4.91	4615	2400

Table VII. 2000 lost flow estimations based on yield coefficients from table II for pile 4.

SAMPLE DATE	RAIN (in.)	INPUT FLOW (L)	PILE 4 LOST FLOW (L)
8/14	2.03	1908	1164
8/29	1.22	1147	700
9/12	3.28	3083	1881
10/17	2.07	1946	1187
11/07	3.33	3130	1909
11/20	0.19	179	109
Total	12.12	11,393	6950

REFERENCES

Eger, P., Lapakko, K. 1985. Heavy metals study: Progress report on the field leaching and reclamation program: 1977-1983. Minnesota Department of Natural Resources, Division of Minerals. St. Paul, MN. 53p.

Attachment A2.3. Page 1 of 3. Dissolved oxygen and temperature field notes.

8/2/00	Used GC 502 meter, calibrated to 20.9 %. Set up pump and purged volume of tubing. Practiced oxygen measurements on Piles 1 & 4. Oxygen measurements ranged from 19.6% to 20.9%. Rock pile temperatures ranged from 18.1 to 23.9°C.
8/15/00	Calibrate GC 502 oxygen meter to 20.9% with probe in air. Once probe was placed in the measuring chamber, oxygen fell to 17.3%. Measured percent oxygen for Piles 1, 3, and 4. Oxygen measurements ranged from 8.5% to 17.3%. At the end of the recording time, removed probe out of measuring chamber and oxygen reading was 17.5%. Rock pile temperatures ranged from 18.1 to 23.9°C
8/18/00	Early AM, first comparison of YSI and GC 502 oxygen meters. Calibrated YSI according to conditions at site (air temp of 22C, and elevation of 1600' = saturation of 8.28 mg/L). GC 502 calibration was set to 20.9 % with probe in air. Performed a preliminary comparison on Piles 1 and 4. Oxygen readings were in the 10 to 17 % range.
	Late AM to early PM, performed a more rigorous comparison of the oxygen meters. Connected both YSI and CG 502 oxygen probes in tandem and read meters simultaneously at 1, 3, 5, and 10 minute intervals. Some difficulties keeping the YSI probe from leaking air into its measuring chamber. This was corrected by placing a layer of silicon around the rubber stopper that held the probe into the measuring chamber. The majority of the readings from the two meters differed by 1 to 2 %. The YSI meter typically produced the higher percent oxygen reading. On one sampling port (Pile 4, upper, 10'), the YSI meter read as much as 4.9% higher than the GC 502 meter. Oxygen readings were in the 16.9 to 22.8 % range. Rock pile temperatures ranged from 17.1 to 19.8°C
9/26/00	Measurement of oxygen in Piles 1,3, and 4 using the GC 502 meter. Meter was calibrated at 21.0% with the probe in the air. Oxygen readings were collected at 1, 3, and 5 minute intervals, and measurements ranged from 8.8 to 17.3%. Rock temperatures ranged from 11.5 to 12.2 C for the tops of the piles, and 11.3 to 14.6°C for the bottoms of the piles (partly cloudy conditions).
3/27/01	Measurement of oxygen in Piles 1 through 4 using the GC 502 meter. Meter was calibrated at 21.1% with the probe in the air. Oxygen readings were collected at 1, 3, and 5 minute intervals, and measurements ranged from 6.7 to 19.3%. Rock temperatures ranged from –2.2 to –1.60 C for the tops of the piles, and –0.5 to –0.1°C for the bottoms of the piles (sunny changing to partly cloudy).

Attachment A2.3. Page 2 of 3. Dissolved oxygen and temperature field notes.

6/21/01 Measurement of oxygen in Piles 1 through 4 using the YSI meter. Oxygen readings were collected at 1, 3, and 5 minute intervals, and measurements ranged from 16.7 to 22.0%. Rock temperatures ranged from 14.4 to 15.5 °C for the tops of the piles, and 11.8 to 13.1°C for the bottoms of the piles (partly cloudy conditions).

7/11/01 Measurement of oxygen in Piles 1 through 4 using the YSI meter. Oxygen readings were collected at 1, 3, and 5 minute intervals, and measurements ranged from 18.8 to 21.1%. Rock temperatures ranged from 17.6 to 20.0°C for the tops of the piles, and 14.6 to 15.6°C for the bottoms of the piles (partly cloudy, air temp 70's). It appears that the YSI reads high oxygen (%) at high temperatures.

7/24/01 Measurement of oxygen in Piles 1 through 4 using the YSI meter. Oxygen readings were collected at 1, 3, and 5 minute intervals, and measurements ranged from 20.5 to 21.9%. Rock temperatures ranged from 21.4 to 23.2°C for the tops of the piles, and 17.4 to 18.4°C for the bottoms of the piles (mostly cloudy, windy). It appears that the YSI reads high oxygen (%) at high temperatures.

8/2/01 Measurement of oxygen and temperature of rock piles using YSI meter. Calibration of meter in lab with probe in chamber. Oxygen readings were collected at 1, 3, and 5 minute intervals, and measurements ranged from 15.3 to 19.4%. Rock temperatures ranged from 19.0 to 20.6°C for the tops of the piles, and 17.1 to 17.9°C for the bottoms of the piles.

8/23/01 Measurement of oxygen and temperature of rock piles using YSI meter. Calibration of meter in lab with probe in chamber. . Oxygen readings at 5 minute intervals ranged from 13.9 to 22.2%. rock temperatures ranged from 18.6 to 20.0°C for the tops of the piles, and 16.7 to 18.0°C for the bottoms of the piles. YSI meter readings erratic during Pile 2 bottom 10' measurement. Let probe sit during lunch and it bounced back to normal. Both lower sampling ports of Pile 3, and one of Pile 4 appeared to have obstructed tubing.

9/25/01 Measurement of oxygen and temperature of rock piles using YSI meter. Calibration of meter in lab with probe in chamber. Oxygen readings at 5 minute intervals ranged from 20.2 to 20.9%. rock temperatures ranged from 12.8 to 12.9°C for the tops of the piles, and 13.8 to 15.1°C for the bottoms of the piles. The lower sampling ports (5') of Pile 3 and 4 appeared to have obstructed tubing.

10/17/01 Measurement of oxygen and temperature of rock piles using YSI meter. Calibration of meter in lab with probe in chamber. Oxygen readings at 5 minute intervals ranged from 22.9 to 25.4%. rock temperatures ranged from 8.5 to 9.2°C for the tops of the piles, and 11.5 to 11.8°C for the bottoms of the piles. The lower sampling ports (5') of Pile 3 and 4 appeared to have obstructed tubing.

Attachment A2.3. Page 3 of 3. Dissolved oxygen and temperature field notes.

5/15/02 Measurement of oxygen and temperature of rock piles using YSI meter. Calibration of meter in lab with probe in chamber. Oxygen readings at 5 minute intervals ranged from 14.8 to 20.6%. rock temperatures ranged from 3.0 to 4.4°C for the tops of the piles, and 0.7 to 2.7°C for the bottoms of the piles. The lower sampling ports (5') of Pile 3 and 4 appeared to have obstructed tubing.

7/11/02 Measurement of oxygen and temperature of rock piles using YSI meter. Calibration of meter in lab with probe in chamber. Oxygen readings at 5 minute intervals ranged from 16.7 to 19.4%. rock temperatures ranged from 19.8 to 20.7°C for the tops of the piles, and 16.8 to 17.5°C for the bottoms of the piles. The lower sampling ports (5') of Pile 3 and 4 appeared to have obstructed tubing.

8/26/02 Measurement of oxygen and temperature of rock piles using YSI meter. Calibration of meter in lab with probe in chamber. Oxygen readings at 5 minute intervals ranged from 15.6 to 19.0%. rock temperatures ranged from 17.8 to 19.1°C for the tops of the piles, and 15.6 to 16.9°C for the bottoms of the piles. The lower sampling ports (5') of Pile 3 and 4 appeared to have obstructed tubing.

Attachment A2.4. Page 1 of 3. Limestone addition field notes.

2000

10/24/00 - Filled limestone tanks.

10/27/00 - 1.29" rain on 10/26. No flow into the limestone tanks.

11/1/00 - 0.7"(or 0.07) rain. No flow.

11/6/00 - 0.15" rain, limestone tanks 1/5 full (1130), flow = slow drips

11/7/00 - 0.96" rain. Measured water volume with rain stick (in). First sample collected from tanks (-750 mL). Pumped water to sump. Unable to get all water out so remeasured water for leftover volume. Tanks still flowing slow drip (10 to 20 mL/drip).

11/8/00 - All tanks still dripping slowly. Measured volumes (924).

11/9/00 - All tanks had frozen pipes (1530). Measured volumes.

11/13/00 - Rain and snow over the weekend. All tanks had frozen pipes, however, must have been slight flow. Measured volumes (1245), pumped out water and sponged tanks dry.

11/14/00 - No flow, pipes frozen.

2001

3/22/01 - Noticed flow on 3/21. Measured volumes (1330), collected 500 mL samples for analysis, then pumped out water and sponged tanks dry. Water had a greenish-brown color.

3/23/01 - All tanks had some water (frozen), but not enough to cover bottom of sump. Weather turning cool again and inlet pipes frozen.

4/2/01 - Measured volume of water in sumps with rain stick. No water in sump 1 and 4 due to upheaval from ice. Some ice in most outflow pipes. Did not remove water from piles.

4/3/01 - All piles have heaved with melt during previous day and freeze overnight. Unable to reset piles due to ice in sand.

4/6/01 - Emptied piles (measured volume by pouring into 10L bucket). Attempted to reset piles, but still some ice under piles.

4/9/01 - 1.9" rain on 4/7. Collected water samples from all piles, but did not measure volumes due to upheaval of piles again.

Attachment A2.4. Page 2 of 3. Limestone addition field notes.

4/11/01 - Unable to take normal measurement for volume (sump upheaval), so took readings at the 4 corners of sump and averaged them. Pumped out all tanks.

4/13/01 - Measured volume of water with calibrated red buckets to nearest 0.5 L. Reset sumps #3 & #6.

4/17/01 - All tanks approximately 1 inch of water, tanks 1 and 4 dripping, all have ice.

4/20/01 - Measured water volume and dumped out water.

4/25/01 - Collected samples for analysis, and emptied sumps.

5/4/01 - Measured flow and emptied sumps.

5/10/01 - Collected samples for analysis and emptied sumps. All tanks appeared to be dripping except 4 and 5.

5/14/01 - Measured sumps for water volume. Outlet pipes from tanks have algae in them.

5/23/01 - Collected water samples for metal analysis, then pumped out remaining water. All sumps had yellow/brown water with floating algae, and tent caterpillars. Algae in tank pipes.

5/29/01 - Water in sumps is yellowish/brown. Tent caterpillars everywhere.

6/1/01 - Tent caterpillars on and in sump. Cleaned caterpillars out of sumps, but did not empty water.

6/9/01 - Measured water volume, then cleaned sumps and dumped out water (yellowish/green).

6/12/01 - Rain over the weekend. Measured water volume, but did not dump water. Water yellowish/green, and some caterpillars were present. Plants starting to grow in rocks in the tanks

6/14/01 - Rain storms previous evening. Measured water volume, but did not empty.

6/15/01 - Collected water samples for analysis. Measured water volume, but did not empty water. Algae present in most piles.

6/25/01 - Measured water volume and emptied sumps.

7/11/01 - Removed plants growing in tanks.

Attachment A2.4. Page 3 of 3. Limestone addition field notes.

8/1/01 - Thunderstorms previous evening, 0.96". Water stained in tanks: (1) light brown, (2) green, (3) green/brown with filamentous clump of green and orange, (4) light green with some algae clumps from inlet pipes, (5) green with few green clumps, (6) mostly clear with slight green tint. Also, usual spiders and some pin head sized insects (Collembola?).

8/2/01 - Collected water samples for analysis, and recorded water volume.

8/8/01 - Thunderstorms early AM with high winds. Tanks had low flow but not measurable.

8/20/01 - Collected water samples for analysis, and recorded water volume.

9/12/01 - Collected water samples for analysis, and recorded water volume. Emptied water from sumps.

9/24/01 - Heavy frost overnight, 0.93 inches of rain on 9/22.

9/28/01 - Took measurements of sumps and pumped water out. Water in all sumps slightly green, with tank 6 having the clearest water.

10/11/01 - Rain on Wednesday. Measured volume in tanks and pumped tanks dry. All tanks slowly dripping, 1 drop/3sec.

10/30/01 - Collected water samples for analysis, and pumped sumps dry.

11/26/01 - Rain over weekend (Saturday). Snow and wind today with more predicted overnight. Measured volumes from tanks, collected sample for analysis, bailed water out of sumps. Pipes from tanks to sumps frozen.

12/01 - Have thawing and freezing conditions. Some lost flow due to loose pipes from tank to sump. Very little water in sumps.

APPENDIX 3

DRAINAGE QUALITY FROM FIELD PREDICTION PILES AND LIMESTONE ADDITION TANKS

Prediction Piles

Limestone Addition Tanks

Attachment A3.1. Anomalous drainage quality data. The data have been verified to be as reported values (PPM), and appear to be anomalous. Anomalous data have been omitted from the cumulative mass release tables and figures.

Table	Reactor	Comment
Table A3.2	Prediction Pile 0.20% S	SO4 value 17.2 (4/9/01).
Table A3.3	Prediction Pile 0.39% S	SO4 value 5.14 (4/9/01).
Table A3.4	Prediction Pile 0.67% S	Ca value of 126 and Zn value of 126 (8/14/01)

Table A3.1. Drainage quality for the 0.02% sulfur field prediction pile 1.

Concentrations are in mg/L, pH is in standard units, conductivity is in μS/cm, and net alkalinity is in mg/L as $CaCO_3$.

Date	Volume (L)	Conductivity	pH	Net Alk	SO_4	Ca	Mg	Na	K	Co	Cu	Ni	Zn
08/14/00	1202	1450	8.06	35	42.0	122	29.6	114	19.2	<0.002	0.006	<0.002	<0.002
08/29/00	722	1525	8.04	60	46.4	140	34.3	116	16.5				
08/31/00		950	7.45	35									
09/12/00	1942	5500	7.96	50	102	576	78.7	396	36.8				
10/11/00		7500	7.95	65									
10/17/00	1226	4500	7.39	50	102	468	57.9	375	34.4	0.003	0.017	0.004	<0.002
10/27/00		3700	7.41										
11/07/00	1587	1275	7.57	50	58.3	121	17.8	197	24.1	<0.002	0.009	<0.002	0.006
11/20/00	350	2100	7.78	35	42.0	184	22.8	205	21.5				
04/10/01	2702	525	7.50	45	22.7	34.6	3.87	63.8	7.8	<0.002	0.005	<0.002	<0.002
04/25/01	3219	600	7.84	60	22.2	43.3	6.30	57.6	7.4				
05/10/01	1650	850	8.33	40	35.4	67.1	9.00	117	11.6				
05/23/01	1965	450	8.50		22.6	22.4	3.30	62.1	8.4	<0.002	0.004	<0.002	<0.002
06/15/01	3009	450	8.79	70	18.1	17.5	1.99	56.2	7.8				
08/02/01	1675	390	8.37	100	21.7	22.4	2.54	50.3	9.6	<0.002	0.007	<0.002	<0.002
08/20/01	931	525	8.09	100	29.8	30.6	3.92	59.0	9.7				
09/12/01	238	500	7.96	100	32.8	33.9	4.31	57.8	10.6				
10/11/01	2195	375	7.58	75	33.2	33.6	4.73	47.9	8.5	<0.002	0.002	<0.002	0.003
10/30/01	824	375	7.72	65	31.0	30.8	3.91	48.9	7.5				
11/28/01	981	375	7.95	75	27.6	25.0	3.62	35.2	8.3				
4/28/02	938	250	7.63	40	19.6	20.7	2.51	30.7	4.74	<0.002	0.00657	0.00244	0.0333
6/4/02	765	320	8.71	70	23	28	3.42	36.4	6.28				
6/24/02	4265	280	8.66	60	19.7	18.5	2.11	30.1	6.21	<0.002	0.00304	<0.002	<0.002
7/8/02	1242	280	9.01	55	19.3	15.8	1.79	34.4	8.51				
7/29/02	1119	265	7.66	65	19.1	19.3	2.05	29.9	9.36				
8/13/02	2260	275	7.76	65	19.6	18.3	1.94	31.3	6.64	<0.002	0.00291	<0.002	<0.002
9/3/02	1597	245	7.61	70	21.5	18.5	1.92	28.6	7.38				
9/19/02	889	220	7.85	70	15.9	16.9	1.69	20.8	7.53	<0.002	0.00233	<0.002	0.00355
10/7/02	1196	230	7.8	55	21.4	20.4	2.18	22.8	5.9				
10/28/02	511	285	9.05	75	17.1	20.4	2.23	24.5	11.6				

Table A3.1. Drainage quality for the 0.02% sulfur field prediction pile 1.

Concentrations are in mg/L.

Date	Volume (L)	Fe	Mn	Al	Si	N	NH_3N	NO_3^2	TP
08/14/00	1202	0.079	<0.002	<0.002	5.45	<0.020	0.458	142	0.014
08/29/00	722	0.056	<0.002	<0.002	5.84	1.3	0.290	146	0.020
08/31/00									
09/12/00	1942	0.278	0.007	<0.002	4.30	3.4	1.93	528	0.020
10/11/00									
10/17/00	1226	0.271	0.033	<0.002	4.56	2.8	2.32	472	0.031
10/27/00									
11/07/00	1587	0.052	0.015	<0.002	4.32	1.1	0.220	184	0.029
11/20/00	350	0.070	0.017	<0.002	4.15				
04/09/01	2702	<0.002	<0.002	0.010	1.78	0.94	0.561	39.8	0.041
04/25/01	3219	<0.002	0.002	0.003					
05/10/01	1650	<0.002	<0.002	0.004		0.52	0.050	68.7	0.024
05/23/01	1965	<0.002	<0.002	0.002		0.48	0.024	30.9	<0.010
06/15/01	3009	<0.002	<0.002	0.007					
08/02/01	1675	<0.002	<0.002	0.006	5.35	0.43	0.030	24.6	0.020
08/20/01	931	0.026	<0.002	0.004		0.57	0.029	35.2	0.019
09/12/01	238	0.021	<0.002	0.007					
10/11/01	2195	0.014	<0.002	0.004	9.03	0.50	0.023	28.9	0.016
10/30/01	824	0.018	<0.002	0.003					
11/28/01	981	0.010	<0.002	<0.002		<0.20	<0.020	17.0	0.014
4/28/02	938	0.0312	0.00266	0.00616	2.73	0.56	0.067	12.9	0.026
6/4/02	765	0.0258	<0.002	0.00366					
6/24/02	4265	0.0158	<0.002	0.0167	3.78	0.29	0.02	13.6	0.073
7/8/02	1242	0.0177	<0.002	0.0139					
7/29/02	1119	0.0198	<0.002	0.00948					
8/13/02	2260	0.0161	<0.002	0.0129	5.81		0.055	10.3	0.03
9/3/02	1597	0.0182	<0.002	0.0051					
9/19/02	889	0.0163	<0.002	0.00795	5.04		<0.02	5.76	0.045
10/7/02	1196	0.028	0.002	0.00207					
10/28/02	511	0.016	<0.002	<0.002					

Table A3.2. Drainage quality for the 0.20% sulfur field prediction pile 2.

Concentrations are in mg/L, pH is in standard units, conductivity is in μS/cm, and net alkalinity is in mg/L as $CaCO_3$.

Date	Volume (L)	Conductivity	pH	Net Alk	SO_4	Ca	Mg	Na	K	Co	Cu	Ni	Zn
10/17/00	1576	2125	7.68	60	119	274	51.0	61.8	13.1	0.006	0.005	<0.002	<0.002
10/27/00		2900	7.52										
11/07/00	1739	3225	7.52	65	263	467	56.9	164	19.0	0.008	0.008	0.002	<0.002
11/20/00	493	4850	7.70	65	292	649	78.1	177	23.5				
04/09/01	1992	5000	7.42	70	**17.2**	655	85.5	183	25.3	0.013	0.020	0.014	<0.002
04/25/01	2982	1325	7.83	60	112	157	17.2	60.1	12.6				
05/10/01	1114	1350	8.40	65	121	165	17.2	76.7	16.7				
05/23/01	1680	825	8.05		70.1	86.3	8.47	52.3	13.2	0.002	0.006	<0.002	<0.002
06/15/01	2674	850	8.22	60	100	105	8.38	52.1	14.1				
08/02/01	1612	975	7.87	105	115	129	11.2	44.1	15.2	0.002	0.010	0.006	<0.002
08/20/01	1961	1000	7.72	105	124	128	11.6	46.4	14.4				
09/12/01	814	825	7.93	90	112	105	9.63	37.0	14.3				
10/11/01	2677	600	7.35	75	95.2	92.5	9.33	28.9	9.40	<0.002	0.004	<0.002	<0.002
10/30/01	851	550	7.78	75	106	87.0	7.95	25.7	9.21				
11/28/01	839	675	7.85	78	99.4	85.0	8.37	23.7	8.15				
4/28/02	839	300	7.66	60	58.8	47.4	4.23	12.2	5.4	<0.002	0.0062	0.0029	<0.002
6/4/02	661	430	8.53	60	89.5	61.3	6.19	16.3	7.5				
6/24/02	4301	500	8.09	60	73.1	61.4	5.33	19	8.28	<0.002	0.00505	<0.002	<0.002
7/8/02	1414	500	9.06	60	95.2	61.2	5.61	21.2	10.9				
7/29/02	1011	425	7.53	75	72.5	61.1	4.91	17.7	9.76				
8/13/02	2556	450	7.65	70	86	57.7	4.67	16.7	9.19	<0.002	0.00604	<0.002	0.00237
9/3/02	1584	340	7.51	75	64.3	48.9	3.8	13.5	8.58				
9/19/02	776	320	7.66	70	56.1	41.1	3.28	9.64	7.01	<0.002	0.00423	0.00223	<0.002
10/7/02	1449	375	7.66	75	74.9	54.2	4.44	11.7	6.77				
10/28/02	215	355	8.86	60	75.7	47.3	4.33	11.2	6.67				

Values that appear anomalous are in bold.

Table A3.2. Drainage quality for the 0.20% sulfur field prediction pile 2.

Concentrations are in mg/L.

Date	Volume (L)	Fe	Mn	Al	Si	N	NH_3N	NO_3^{2}	TP
10/17/00	1576	0.136	<0.002	<0.002	4.23	2.6	0.516	190	0.059
10/27/00									
11/07/00	1739	0.160	0.005	<0.002	5.08	3.4	1.10	284	0.067
11/20/00	493	0.316	0.011	<0.002	4.29				
04/09/01	1992	0.009	0.066	<0.002	5.00	4.7	2.88	486	0.112
04/25/01	2982	<0.002	0.002	<0.002					
05/10/01	1114	<0.002	<0.002	<0.002		2.7	0.935	88.7	0.041
05/23/01	1680	<0.002	<0.002	<0.002		2.0	0.574	48.2	0.019
06/15/01	2674	<0.002	0.004	<0.002					
08/02/01	1612	<0.002	0.006	<0.002	7.86	1.2	0.047	69.2	0.013
08/20/01	1961	0.142	<0.002	0.002		1.9	0.115	42.3	0.023
09/12/01	814	0.089	<0.002	<0.002					
10/11/01	2677	0.064	<0.002	<0.002	9.60	1.8	<0.020	37.1	0.015
10/30/01	851	0.071	<0.002	<0.002					
11/28/01	839	0.064	<0.002	<0.002		1.6	<0.020	28.5	<0.010
4/28/02	839	0.0347	<0.002	<0.002	5.35	0.9	0.086	13.1	<0.02
6/4/02	661	0.0572	0.0126	0.0022					
6/24/02	4301	0.0473	<0.002	0.00447	10.1	0.86	0.021	21.6	0.039
7/8/02	1414	0.0575	<0.002	0.00582					
7/29/02	1011	0.0582	<0.002	0.00459					
8/13/02	2556	0.046	<0.002	0.00411	14.2		0.027	15	<0.02
9/3/02	1584	0.0419	<0.002	<0.002					
9/19/02	776	0.0381	<0.002	0.00221	8.28		0.022	7.4	<0.02
10/7/02	1449	0.059	<0.002	<0.002					
10/28/02	215	0.041	<0.002	<0.002					

Table A3.3. Drainage quality for the 0.39% sulfur prediction pile 3.

Concentrations are in mg/L, pH is in standard units, conductivity is in µS/cm, and net alkalinity is in mg/L as $CaCO_3$.

Date	Volume (L)	Conductivity	pH	Net Alk	SO_4	Ca	Mg	Na	K	Co	Cu	Ni	Zn
10/17/00	1721	2225	7.61	60	77.4	242	54.8	89.1	17.4	0.012	0.006	<0.002	<0.002
10/27/00		3250	7.47										
11/07/00	1403	4300	7.36	45	191	474	76.9	233	32.4	0.021	0.011	0.003	0.004
11/20/00	536	5000	7.56	45	220	574	84.0	283	34.7				
04/09/01	2696	1850	7.31	50	**5.14**	197	26.2	86.2	16.9	0.008	0.008	0.005	<0.002
04/25/01	3143	950	7.80	45	47.0	102	12.8	62.3	14.2				
05/10/01	1050	1050	7.79	55	55.4	120	14.2	89.8	17.9				
05/23/01	1555	800	7.82		45.8	75.4	8.41	59.9	14.7	0.003	0.006	<0.002	<0.002
06/15/01	2565	750	7.86	50	68.1	85.9	8.69	44.0	14.7				
08/02/01	1177	625	7.81	100	105	76.2	7.95	29.6	14.0	0.002	0.008	<0.002	<0.002
08/20/01	778	700	7.98	100	113	83.0	9.38	31.5	14.2				
09/12/01	219	600	7.88	80	115	82.3	9.18	27.2	13.4				
10/11/01	1746	550	7.50	70	138	86.0	10.8	22.6	11.6	<0.002	0.003	<0.002	<0.002
10/30/01	879	600	7.74	70	175	99.6	11.4	21.6	11.3				
11/28/01	804	725	7.86	65	172	94.2	11.5	19.0	10.1				
4/28/02	903	400	7.63	40	107	60.7	5.98	12.3	6.15	<0.002	0.00346	<0.002	<0.002
6/4/02	566	500	7.94	60	126	70.4	7.32	14.8	8.33				
6/24/02	3593	440	7.71	40	106	56.2	5.45	12.4	8.36	<0.002	0.0039	<0.002	<0.002
7/8/02	1232	440	8.12	50	127	58.4	5.78	13	10.4				
7/29/02	957	425	7.53	55	116	62.6	5.79	11.9	10.4				
8/13/02	2080	490	7.56	50	150	64.6	6.02	11.2	9.64	<0.002	0.00466	<0.002	0.00242
9/3/02	1528	460	7.5	50	155	69.4	6.54	9.87	10.5				
9/19/02	538	500	7.64	50	142	60	5.42	7.25	13.4	<0.002	0.00247	<0.002	<0.002
10/7/02	1106	525	7.64	55	186	83.6	8.11	8.75	8.45				
10/28/02	539	525	8.21	50	185	82.4	7.99	8.86	8.18				

Values that appear anomalous are in bold.

Table A3.3. Drainage quality for the 0.39% sulfur prediction pile 3.

Concentrations are in mg/L.

Date	Volume (L)	Fe	Mn	Al	Si	N	NH_3N	NO_3^2	TP
10/17/00	1721	0.114	<0.002	<0.002	4.60	3.7	2.98	204	0.031
10/27/00									
11/07/00	1403	0.212	0.008	<0.002	4.88	7.0	5.92	447	0.036
11/20/00	536	0.317	0.012	<0.002	5.05				
04/10/01	2696	<0.002	0.012	0.003	2.18	2.5	3.66	199.9	0.044
04/25/01	3143	<0.002	<0.002	<0.002					
05/10/01	1050	<0.002	<0.002	0.002		3.2	2.00	85.3	0.022
05/23/01	1555	<0.002	<0.002	<0.002		1.9	1.01	57.9	<0.010
06/15/01	2565	<0.002	0.002	0.007					
08/02/01	1177	<0.002	<0.002	<0.002	5.78	0.87	0.037	40.7	<0.010
08/20/01	778	0.078	<0.002	<0.002		1.5	0.029	37.3	0.017
09/12/01	219	0.069	<0.002	0.007					
10/11/01	1746	0.057	<0.002	0.012	8.08	0.93	0.024	27.0	<0.010
10/30/01	879	0.080	<0.002	<0.002					
11/28/01	804	0.076	<0.002	<0.002		1.3	<0.020	24.9	0.014
4/28/02	903	0.0444	<0.002	<0.002	3.78	0.73	0.037	14.3	<0.02
6/4/02	566	0.07	<0.002	<0.002					
6/24/02	3593	0.0447	<0.002	0.00536	6.65	0.5	0.022	12	0.033
7/8/02	1232	0.056	<0.002	0.00786					
7/29/02	957	0.0574	<0.002	0.00515					
8/13/02	2080	0.0574	<0.002	0.00588	6.61		0.03	8.57	<0.02
9/3/02	1528	0.0635	<0.002	0.00324					
9/19/02	538	0.0588	<0.002	0.00379	5.54		<0.02	5.74	<0.02
10/7/02	1106	0.079	<0.002	<0.002					
10/28/02	539	0.076	<0.002	<0.002					

Table A3.4. Drainage quality for the 0.67% sulfur field prediction pile 4.

Concentrations are in mg/L, pH is in standard units, conductivity is in µS/cm, and net alkalinity is in mg/L as $CaCO_3$.

Date	Volume (L)	Conductivity	pH	Net Alk	SO_4	Ca	Mg	Na	K	Co	Cu	Ni	Zn
08/14/00	1164	550	8.74	185	79.5	48.0	39.2	12.3	2.42	<0.002	**0.126**	0.027	**0.126**
08/29/00		600	8.54	205									
09/12/00	2581	3200	7.51	45	197	408	57.4	145	28.1				
10/11/00		3750	7.84	80									
10/17/00	1187	6750	7.30	60	386	834	108	265	33.5	0.007	0.015	0.006	<0.002
10/27/00		5000	7.36										
11/07/00	1989	2700	7.37	48	332	357	31.3	136	29.9	0.003	0.008	0.003	0.004
11/20/00	664	2750	7.65	48	363	386	31.7	138	27.5				
04/12/01	2889	1075	7.59	55	179	147	12.0	44.6	12.0	<0.002	0.004	<0.002	<0.002
04/25/01	2637	825	7.78	40	142	110	9.04	49.6	8.80				
05/10/01	1213	775	7.85	55	142	98.6	8.99	40.3	12.1				
05/23/01	1974	600	7.99		112	70.7	5.64	32.3	12.1	<0.002	0.004	<0.002	<0.002
06/15/01	3290	500	8.06	60	107	67.8	4.66	22.4	11.9				
08/02/01	1483	500	7.82	95	123	67.7	5.03	15.7	12.7	<0.002	0.006	0.003	<0.002
08/20/01	904	600	8.36	85	173	83.2	6.65	16.4	13.3				
09/12/01	250	600	7.83	75	191	91.5	7.32	15.0	13.5				
10/11/01	2042	550	8.17	63	210	100	8.78	13.4	11.2	<0.002	<0.002	<0.002	<0.002
10/30/01	1016	675	7.66	60	276	128	10.3	12.5	11.5				
11/28/01	927	825	7.77	55	282	130	10.9	12.3	10.6				
4/28/02	982	430	7.52	40	164	71.4	5.43	7.4	5.61	<0.002	0.00338	0.00217	<0.002
6/4/02	571	650	7.81	50	254	115	8.51	10.7	9.04				
6/24/02	5048	550	7.57	40	198	85.7	5.87	8.96	8.74	<0.002	0.00275	<0.002	<0.002
7/8/02	1553	500	8.06	40	178	76	5.36	6.85	10.2				
7/29/02	1154	520	7.49	55	196	88.6	5.78	7.21	10.9				
8/13/02	2544	650	7.52	45	212	88.4	5.9	6.66	9.68	<0.002	0.00404	<0.002	<0.002
9/3/02	1755	650	7.44	45	268	115	7.52	6.52	11.3				
9/19/02	804	550	7.54	50	194	81.9	5.38	5.29	8.4	<0.002	<0.002	<0.002	<0.002
10/7/02	1247	675	7.54	50	300	124	8.25	5.78	8.97				
10/28/02	604	725	7.98	50	308	128	8.09	5.7	8.64				

Values that appear anomalous are in bold.

Table A3.4. Drainage quality for the 0.67% sulfur field prediction pile 4.

Concentrations are in mg/L.

Date	Volume (L)	Fe	Mn	Al	Si	N	NH_3N	NO_3^2	TP
08/14/00	1164	0.018	0.008	<0.002	5.96	<0.02	0.054	0.80	0.014
09/12/00	2581	0.165	0.004	<0.002	5.98	3.9	1.59	214	0.014
10/17/00	1187	0.368	0.030	<0.002	5.30	3.1	0.491	466	0.031
11/07/00	1989	0.132	0.021	0.013	4.18	2.5	0.103	183	0.029
11/20/00	664	0.172	0.019	<0.002	4.22				
04/11/01	2889	<0.002	0.014	<0.002	2.24	1.2	0.355	53.1	0.036
04/25/01	2637	<0.002	<0.002	<0.002					
05/10/01	1213	<0.002	<0.002	<0.002		0.58	0.047	30.8	0.018
05/23/01	1974	<0.002	<0.002	<0.002		0.65	0.027	24.4	<0.010
06/15/01	3290	<0.002	<0.002	0.004					
08/02/01	1483	<0.002	<0.002	<0.002		0.64	0.033	11.4	<0.010
08/20/01	904	0.076	<0.002	0.003		0.40	0.024	10.9	0.018
09/12/01	250	0.072	<0.002	<0.002					
10/11/01	2042	0.073	<0.002	<0.002	8.52	0.53	0.052	9.32	0.014
10/30/01	1016	0.108	<0.002	0.003					
11/28/01	927	0.099	<0.002	0.003		0.25	<0.020	9.72	0.013
4/28/02	982	0.0533	<0.002	<0.0025	2.93	0.33	0.06	5.58	<0.02
6/4/02	571	0.114	<0.002	<0.002					
6/24/02	5048	0.0679	<0.002	0.00467	5.55	0.21	0.023	6.6	0.025
7/8/02	1553	0.083	<0.002	0.0136					
7/29/02	1154	0.0826	<0.002	0.00515					
8/13/02	2544	0.0787	<0.002	0.00453	5.75		0.046	4.39	<0.02
9/3/02	1755	0.109	<0.002	0.00235					
9/19/02	804	0.078	<0.002	0.00308	5.38		<0.02	3.25	<0.02
10/7/02	1247	0.121	<0.002	<0.002					
10/28/02	604	0.099	<0.002	<0.002					

Figure A3.1. Drainage quality vs. time for the 0.02% S prediction field pile 1.

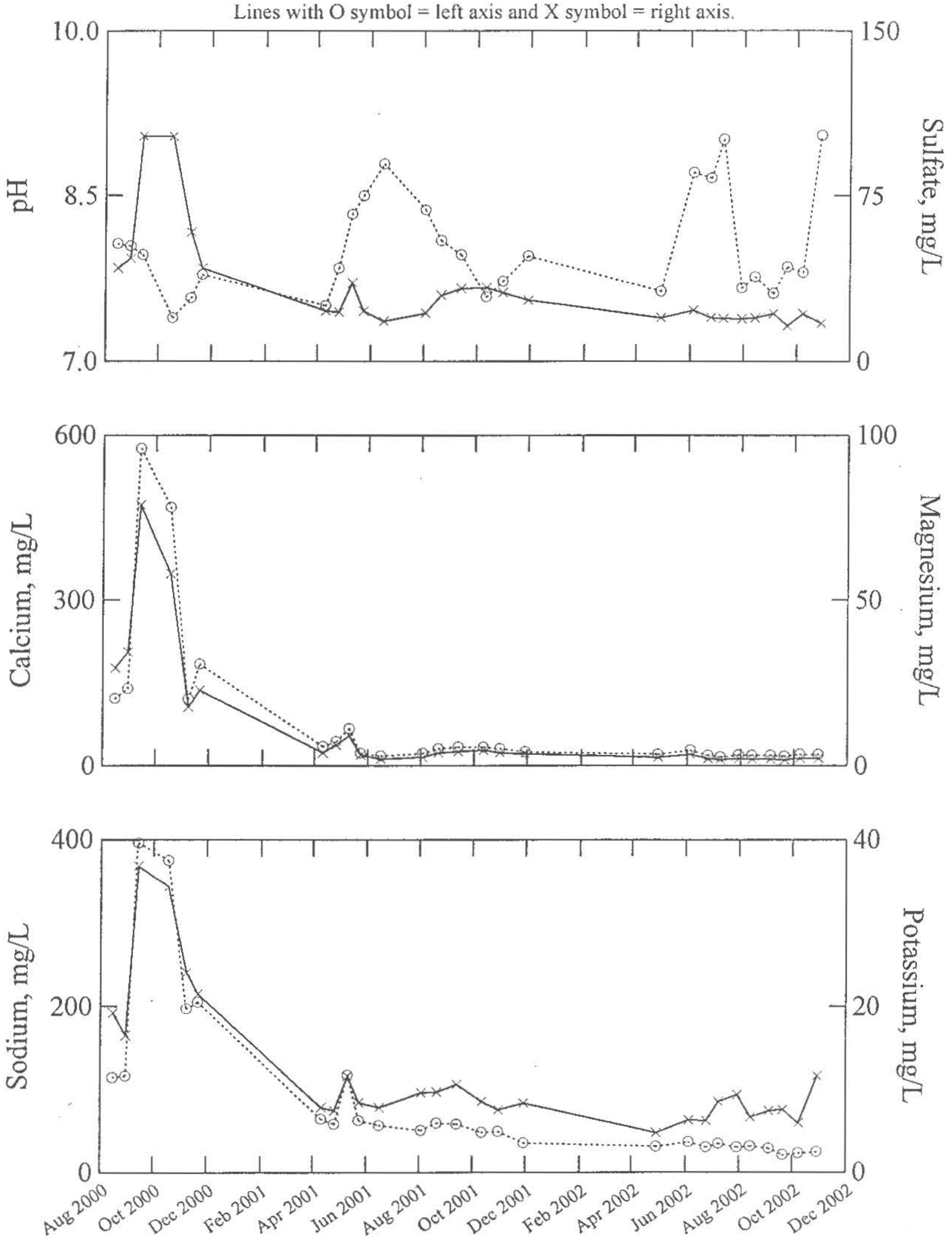


Figure A3.2. Drainage quality vs. time for the 0.20% S prediction field pile 2.

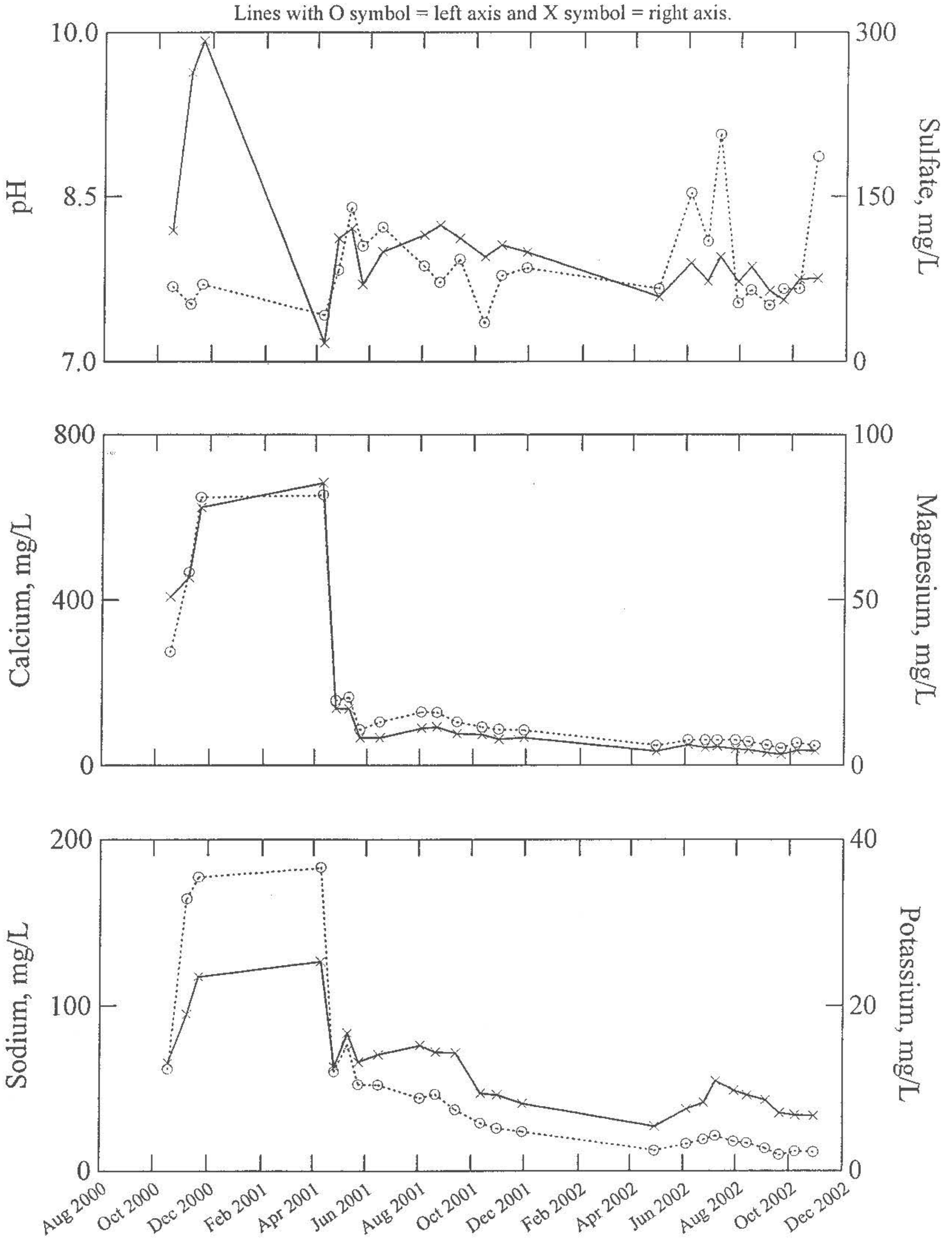


Figure A3.3. Drainage quality vs. time for the 0.39% S prediction field pile 3.

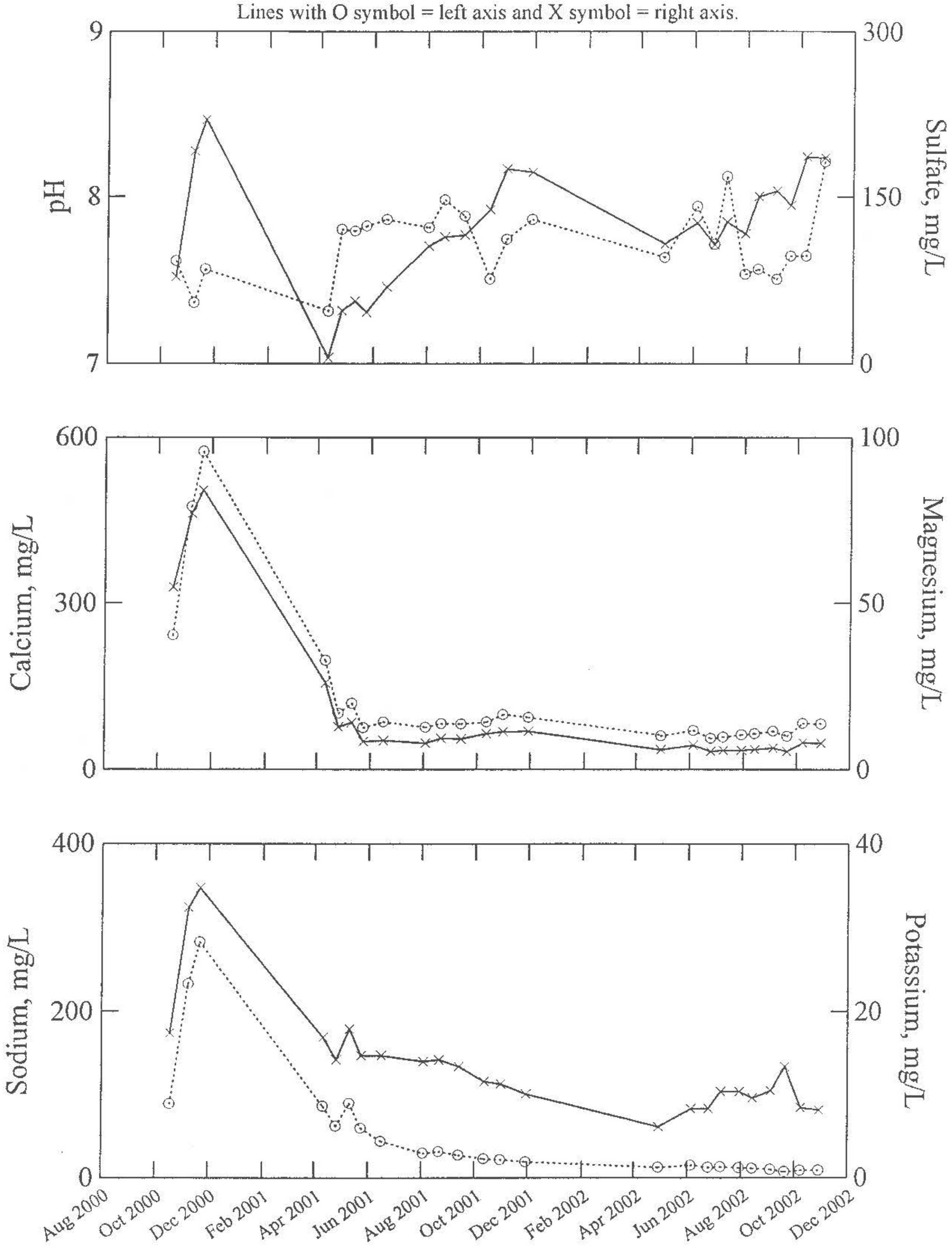
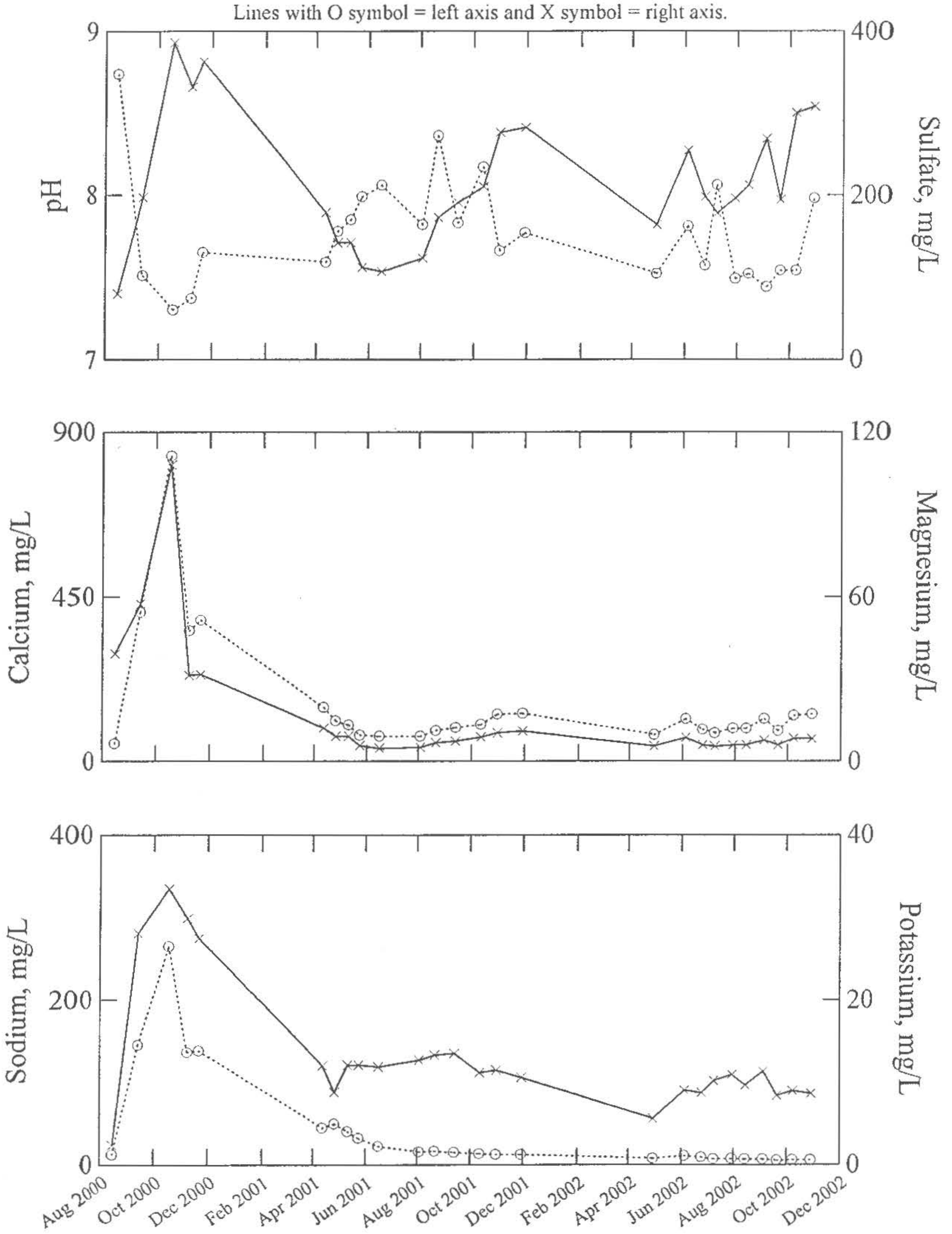


Figure A3.4. Drainage quality vs. time for the 0.67% S prediction field pile 4.



APPENDIX 4

CUMULATIVE SULFATE, CALCIUM, AND MAGNESIUM MASS RELEASE FROM FIELD PREDICTION PILES AND LIMESTONE ADDITION TANKS

Prediction Piles

Limestone Addition Tanks

Table A4.1. Cumulative sulfate, calcium, magnesium, sodium, and potassium mass release from the 0.02% sulfur field prediction pile 1.

Mass release recorded in moles, concentration in mg/L and volume in liters.

			Sulfate			Calcium			Magnesium	
Date	Volume	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass
08/14/00	1202	42.0	0.526	0.526	122	3.66	3.66	29.6	1.46	1.46
08/29/00	722	46.4	0.349	0.874	140	2.52	6.18	34.3	1.02	2.48
09/12/00	1942	102	2.06	2.94	576	27.9	34.1	78.7	6.29	8.77
10/17/00	1226	102	1.30	4.24	468	14.3	48.4	57.9	2.92	11.7
11/07/00	1587	58.3	0.963	5.20	121	4.79	53.2	17.8	1.16	12.9
11/20/00	350	42.0	0.153	5.35	184	1.61	54.8	22.8	0.328	13.2
04/10/01	2702	22.7	0.639	5.99	34.6	2.33	57.1	3.87	0.430	13.6
04/25/01	3219	22.2	0.744	6.74	43.3	3.48	60.6	6.30	0.835	14.4
05/10/01	1650	35.4	0.608	7.34	67.1	2.76	63.3	9.00	0.611	15.1
05/23/01	1965	22.6	0.462	7.81	22.4	1.10	64.4	3.30	0.267	15.3
06/15/01	3009	18.1	0.567	8.37	17.5	1.31	65.8	1.99	0.246	15.6
08/02/01	1675	21.7	0.378	8.75	22.4	0.936	66.7	2.54	0.175	15.7
08/20/01	931	29.8	0.289	9.04	30.6	0.710	67.4	3.92	0.150	15.9
09/12/01	238	32.8	0.081	9.12	33.9	0.201	67.6	4.31	0.042	15.9
10/11/01	2195	33.2	0.759	9.88	33.6	1.84	69.4	4.73	0.427	16.4
10/30/01	824	31.0	0.266	10.1	30.8	0.633	70.1	3.91	0.133	16.5
11/28/01	981	27.6	0.282	10.4	25.0	0.612	70.7	3.62	0.146	16.6
4/28/02	938	19.6	0.191	10.6	20.7	0.484	71.2	2.51	0.097	16.7
6/4/02	765	23	0.183	10.8	28.0	0.534	71.7	3.42	0.108	16.9
6/24/02	4265	19.7	0.875	11.7	18.5	1.968	73.7	2.11	0.370	17.2
7/8/02	1242	19.3	0.250	11.9	15.8	0.489	74.2	1.79	0.091	17.3
7/29/02	1119	19.1	0.222	12.2	19.3	0.539	74.7	2.05	0.094	17.4
8/13/02	2260	19.6	0.461	12.6	18.3	1.031	75.7	1.94	0.180	17.6
9/3/02	1597	21.5	0.357	13.0	18.5	0.737	76.5	1.92	0.126	17.7
9/19/02	889	15.9	0.147	13.1	16.9	0.375	76.8	1.69	0.062	17.8
10/7/02	1196	21.4	0.266	13.4	20.4	0.608	77.5	2.18	0.107	17.9
10/28/02	511	17.1	0.091	13.5	20.4	0.260	77.7	2.23	0.047	17.9

Note: Measured flow readings started on 3/22/01 and 4/14/02.

Table A4.1. Cumulative sulfate, calcium, magnesium, sodium, and potassium mass release from the 0.02% sulfur field prediction pile 1.

Mass release recorded in moles, concentration in mg/L and volume in liters.

		Sodium			Potassium		
Date	Volume	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass
08/14/00	1202	114	5.96	5.96	19.2	0.590	0.590
08/29/00	722	116	3.64	9.60	16.5	0.305	0.895
09/12/00	1942	396	33.5	43.1	36.8	1.83	2.72
10/17/00	1226	375	20.0	63.1	34.4	1.08	3.80
11/07/00	1587	197	13.6	76.7	24.1	0.978	4.78
11/20/00	350	205	3.12	79.8	21.5	0.192	4.97
04/10/01	2702	63.8	7.50	87.3	7.80	0.539	5.51
04/25/01	3219	57.6	8.07	95.3	7.40	0.609	6.12
05/10/01	1650	117	8.40	104	11.6	0.490	6.61
05/23/01	1965	62.1	5.31	109	8.40	0.422	7.03
06/15/01	3009	56.2	7.36	116	7.80	0.600	7.63
08/02/01	1675	50.3	3.66	120	9.60	0.411	8.04
08/20/01	931	59.0	2.39	122	9.70	0.231	8.27
09/12/01	238	57.8	0.598	123	10.6	0.065	8.34
10/11/01	2195	47.9	4.57	128	8.50	0.477	8.82
10/30/01	824	48.9	1.75	129	7.50	0.158	8.97
11/28/01	981	35.2	1.50	131	8.30	0.208	9.18
4/28/02	938	30.7	1.25	132	4.74	0.114	9.30
6/4/02	765	36.4	1.21	133	6.28	0.123	9.42
6/24/02	4265	30.1	5.58	139	6.21	0.677	10.1
7/8/02	1242	34.4	1.86	141	8.51	0.270	10.4
7/29/02	1119	29.9	1.46	142	9.36	0.268	10.6
8/13/02	2260	31.3	3.08	145	6.64	0.384	11.0
9/3/02	1597	28.6	1.99	147	7.38	0.301	11.3
9/19/02	889	20.8	0.80	148	7.53	0.171	11.5
10/7/02	1196	22.8	1.19	149	5.90	0.180	11.7
10/28/02	511	24.5	0.545	150	11.6	0.152	11.8

Note: Measured flow readings started on 3/22/01 and 4/14/02.

Table A4.2. Cumulative sulfate, calcium, magnesium, sodium, and potassium mass release from the 0.20% sulfur field prediction pile 2.

Mass release recorded in moles, concentration in mg/L, and volume in liters.

			Sulfate			Calcium			Magnesium	
Date	Volume	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass
10/17/00	1576	119	1.95	1.95	274	10.8	10.8	51.0	3.31	3.31
11/07/00	1739	263	4.76	6.71	467	20.3	31.0	56.9	4.07	7.38
11/20/00	493	292	1.50	8.21	649	7.98	39.0	78.1	1.58	8.96
4/9/2001*	1992	202	4.19	12.4	655	32.5	71.5	85.5	7.01	16.0
04/25/01	2982	112	3.48	15.9	157	11.7	83.2	17.2	2.11	18.1
05/10/01	1114	121	1.40	17.3	165	4.58	87.8	17.2	0.789	18.9
05/23/01	1680	70.1	1.23	18.5	86.3	3.62	91.4	8.47	0.586	19.5
06/15/01	2674	100	2.78	21.3	105	7.00	98.4	8.38	0.922	20.4
08/02/01	1612	115	1.93	23.2	129	5.19	104	11.2	0.743	21.1
08/20/01	1961	124	2.53	25.8	128	6.26	110	11.6	0.936	22.1
09/12/01	814	112	0.949	26.7	105	2.13	112	9.63	0.323	22.4
10/11/01	2677	95.2	2.65	29.4	92.5	6.18	118	9.33	1.03	23.4
10/30/01	851	106	0.939	30.3	87.0	1.85	120	7.95	0.278	23.7
11/28/01	839	99.4	0.868	31.2	85.0	1.78	122	8.37	0.289	24.0
4/28/02	839	58.8	0.514	31.7	47.4	0.99	123	4.23	0.146	24.1
6/4/02	661	89.5	0.616	32.3	61.3	1.01	124	6.19	0.168	24.3
6/24/02	4301	73.1	3.273	35.6	61.4	6.59	130	5.33	0.943	25.2
7/8/02	1414	95.2	1.401	37.0	61.2	2.16	133	5.61	0.326	25.6
7/29/02	1011	72.5	0.763	37.7	61.1	1.54	134	4.91	0.204	25.8
8/13/02	2556	86	2.288	40.0	57.7	3.68	138	4.67	0.491	26.3
9/3/02	1584	64.3	1.060	41.1	48.9	1.93	140	3.80	0.248	26.5
9/19/02	776	56.1	0.453	41.5	41.1	0.80	140	3.28	0.105	26.6
10/7/02	1449	74.9	1.130	42.7	54.2	1.96	142	4.44	0.265	26.9
10/28/02	215	75.7	0.169	42.8	47.3	0.25	143	4.33	0.038	26.9

Note: Starred (*) weeks concentrations for SO_4 were estimated by linear interpolation between the previous and subsequent.

Note: Measured flow readings started on 3/22/01 and 4/14/02.

Table A4.2. Cumulative sulfate, calcium, magnesium, sodium, and potassium mass release from the 0.20% sulfur field prediction pile 2.

Mass release recorded in moles, concentration in mg/L, and volume in liters.

			Sodium			Potassium	
Date	Volume	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass
10/17/00	1576	61.8	4.24	4.24	13.1	0.528	0.528
11/07/00	1739	164	12.4	16.6	19.0	0.845	1.37
11/20/00	493	177	3.80	20.4	23.5	0.296	1.67
4/9/2001*	1992	183	15.9	36.3	25.3	1.29	2.96
04/25/01	2982	60.1	7.80	44.1	12.6	0.961	3.92
05/10/01	1114	76.7	3.72	47.8	16.7	0.476	4.40
05/23/01	1680	52.3	3.82	51.6	13.2	0.567	4.96
06/15/01	2674	52.1	6.06	57.7	14.1	0.964	5.93
08/02/01	1612	44.1	3.09	60.8	15.2	0.627	6.55
08/20/01	1961	46.4	3.96	64.7	14.4	0.722	7.28
09/12/01	814	37.0	1.31	66.0	14.3	0.298	7.57
10/11/01	2677	28.9	3.37	69.4	9.40	0.644	8.22
10/30/01	851	25.7	0.951	70.4	9.21	0.200	8.42
11/28/01	839	23.7	0.865	71.2	8.15	0.175	8.59
4/28/02	839	12.2	0.445	71.7	5.40	0.116	8.71
6/4/02	661	16.3	0.469	72.1	7.50	0.127	8.83
6/24/02	4301	19.0	3.55	75.7	8.28	0.911	9.75
7/8/02	1414	21.2	1.30	77.0	10.9	0.394	10.1
7/29/02	1011	17.7	0.778	77.8	9.76	0.252	10.4
8/13/02	2556	16.7	1.86	79.6	9.19	0.601	11.0
9/3/02	1584	13.5	0.930	80.6	8.58	0.348	11.3
9/19/02	776	9.64	0.325	80.9	7.01	0.139	11.5
10/7/02	1449	11.7	0.737	81.6	6.77	0.251	11.7
10/28/02	215	11.2	0.105	81.7	6.67	0.037	11.8

Note: Starred (*) weeks concentrations for SO_4 were estimated by linear interpolation between the previous and subsequ

Note: Measured flow readings started on 3/22/01 and 4/14/02.

Table A4.4. Cumulative sulfate, calcium, magnesium, sodium, and potassium mass release from the 0.67% sulfur field prediction pile 4.

Mass release recorded in moles, concentration in mg/L, and volume in liters.

		Sulfate			Calcium			Magnesium		
Date	Volume	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass
08/14/00	1164	79.5	0.963	0.963	48.0	1.39	1.39	39.2	1.88	1.88
09/12/00	2581	197	5.29	6.26	408	26.3	27.7	57.4	6.10	7.97
10/17/00	1187	386	4.77	11.0	834	24.7	52.3	108	5.28	13.2
11/07/00	1989	332	6.87	17.9	357	17.7	70.0	31.3	2.56	15.8
11/20/00	664	363	2.51	20.4	386	6.4	76.4	31.7	0.866	16.7
04/12/00	2889	179	5.38	25.8	147	10.6	87.0	12.0	1.43	18.1
04/25/01	2637	142	3.90	29.7	110	7.23	94.3	9.04	0.981	19.1
05/10/01	1213	142	1.79	31.5	98.6	2.98	97.2	8.99	0.449	19.5
05/23/01	1974	112	2.30	33.8	70.7	3.48	101	5.64	0.458	20.0
06/15/01	3290	107	3.66	37.5	67.8	5.56	106	4.66	0.631	20.6
08/02/01	1483	123	1.90	39.3	67.7	2.50	109	5.03	0.307	20.9
08/20/01	904	173	1.63	41.0	83.2	1.88	111	6.65	0.247	21.2
09/12/01	250	191	0.497	41.5	91.5	0.570	111	7.32	0.075	21.3
10/11/01	2042	210	4.46	45.9	100	5.09	116	8.78	0.738	22.0
10/30/01	1016	276	2.92	48.9	128	3.24	120	10.3	0.431	22.4
11/28/01	927	282	2.72	51.6	130	3.01	123	10.9	0.416	22.8
4/28/02	982	164	1.68	53.3	71.4	1.75	124	5.4	0.219	23.1
6/4/02	571	254	1.51	54.8	115	1.64	126	8.5	0.200	23.3
6/24/02	5048	198	10.4	65.2	85.7	10.79	137	5.9	1.219	24.5
7/8/02	1553	178	2.88	68.0	76.0	2.94	140	5.4	0.343	24.8
7/29/02	1154	196	2.35	70.4	88.6	2.55	142	5.8	0.274	25.1
8/13/02	2544	212	5.61	76.0	88.4	5.61	148	5.9	0.618	25.7
9/3/02	1755	268	4.90	80.9	115	5.03	153	7.5	0.543	26.3
9/19/02	804	194	1.62	82.5	81.9	1.64	155	5.4	0.178	26.4
10/7/02	1247	300	3.89	86.4	124	3.86	158	8.3	0.423	26.9
10/28/02	604	308	1.94	88.4	128	1.93	160	8.1	0.201	27.1

Note: Measured flow readings started on 3/22/01 and 4/14/02.

Table A4.3. Cumulative sulfate, calcium, magnesium, sodium, and potassium mass release from the 0.39% sulfur prediction pile 3.

Mass release recorded in moles, concentration in mg/L, and volume in liters.

		Sodium			Potassium		
Date	Volume	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass
10/17/00	1721	89.1	6.67	6.67	17.4	0.766	0.766
11/07/00	1403	233	14.2	20.9	32.4	1.16	1.93
11/20/00	536	283	6.60	27.5	34.7	0.476	2.40
4/10/2001*	2696	86.2	10.1	37.6	16.9	1.17	3.57
04/25/01	3143	62.3	8.52	46.1	14.2	1.14	4.71
05/10/01	1050	89.8	4.10	50.2	17.9	0.481	5.19
05/23/01	1555	59.9	4.05	54.3	14.7	0.585	5.78
06/15/01	2565	44.0	4.91	59.2	14.7	0.964	6.74
08/02/01	1177	29.6	1.52	60.7	14.0	0.421	7.16
08/20/01	778	31.5	1.07	61.8	14.2	0.283	7.44
09/12/01	219	27.2	0.259	62.0	13.4	0.075	7.52
10/11/01	1746	22.6	1.72	63.7	11.6	0.518	8.04
10/30/01	879	21.6	0.826	64.6	11.3	0.254	8.29
11/28/01	804	19.0	0.664	65.2	10.1	0.208	8.50
4/28/02	903	12.3	0.483	65.7	6.15	0.142	8.64
6/4/02	566	14.8	0.364	66.1	8.33	0.121	8.76
6/24/02	3593	12.4	1.94	68.0	8.36	0.768	9.53
7/8/02	1232	13.0	0.697	68.7	10.4	0.328	9.86
7/29/02	957	11.9	0.495	69.2	10.4	0.255	10.1
8/13/02	2080	11.2	1.01	70.2	9.64	0.513	10.6
9/3/02	1528	9.87	0.656	70.9	10.5	0.410	11.0
9/19/02	538	7.25	0.170	71.0	13.4	0.184	11.2
10/7/02	1106	8.75	0.421	71.5	8.45	0.239	11.5
10/28/02	539	8.86	0.208	71.7	8.18	0.113	11.6

Note: Starred (*) weeks concentrations for SO_4 were estimated by linear interpolation between the previous and subsequ

Note: Measured flow readings started on 3/22/01 and 4/14/02.

Table A4.3. Cumulative sulfate, calcium, magnesium, sodium, and potassium mass release from the 0.39% sulfur prediction pile 3.

Mass release recorded in moles, concentration in mg/L, and volume in liters.

		Sodium			Potassium		
Date	Volume	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass
10/17/00	1721	89.1	6.67	6.67	17.4	0.766	0.766
11/07/00	1403	233	14.2	20.9	32.4	1.16	1.93
11/20/00	536	283	6.60	27.5	34.7	0.476	2.40
4/10/2001*	2696	86.2	10.1	37.6	16.9	1.17	3.57
04/25/01	3143	62.3	8.52	46.1	14.2	1.14	4.71
05/10/01	1050	89.8	4.10	50.2	17.9	0.481	5.19
05/23/01	1555	59.9	4.05	54.3	14.7	0.585	5.78
06/15/01	2565	44.0	4.91	59.2	14.7	0.964	6.74
08/02/01	1177	29.6	1.52	60.7	14.0	0.421	7.16
08/20/01	778	31.5	1.07	61.8	14.2	0.283	7.44
09/12/01	219	27.2	0.259	62.0	13.4	0.075	7.52
10/11/01	1746	22.6	1.72	63.7	11.6	0.518	8.04
10/30/01	879	21.6	0.826	64.6	11.3	0.254	8.29
11/28/01	804	19.0	0.664	65.2	10.1	0.208	8.50
4/28/02	903	12.3	0.483	65.7	6.15	0.142	8.64
6/4/02	566	14.8	0.364	66.1	8.33	0.121	8.76
6/24/02	3593	12.4	1.94	68.0	8.36	0.768	9.53
7/8/02	1232	13.0	0.697	68.7	10.4	0.328	9.86
7/29/02	957	11.9	0.495	69.2	10.4	0.255	10.1
8/13/02	2080	11.2	1.01	70.2	9.64	0.513	10.6
9/3/02	1528	9.87	0.656	70.9	10.5	0.410	11.0
9/19/02	538	7.25	0.170	71.0	13.4	0.184	11.2
10/7/02	1106	8.75	0.421	71.5	8.45	0.239	11.5
10/28/02	539	8.86	0.208	71.7	8.18	0.113	11.6

Note: Starred (*) weeks concentrations for SO_4 were estimated by linear interpolation between the previous and subsequ

Note: Measured flow readings started on 3/22/01 and 4/14/02.

Table A4.4. Cumulative sulfate, calcium, magnesium, sodium, and potassium mass release from the 0.67% sulfur field prediction pile 4.

Mass release recorded in moles, concentration in mg/L, and volume in liters.

			Sodium			Potassium	
Date	Volume	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass
08/14/00	1164	12.3	0.623	0.623	2.42	0.072	0.072
09/12/00	2581	145	16.3	16.9	28.1	1.85	1.93
10/17/00	1187	265	13.7	30.6	33.5	1.017	2.94
11/07/00	1989	136	11.8	42.3	29.9	1.52	4.46
11/20/00	664	138	3.99	46.3	27.5	0.467	4.93
04/12/00	2889	44.6	5.60	51.9	12.0	0.887	5.82
04/25/01	2637	49.6	5.69	57.6	8.80	0.593	6.41
05/10/01	1213	40.3	2.13	59.8	12.1	0.375	6.79
05/23/01	1974	32.3	2.77	62.5	12.1	0.611	7.40
06/15/01	3290	22.4	3.21	65.7	11.9	1.001	8.40
08/02/01	1483	15.7	1.01	66.7	12.7	0.482	8.88
08/20/01	904	16.4	0.645	67.4	13.3	0.307	9.19
09/12/01	250	15.0	0.163	67.6	13.5	0.086	9.28
10/11/01	2042	13.4	1.19	68.7	11.2	0.585	9.86
10/30/01	1016	12.5	0.552	69.3	11.5	0.299	10.2
11/28/01	927	12.3	0.496	69.8	10.6	0.251	10.4
4/28/02	982	7.40	0.316	70.1	5.61	0.141	10.6
6/4/02	571	10.7	0.266	70.4	9.04	0.132	10.7
6/24/02	5048	8.96	1.97	72.3	8.74	1.128	11.8
7/8/02	1553	6.85	0.463	72.8	10.2	0.405	12.2
7/29/02	1154	7.21	0.362	73.2	10.9	0.322	12.5
8/13/02	2544	6.66	0.737	73.9	9.68	0.630	13.2
9/3/02	1755	6.52	0.498	74.4	11.3	0.507	13.7
9/19/02	804	5.29	0.185	74.6	8.40	0.173	13.8
10/7/02	1247	5.78	0.314	74.9	8.97	0.286	14.1
10/28/02	604	5.70	0.150	75.1	8.64	0.133	14.3

Note: Measured flow readings started on 3/22/01 and 4/14/02.

Figure A4.1. Cumulative sulfate, calcium, magnesium, sodium,and potassium mass release for 0.02% sulfur field prediction pile 1.

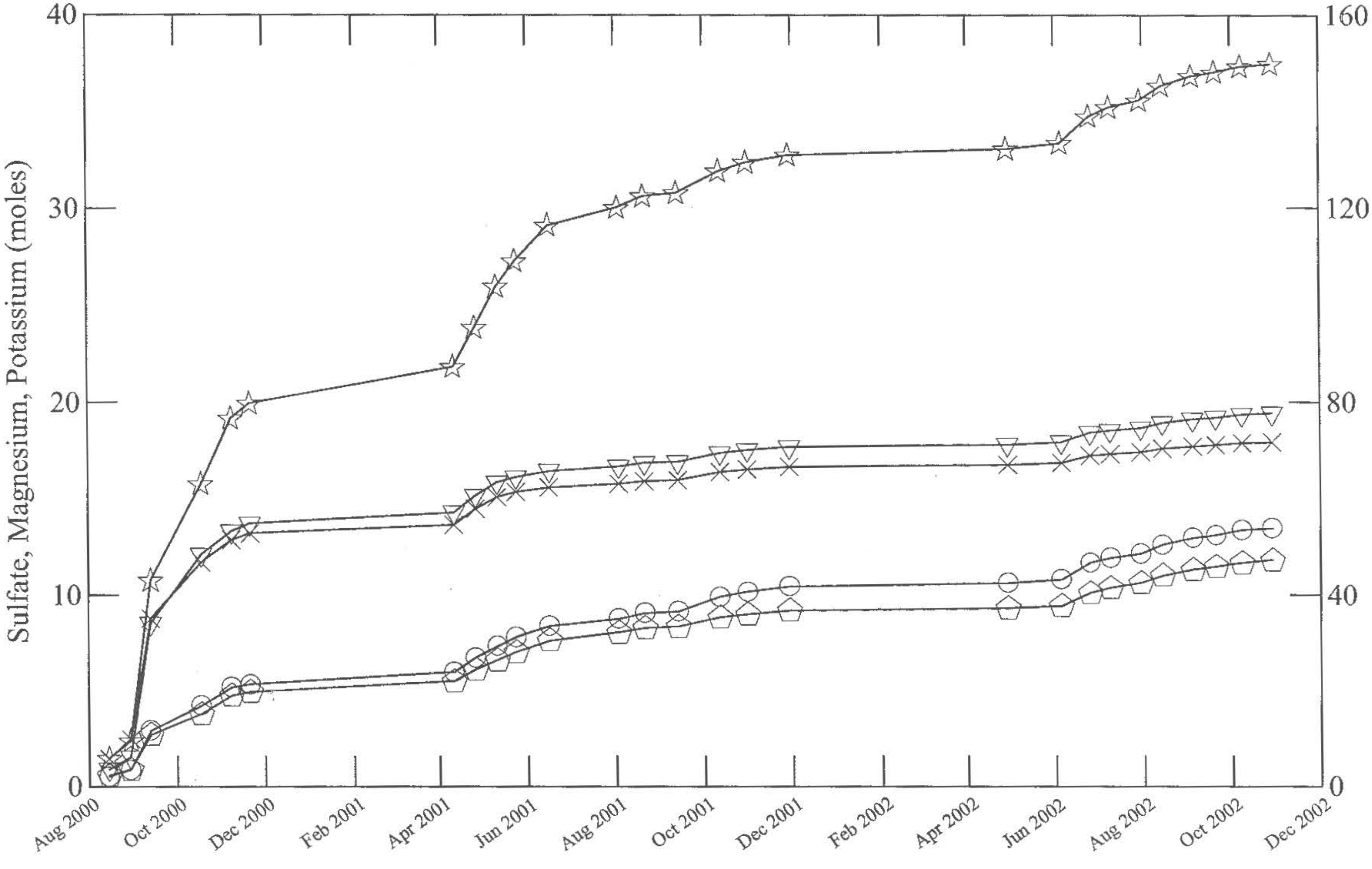


Figure A4.2. Cumulative sulfate, calcium, magnesium, sodium, and potassium mass release for 0.20% sulfur field prediction pile 2.

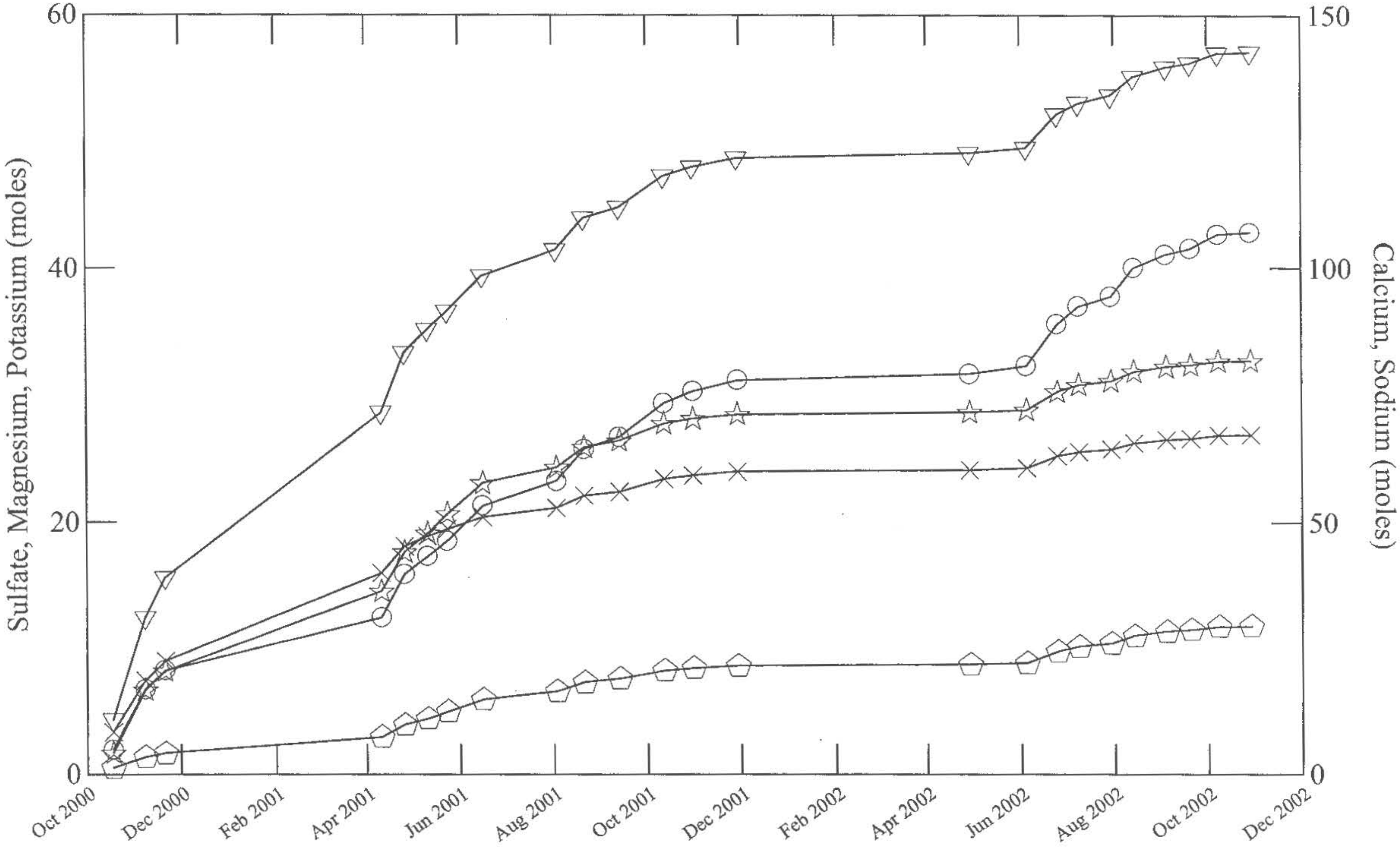


Figure A4.3. Cumulative sulfate, calcium,magnesium, sodium, and potassium mass release for 0.39% sulfur field prediction pile 3.

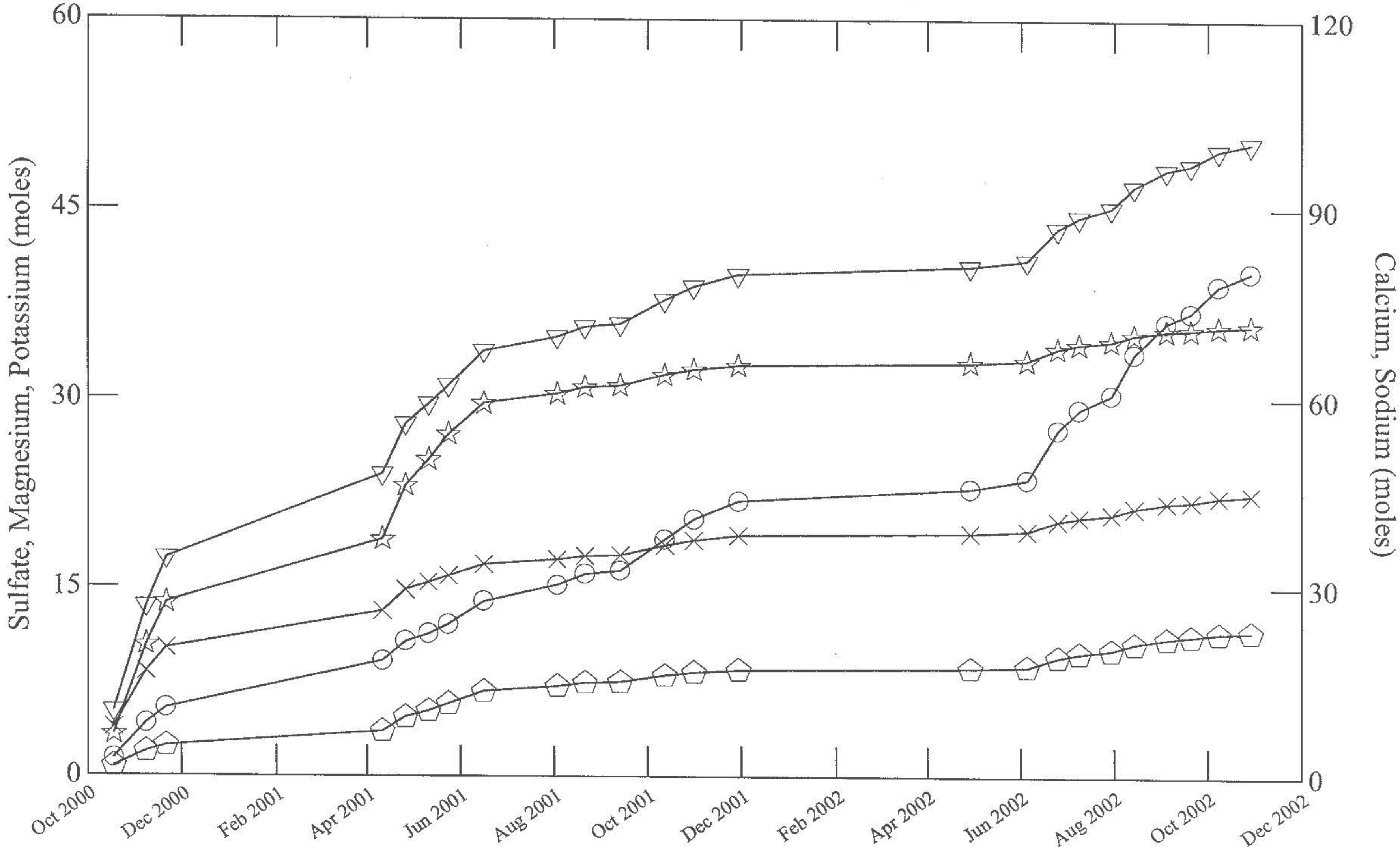


Figure A4.4. Cumulative sulfate, calcium, magnesium, sodium, and potassium mass release for 0.67% sulfur field prediction pile 4.

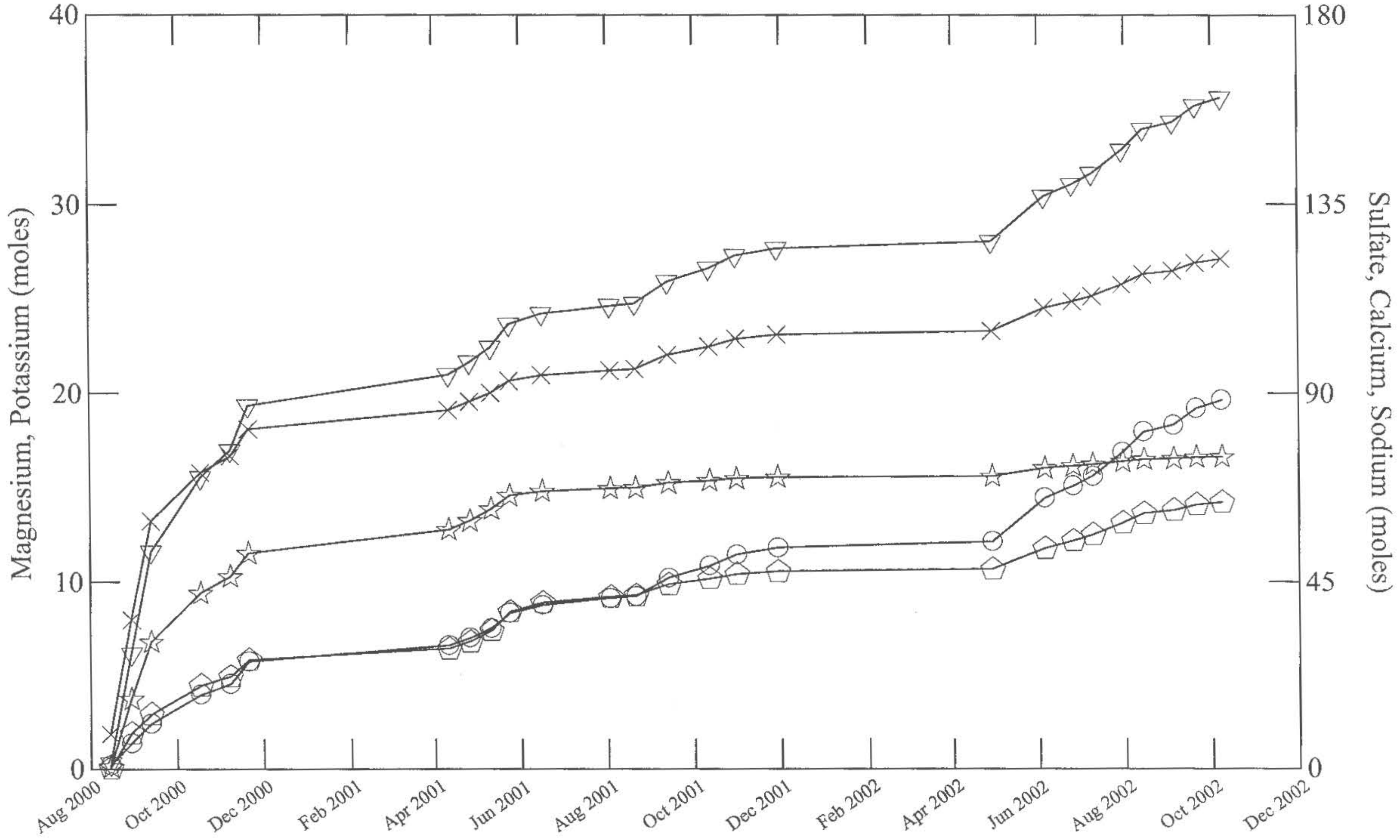


Table A4.5. Cumulative sulfate, calcium and magnesium mass release from the limestone addition control field tank #1.

Mass release recorded in moles, concentration in mg/L, and volume in liters.

			Sulfate			Calcium			Magnesium	
Date	Volume	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass
11/07/00	35	683	0.249	0.249	926	0.808	0.808	104	0.150	0.150
03/22/01	60	253	0.159	0.408	413	0.621	1.43	46.4	0.115	0.265
04/09/01	34	260	0.091	0.498	206	0.172	1.60	21.6	0.030	0.295
04/25/01	121	108	0.136	0.634	138	0.417	2.02	13.4	0.067	0.361
05/10/01	25	210	0.055	0.690	190	0.120	2.14	20.0	0.021	0.382
05/23/01	58	180	0.109	0.799	138	0.201	2.34	13.0	0.031	0.413
06/15/01	92	258	0.247	1.05	120	0.275	2.61	10.9	0.041	0.455
08/02/01	40	256	0.108	1.15	136	0.137	2.75	11.4	0.019	0.474
08/20/01	28	355	0.103	1.26	149	0.103	2.85	12.3	0.014	0.488
09/12/01	9	522	0.047	1.30	233	0.050	2.90	20.3	0.007	0.495
09/28/01	24	398	0.099	1.40	175	0.104	3.01	15.6	0.015	0.510
10/11/01	49	398	0.203	1.61	163	0.199	3.21	15.0	0.030	0.541
10/30/01	31	433	0.140	1.75	178	0.138	3.35	16.5	0.021	0.562
11/26/01	64	428	0.287	2.03	175	0.281	3.63	16.7	0.044	0.606
4/10/02	51	164	0.087	2.12	67.4	0.086	3.71	6.0	0.013	0.618
5/10/02	24	420	0.105	2.22	176	0.105	3.82	17.0	0.017	0.635
6/4/02	7	601	0.044	2.27	216	0.038	3.86	21.6	0.006	0.641
6/24/02	194.2	265	0.536	2.80	113	0.547	4.40	10.8	0.086	0.728
7/8/02	42	328	0.143	2.95	132	0.138	4.54	13.0	0.022	0.750
7/29/02	29.8	295	0.092	3.04	120	0.089	4.63	13.4	0.016	0.767
8/12/02	72.8	391	0.296	3.34	158	0.287	4.92	17.2	0.052	0.818
9/19/02	88.5	417	0.384	3.72	166	0.366	5.28	18.8	0.068	0.887
10/28/02	55.8	372	0.216	3.94	142	0.198	5.48	16.6	0.038	0.925

6/24/02 flow volume is an estimate based on the flow and precipitation for the peroid 4/10/02 to 10/28/02.

Table A4.6. Cumulative sulfate, calcium and magnesium mass release from the limestone addition control field tank #6.

Mass release recorded in moles, concentration in mg/L, and volume in liters.

		Sulfate			Calcium			Magnesium		
Date	Volume	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass
11/07/00	32	374	0.126	0.126	689	0.558	0.558	82.1	0.110	0.110
03/22/01	34	311	0.112	0.238	418	0.359	0.917	49.1	0.070	0.179
04/09/01	38	262	0.104	0.341	260	0.246	1.16	29.0	0.045	0.225
04/25/01	110	91	0.104	0.446	81	0.224	1.39	8.85	0.040	0.265
05/10/01	15	172	0.027	0.473	109	0.041	1.43	10.8	0.007	0.271
05/23/01	58	188	0.114	0.587	110	0.160	1.59	10.9	0.026	0.298
06/15/01	76	218	0.173	0.760	107	0.203	1.79	9.82	0.031	0.328
08/02/01	32	219	0.072	0.832	119	0.094	1.89	10.9	0.014	0.343
08/20/01	26	381	0.105	0.937	164	0.108	1.99	14.6	0.016	0.359
09/12/01	8	564	0.047	0.984	236	0.047	2.04	21.9	0.007	0.366
09/28/01	23	405	0.098	1.08	160	0.092	2.13	15.6	0.015	0.381
10/11/01	50	353	0.183	1.26	138	0.171	2.30	13.6	0.028	0.408
10/30/01	25	345	0.090	1.35	131	0.082	2.39	13.6	0.014	0.423
11/26/01	60.4	384	0.241	1.60	151	0.227	2.61	15.8	0.039	0.462
4/10/02	59	256	0.157	1.75	100	0.147	2.76	10.9	0.026	0.488
5/10/02	30	377	0.118	1.87	154	0.115	2.88	15.7	0.019	0.508
6/4/02	7.3	437	0.033	1.90	176	0.032	2.91	18.6	0.006	0.513
6/24/02	202.5	308	0.649	2.55	119	0.601	3.51	11.9	0.099	0.612
7/8/02	42.9	309	0.138	2.69	121	0.129	3.64	12.3	0.022	0.634
7/29/02	30.5	328	0.104	2.80	127	0.097	3.73	14.7	0.018	0.653
8/12/02	80.1	420	0.350	3.15	164	0.328	4.06	20.3	0.067	0.719
9/19/02	84	432	0.378	3.52	180	0.377	4.44	19.1	0.066	0.785
10/28/02	53	316	0.174	3.70	122	0.161	4.60	15.7	0.034	0.820

6/24/02 flow volume is an estimate based on the flow and precipitation for the peroid 4/10/02 to 10/28/02.

Table A4.7. Cumulative sulfate, calcium and magnesium mass release from the limestone addition 1:1 ratio field tank #2.

Mass release recorded in moles, concentration in mg/L, and volume in liters.

		Sulfate			Calcium			Magnesium		
Date	Volume	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass
11/7/00	36	496	0.186	0.186	708	0.636	0.636	84.5	0.125	0.125
3/22/01	30	519	0.162	0.348	486	0.364	1.00	54	0.067	0.192
4/9/01	35	184	0.067	0.415	163	0.142	1.14	17.9	0.026	0.218
4/25/01	114	85	0.101	0.516	95.6	0.272	1.41	10.6	0.050	0.267
5/10/01	21	118	0.026	0.542	122	0.064	1.48	13.4	0.012	0.279
5/23/01	56	163	0.095	0.637	108	0.151	1.63	10.9	0.025	0.304
6/15/01	83	180	0.156	0.792	99.3	0.206	1.83	9.91	0.034	0.338
8/2/01	46	230	0.110	0.902	118	0.135	1.97	12	0.023	0.361
8/20/01	26	333	0.090	0.993	144	0.093	2.06	14.9	0.016	0.377
9/12/01	5	434	0.023	1.02	201	0.025	2.09	21	0.004	0.381
9/28/01	22	376	0.086	1.10	161	0.088	2.18	17.7	0.016	0.397
10/11/01	51	275	0.146	1.25	125	0.159	2.33	14	0.029	0.426
10/30/01	28	222	0.065	1.31	114	0.080	2.41	12.6	0.015	0.441
11/26/01	41	305	0.130	1.44	132	0.135	2.55	16	0.027	0.468
4/10/02	61	152	0.097	1.54	70.4	0.107	2.66	7.81	0.020	0.487
5/10/02	21.2	315	0.070	1.61	140	0.074	2.73	17.6	0.015	0.503
6/4/02	4.6	493	0.024	1.63	180	0.021	2.75	22.9	0.004	0.507
6/24/02	196.2	180	0.368	2.00	87.1	0.426	3.18	9.85	0.080	0.587
7/8/02	43	220	0.098	2.10	102	0.109	3.29	12.8	0.023	0.609
7/29/02	30.5	196	0.062	2.16	84.8	0.064	3.35	12.5	0.016	0.625
8/12/02	75.2	244	0.191	2.35	113	0.212	3.56	16.8	0.052	0.677
9/19/02	89.6	272	0.254	2.60	124	0.277	3.84	16.8	0.062	0.739
10/28/02	49.8	208	0.108	2.71	88.3	0.110	3.95	13	0.027	0.766

6/24/02 flow volume is an estimate based on the flow and precipitation for the peroid 4/10/02 to 10/28/02.

Table A4.8. Cumulative sulfate, calcium and magnesium mass release from the limestone addition 1:1 ratio field tank #5.

Mass release recorded in moles, concentration in mg/L, and volume in liters

			Sulfate			Calcium			Magnesium	
Date	Volume	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass
11/07/00	34	694	0.249	0.249	1090	0.936	0.936	144	0.204	0.204
03/22/01	25	294	0.077	0.326	360	0.226	1.16	40.7	0.042	0.246
04/09/01	23	306	0.072	0.398	268	0.151	1.31	30.7	0.029	0.275
04/25/01	109	85	0.097	0.495	94	0.257	1.57	10.2	0.046	0.321
05/10/01	18	151	0.028	0.523	153	0.069	1.64	16.6	0.012	0.333
05/23/01	58	231	0.139	0.662	122	0.175	1.81	12.2	0.029	0.362
06/15/01	87	238	0.215	0.877	122	0.264	2.08	11.3	0.040	0.402
08/02/01	48	310	0.154	1.03	169	0.201	2.28	15.8	0.031	0.433
08/20/01	26	434	0.117	1.15	213	0.137	2.42	18.7	0.020	0.453
09/12/01	6	720	0.045	1.19	290	0.043	2.46	28.2	0.007	0.460
09/28/01	22	519	0.118	1.31	217	0.118	2.58	20.9	0.019	0.479
10/11/01	50	406	0.213	1.52	169	0.212	2.79	16.2	0.034	0.512
10/30/01	32	381	0.127	1.65	154	0.123	2.91	15.7	0.021	0.533
11/26/01	50.13	394	0.206	1.86	163	0.204	3.12	16.7	0.034	0.568
4/10/02	46	222	0.106	1.96	97.5	0.112	3.23	10.1	0.019	0.587
5/10/02	20	378	0.079	2.04	164	0.082	3.31	16.5	0.014	0.600
6/4/02	7.3	483	0.037	2.08	191	0.035	3.35	19.9	0.006	0.606
6/24/02	190	256	0.506	2.58	116	0.550	3.90	11.4	0.089	0.695
7/8/02	42.1	250	0.110	2.69	110	0.115	4.01	11.6	0.020	0.716
7/29/02	29.8	287	0.089	2.78	129	0.096	4.11	14.8	0.018	0.734
8/12/02	72.7	346	0.262	3.04	147	0.267	4.37	17.8	0.053	0.787
9/19/02	88.4	384	0.353	3.40	163	0.359	4.73	17.7	0.064	0.851
10/28/02	56.6	252	0.148	3.55	106	0.150	4.88	13.0	0.030	0.882

6/24/02 flow volume is an estimate based on the flow and precipitation for the peroid 4/10/02 to 10/28/02.

Table A4.9. Cumulative sulfate, calcium and magnesium mass release from the limestone addition 3:1 ratio field tank #3.

Mass release recorded in moles, concentration in mg/L, and volume in liters.

			Sulfate			Calcium			Magnesium	
Date	Volume	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass
11/07/00	39	733	0.296	0.296	925	0.894	0.894	112.0	0.179	0.179
03/22/01	64	366	0.242	0.538	436	0.691	1.58	54.4	0.142	0.321
04/09/01	34	165	0.058	0.596	210	0.178	1.76	25.7	0.036	0.357
04/25/01	113	103	0.121	0.718	97.3	0.275	2.04	11.4	0.053	0.410
05/10/01	13	168	0.023	0.741	120	0.040	2.08	14.3	0.008	0.418
05/23/01	58	192	0.116	0.857	110	0.160	2.24	12.4	0.030	0.448
06/15/01	86	224	0.201	1.06	119	0.256	2.49	12.3	0.044	0.491
08/02/01	46	304	0.147	1.20	139	0.161	2.65	16.0	0.031	0.522
08/20/01	26	456	0.126	1.33	207	0.137	2.79	22.7	0.025	0.546
09/12/01	6	704	0.044	1.37	296	0.044	2.83	34.0	0.008	0.555
09/28/01	23	475	0.115	1.49	200	0.116	2.95	24.6	0.023	0.578
10/11/01	49	395	0.202	1.69	167	0.204	3.15	20.5	0.041	0.620
10/30/01	30	397	0.126	1.82	162	0.123	3.28	21.4	0.027	0.646
11/26/01	50.99	394	0.209	2.03	166	0.211	3.49	21.5	0.045	0.691
4/10/02	65	128	0.087	2.11	60.6	0.098	3.59	7.4	0.020	0.711
5/10/02	23	411	0.098	2.21	176	0.101	3.69	24.1	0.023	0.734
6/4/02	4.6	620	0.030	2.24	240	0.028	3.71	33.6	0.006	0.740
6/24/02	186.9	245	0.477	2.72	113	0.527	4.24	14.2	0.109	0.850
7/8/02	38.7	244	0.098	2.82	114	0.110	4.35	15.2	0.024	0.874
7/29/02	29.8	242	0.075	2.89	110	0.082	4.43	16.4	0.020	0.894
8/12/02	50.2	302	0.158	3.05	135	0.169	4.60	20.2	0.042	0.936
9/19/02	88.7	339	0.313	3.36	147	0.325	4.93	20.5	0.075	1.01
10/28/02	57	255	0.151	3.51	111	0.158	5.09	16.7	0.039	1.05

6/24/02 flow volume is an estimate based on the flow and precipitation for the peroid 4/10/02 to 10/28/02.

Table A4.10. Cumulative sulfate, calcium and magnesium mass release from the limestone addition 3:1 ratio field tank #4.

Mass release recorded in moles, concentration in mg/L, and volume in liters.

			Sulfate			Calcium			Magnesium	
Date	Volume	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass	Conc.	Mass	Sum Mass
11/07/00	36	673	0.251	0.251	785	0.700	0.700	99.1	0.146	0.146
03/22/01	48	386	0.192	0.442	396	0.471	1.17	48.5	0.095	0.241
04/09/01	34	191	0.068	0.511	206	0.177	1.35	24.9	0.035	0.276
04/25/01	111	85.6	0.099	0.610	99.6	0.276	1.62	11.9	0.054	0.331
05/10/01	15	169	0.026	0.635	140	0.051	1.67	16.7	0.010	0.341
05/23/01	63	162	0.106	0.741	106	0.166	1.84	12.5	0.032	0.373
06/15/01	83	189	0.163	0.904	105	0.217	2.06	11.1	0.038	0.411
08/02/01	34	222	0.080	0.984	113	0.097	2.15	12.6	0.018	0.429
08/20/01	28	363	0.105	1.09	160	0.111	2.27	17.9	0.020	0.449
09/12/01	8	545	0.045	1.13	230	0.046	2.31	26.6	0.009	0.458
09/28/01	23	387	0.091	1.22	157	0.088	2.40	19.7	0.018	0.476
10/11/01	48	327	0.165	1.39	132	0.159	2.56	16.6	0.033	0.509
10/30/01	31	364	0.117	1.51	143	0.110	2.67	18.6	0.024	0.533
11/26/01	43.97	374	0.171	1.68	150	0.164	2.83	19.5	0.035	0.568
4/10/02	62	185	0.119	1.80	86.3	0.133	2.97	11.2	0.029	0.597
5/10/02	15	341	0.053	1.85	148	0.055	3.02	19.1	0.012	0.608
6/4/02	7.9	483	0.040	1.89	179	0.035	3.06	24.1	0.008	0.616
6/24/02	189.8	216	0.427	2.32	95.3	0.451	3.51	12.2	0.095	0.712
7/8/02	41.7	246	0.107	2.42	105	0.109	3.62	14.0	0.024	0.736
7/29/02	31.5	249	0.082	2.50	109	0.086	3.70	16.3	0.021	0.757
8/12/02	76	260	0.206	2.71	116	0.220	3.92	17.7	0.055	0.812
9/19/02	77.5	285	0.230	2.94	120	0.232	4.15	16.4	0.052	0.864
10/28/02	51	260	0.138	3.08	114	0.145	4.30	16.5	0.035	0.899

6/24/02 flow volume is an estimate based on the flow and precipitation for the peroid 4/10/02 to 10/28/02.

Figure A4.5. Cumulative sulfate, calcium, and magnesium mass release from the limestone addition control field tank 1.

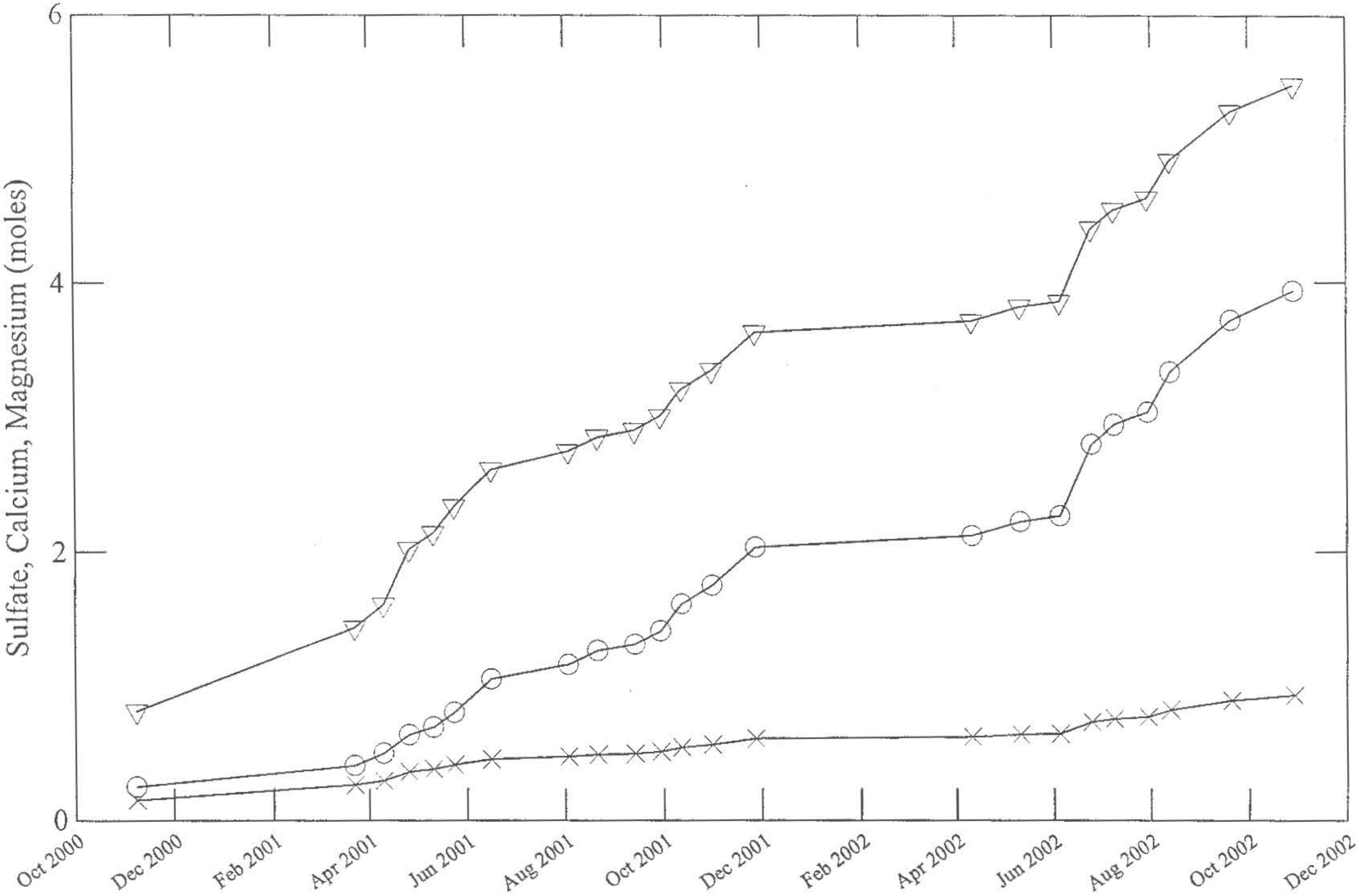


Figure A4.6. Cumulative sulfate, calcium, and magnesium mass release from the limestone addition control field tank 6.

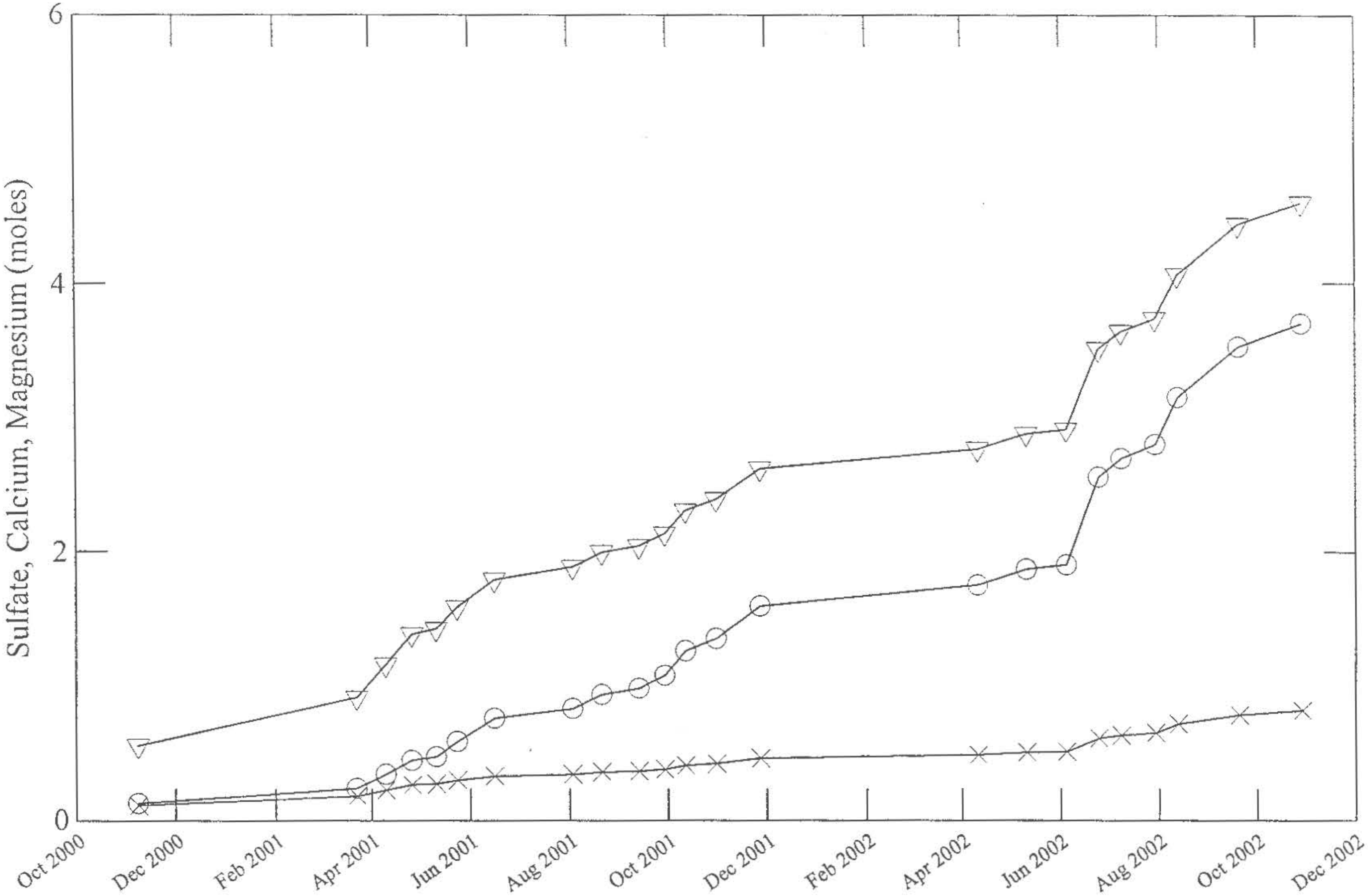


Figure A4.7. Cumulative sulfate, calcium, and magnesium mass release from the limestone addition 1:1 ratio field tank 2.

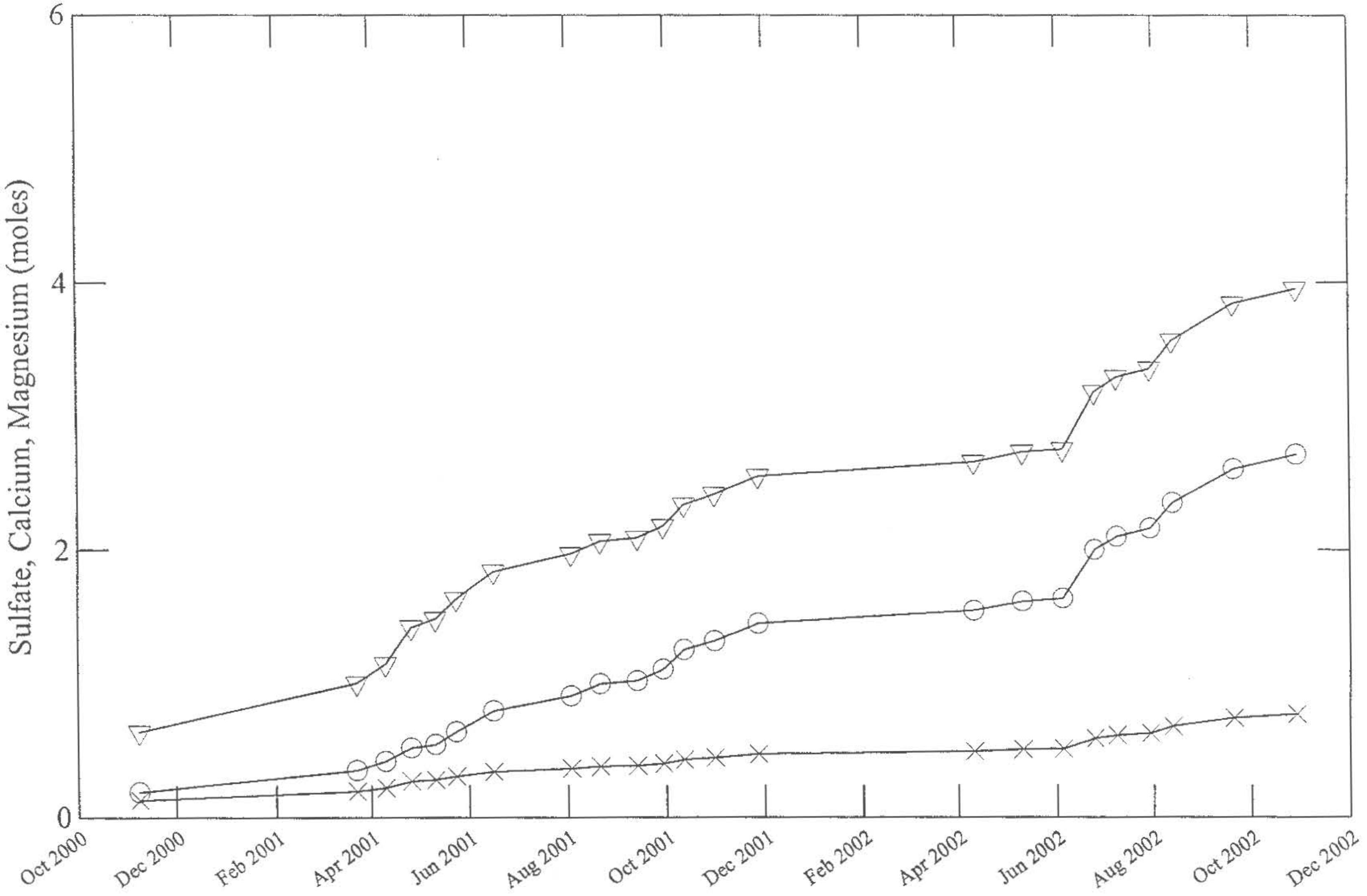


Figure A4.8. Cumulative sulfate, calcium, and magnesium mass release from the limestone addition 1:1 ratio field tank 5.

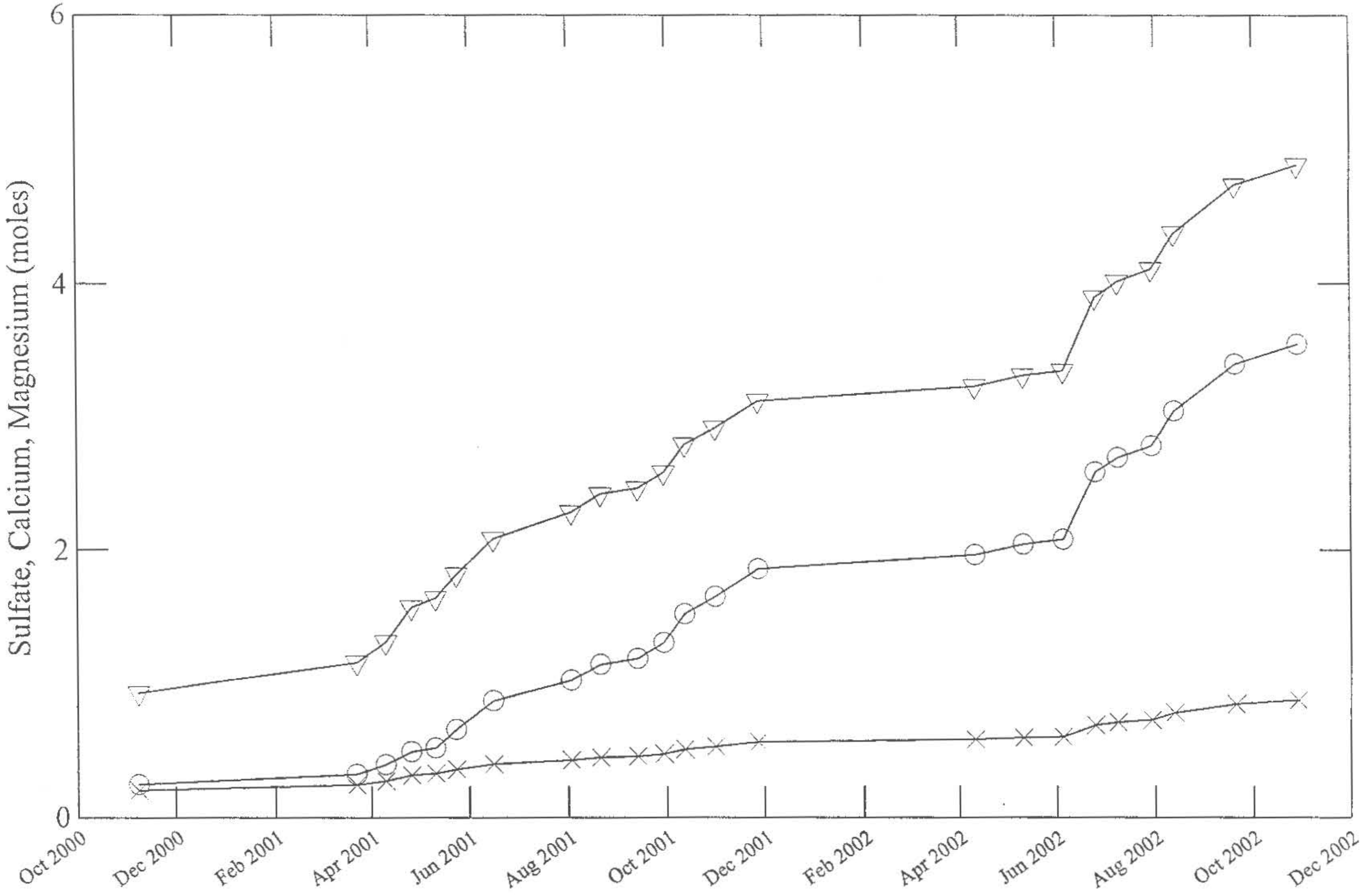


Figure A4.9. Cumulative sulfate, calcium, and magnesium mass release from the limestone addition 3:1 ratio field tank 3.

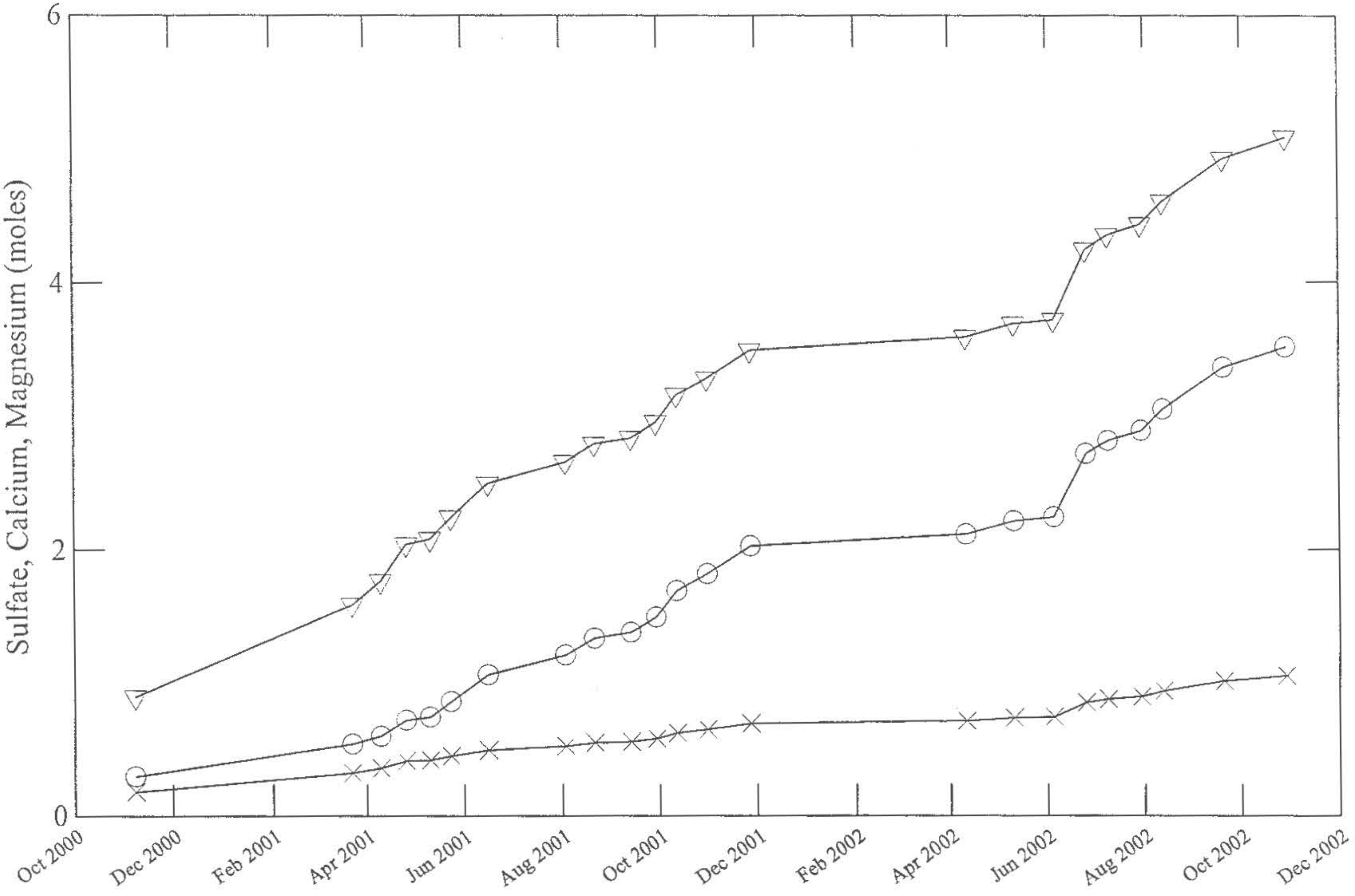
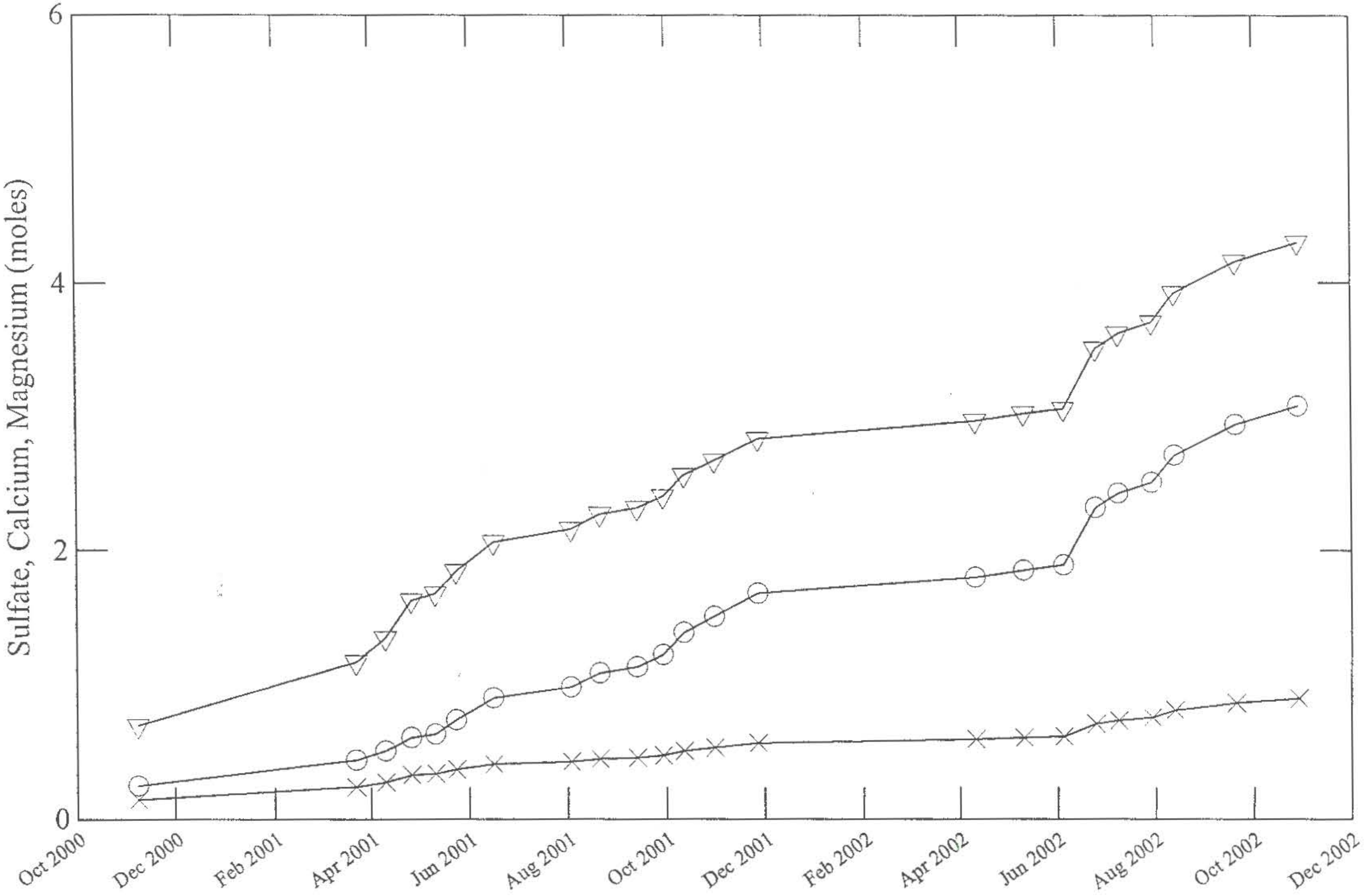


Figure A4.10. Cumulative sulfate, calcium, and magnesium mass release from the limestone addition 3:1 ratio field tank 4.



APPENDIX 5

QUALITY ASSURANCE

Attachment A5.3.

Department of Natural Resources
Laboratory Quality Assurance Program

Laboratory Calibration

- pH and specific conductance (SC) analysis of laboratory distilled water.
- Reference checks of Eh meter and probe.
- Daily calibration of pH meters with standard buffer solutions.
- Calibration of conductivity meters with standard reference solutions.
- Precision comparison between pH meters.
- Calibration at any time meter or probe is suspect.
- Accuracy check with inter-laboratory set point standards for pH, SC and alkalinity.
- Dissolved oxygen meters are calibrated before each sampling.

Laboratory Instrument Maintenance

- pH probes are cleaned according to probe manual instructions (EDTA) plus additional cleaning when used for measuring pH of extraordinarily dirty or organic samples (HCL).
- SC meters are cleaned using a mild cleaning solution when needed.

Analytical set points and distilled water blanks

- One masked set point per 50 metals or sulfate samples sent to the Minnesota Dept. of Agriculture.
- One masked distilled water blank per 50 samples sent to the Minnesota Dept. of Agriculture to monitor for contamination from sample collection or laboratory washing procedures.

Attachment A5.2. MN Department of Agriculture (MDA) quality assurance reporting methods.

The DNR sends MDA batches of samples that contain approximately fifty samples. MDA performs quality assurance/quality control (QA/QC) analyses on each batch following the procedures outlined in Attachment A6.1. QA/QC analysis is run on each parameter in a batch unless sample volume becomes limiting. When sample volume becomes limiting it is up to the discretion of MDA as to which parameters will be analyzed. If re-runs of set point standards that are out of range are needed and if there is an insufficient amount of sample remaining to perform the analysis, it is noted on the final report.

The test typically performed include percent recovery of spiked samples duplicate analyses, laboratory blanks, and analytical set point standards. The following three pages are examples of QA/QC reports for Flame AA analyses, ICP-MS analyses, and sulfate analyses. When reports are received by the DNR they are examined for accuracy and completeness by the DNR laboratory supervisor and then retained on file. Any discrepancies are reported to MDA so the proper corrective action can be performed.

Guide to analytical Values for Flame and Zeeman GFAA

Matrix Water
Date December 1995

The following detection limits were determined by analyzing the corresponding analyses on Flame and Zeeman GFAA.

Seven standard solutions of the same concentration, alternating with seven blanks were used to get the corresponding absorbance.

From the absorbance reading each detection limit was calculated using the Method Detection Limits according to US EPA recommendation.

Analyze	Method	Method Description	Detection Limit	Method	Method Description	Detection Limit
			ug/L			ug/L
Al	3111D	Flame/Nitrous oxide	500			
As				3113B	Furnace Zeeman	0.8
Ca	3111B	Flame/Acetylene	100			
Ca	3111D	Flame/Nitrous oxide	80			
Cd	3111B	Flame/Acetylene	100	3113B	Furnace Zeeman	0.4
Co	3111B	Flame/Acetylene	100	3113B	Furnace Zeeman	0.4
Cu	3111B	Flame/Acetylene	100	3113B	Furnace Zeeman	0.4
Fe	3111D	Flame/Acetylene	100			
Hg				2452	Auto Cold Vapor	0.5
K	3111B	Flame/Acetylene	50	3113B		
Mg	3111B	Flame/Acetylene	80	3113B		
Mn	3111B	Flame/Acetylene	100	3113B		
Na	3111B	Flame/Acetylene	50	3113B		
Ni	3111B	Flame/Acetylene	100	3113B	Furnace Zeeman	0.8
Pb	3111B			3113B	Furnace Zeeman	0.8
Sb				3113B	Furnace Zeeman	0.4
Zn	3111B	Flame/Acetylene	50	3113B		

Key:

3111B = Flame analyses using Air/acetylene gas
3111D = Flame analyses using Acetylene/Nitrous oxide gas
3113D = Zeeman Graphite Furnace analyses using argon gas

Source:

1) Standard Methods for the examination of water and wastewater 18th Ed. 1993.
   Greenberg, E. Arnold: Clesceri, S. Lenore and Easton, D. Andrew.
2) Analytical Methods for Graphite Tube Atomizers, Varian. 1988.
   Rothery, R. Varian Australia Pty. Ltd.
3) Analytical Methods Flame Atomic Absorption Spectrometry. 1989.
   Rothery, E. Varian Australia Pty. Ltd.
4) Methods for the determination of metals in environmental samples. 1992.
   U. S. Environmental Protection Agency.
   Smoley, C. K.

$$\underline{MDL = (t) * (s)}$$

Where t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. (t - 3.14 for several replicates).

s = standard deviation of the replicate analyses.

and reference material analyses and additional field and/or analytical system evaluations by outside agencies or individuals.

QA Reports to Management

A quality assurance report is generated by the Minnesota Department of Agriculture and Laboratory Services Division and sent to MDA and MDNR management at least once a year.

The report may contain the following:

- Changes in Quality Assurance Project Plan,
- Summary of quality assurance/quality control programs, training and accomplishments,
- Results of technical systems and performance evaluation audits,
- Significant quality assurance/quality control problems, recommended solutions and results of corrective actions,
- Summary of data quality assessment for precision, accuracy, representativeness, completeness, comparability and method detection limit,
- Discussion of whether the quality assurance objectives were met and the resulting impact on technical and enforcement areas,
- Limitations on use of the measurement data and discussion of the effects of such limitations on the defensibility of the data.

The MDNR Reclamation Laboratory QA Officer and MDA QA Officer will review this plan once a year.

V = number of measurements judged valid
n = number of measurements necessary to achieve a specified statistical level of confidence in decision making

To determine "n" a judgment must be made regarding the amount of data required to provide adequate evidence that a system is in control. Completeness is calculated for monitoring programs where similar analyses are performed on a regular basis. Loss of data due to such occurrences as breakage of containers, spilling of the sample, contamination, instrument failure or exceeding holding time before analysis must account for no more than 10 percent of all requested analysis. If excessive loss of data occurs, the reasons must be identified and evaluated and, if necessary, action must be taken to solve the problem(s).

Corrective Action

Corrective action is taken whenever data is determined as unacceptable.

Corrective action is taken in the order listed below.
Review of sample collection procedures.
Review of analytical raw data and calculations.
Review of laboratory procedures - Was the analytical method followed?
Review of analytical method - Is it applicable?
Review of instrument operation, calibration and maintenance.
Review of the calibration standard(s) used.
Review of quality control measurement (spike, duplicate, surrogate, etc.).

As a result of the above review, further corrective action may be identified and pursued as necessary:
Repeat the sampling and corresponding documentation.
Issuing an amended analytical report.
Repeat analysis (confirmation methods).
Repair, recalibration or replacement of instrumentation.
Additional training of staff.

Persistent problems require a thorough review of all field and analytical data (including quality control measurements and procedures), increased check sample

Sample standard deviation (s) and coefficient of variation (CV) are used when there are at least three replicate measurements.

The second measure of variability which adjusts for the magnitude of the analyte is relative percent difference (RPD) or relative range (RR). This measure is used when duplicate measurements are made and is defined:

$$RR\ or\ RPD=\frac{|A-B|}{\left(\frac{A+B}{2}\right)}*100$$

where: A = First observed values
B = Second observed values

Precision is monitored by plotting control charts for repetitive analysis. A warning limit of ±2s is established with a control limit of ±3s (see Section 3).

Accuracy is the nearness of a result to the true value and is often described as error, bias or percent recovery. Accuracy estimates are frequently based on the recovery of surrogate spikes and/or the recovery of know analytes. The percent recovery is calculated as:

$$\%R=\left(\frac{SSA-S}{SA}\right)*100$$

where: SSA = measured concentration in spiked aliquot
S = measured concentration in unspiked aliquot
SA = actual concentration of spike added

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount expected to be obtained under correct, normal conditions. For all measurements, completeness is defined:

$$\%C=\left(\frac{V}{n}\right)*100$$

where: %C = percent completeness

- Are subject to frequent failure,
- Have limited useful lifetime,
- Cannot be obtained in a timely manner should failure occur.

Assessment of Data

An objective of the laboratory is to demonstrate that performance on all analyses is in statistical control. Routine procedures used to assess reliability and quality of data are specified in the laboratory standard operating procedures (SOPs).

For residue analysis, duplicates are used to establish precision, spike sample recoveries are used to establish accuracy and blanks are analyzed to assure non-interference from solvents, reagents and laboratory environment.

Precision refers to the reproducibility of replicate results about a mean which is not necessarily the true value. Duplicate analysis is the primary means of evaluating measurement data variability or precision. Two commonly used measures of variability which adjust for the magnitude of analyte concentration are coefficient of variation and relative percent difference.

The coefficient of variation is used most often when the size of the standard deviation changes with the magnitude of the mean. Coefficient of variation (CV), also called relative standard deviation (RSD), is defined:

$$CV\ or\ RSD = \left(\frac{s}{y}\right) * 100$$

where: y = mean of replicate analyses
s = sample standard deviation, defined as:

$$S = \sqrt{\sum_{i=1}^{N} \frac{(y_i - y)^2}{n - 1}}$$

where: y_i = measured valued of the ith replicate
y = mean of replicate analyses
n = number of replicates

- Establishment of maintenance responsibility.
- Establishment of maintenance schedules for major and/or critical instrumentation and apparatus.
- Establishment of an adequate inventory of critical spare parts and equipment.
- Documentation and filing of all service and maintenance records.

The Agronomy Laboratory supervisor is responsible for maintenance of laboratory instruments and equipment. The appropriate program managers are responsible for the maintenance of field equipment. With assistance from the Laboratory and Reclamation Laboratory Services Quality Assurance Officers, the Agronomy Laboratory establishes maintenance procedures and schedules for each piece of major equipment. Responsibility for individual items is delegated to technical personnel. The manufacture's recommendations and/or the protocols for instrument maintenance and calibration are followed. Each piece of major equipment is designated a repair and maintenance logbook where all maintenance activities are dated and documented by laboratory or filed personnel.

In the interest of maintaining instruments in top operating condition, it is management's policy to secure annual service contracts with instrument manufacturers whenever financially possible. The service contracts are especially desirable for laboratory instruments. Under the service contracts, certified service engineers perform preventive maintenance, calibration and repair for instruments. Laboratory personnel perform routine maintenance and repair between manufacturers' service to ensure correct performance of an instrument.

Analytical balances are serviced by certified service engineers at least once a year. In addition to performing repair and maintenance, the engineer calibrates and certifies each analytical balance. Laboratory personnel check the calibration of the balance with a class S weight at least four times a year. Digital pH meters are checked before each use with standards and calibrated according to the manufacturer's directions. Freezers and refrigerators are monitored to assure that proper temperatures are maintained and that failure has not occurred.

An adequate inventory of spare parts is maintained to minimize equipment down time. This inventory emphasizes those parts which:

The Laboratory Quality Assurance Officer performs in-house systems audits to identify strengths, weaknesses, potential problems and solutions to problems. The audits provide an evaluation of the adequacy of the overall measurement systems to provide data of sufficient quantity and quality to meet the comprehensive laboratory pesticide program's objectives. The in-house systems audits are the basis for quality assurance reports to management.

The in-house systems audit consist of observing the various aspects of the laboratory activities related to this project. Check lists which delineate the critical aspects of each procedure are used during the audit and serve to document all observations. At a minimum, the following topics will be evaluated during the internal audit:

1. GENERAL PROCEDURES
    A. Procedures for Sampling and Sample Documentation
    B. Documentation of Procedures
    C. Sample Receipt and Storage
    D. Sample Preparation
    E. Sample Tracking

2. ANALYTICAL PROCEDURES
    A. General Instrumentation Procedures
    B. Calibration Procedures
    C. Internal Quality Control
    D. Data Handling Procedures

Preventative Maintenance Procedure and Schedule

1. Field Maintenance
   None

2. Laboratory Instrument Maintenance
   The primary objective of a comprehensive maintenance program is to ensure the timely and effective completion of a measurement effort. Preventive maintenance is described in the laboratory or field standard operating procedures (SOPs) and appropriated instrument manual. It is designed to minimize the down time of crucial sampling and/or analytical equipment due to component failure. The focus of the program is in four primary areas:

assurance program.

The quality control procedures for analytical methods used for misuse cases may include:

- Demonstration of analytical capability,
- Analysis of a quality control check sample, when available,
- Daily instrument check,
- Recoveries of or matrix spikes,
- Analysis of reagent blank,
- Duplicate analysis,
- Analysis of laboratory control standards,
- Blind performance evaluation samples,
- Analysis of instrument quality control standards,
- Confirmation of analyte.

Performance and System Audits

The Minnesota Department of Agriculture is committed to participate in the evaluation of the laboratory quality assurance program and to lend itself to any coordinated on-site systems audits by qualified representatives of MDNR. The department is also committed to using the results of such performance and systems audits to improve the reliability, defensibility, capability and efficiency of the laboratory and filed operations. A quality assurance/quality control manual will also be available to the MDNR-mineral for review.

LSD will maintain accreditation with the Minnesota Department of Health with respect to clean water requirements including participation in EPA WP and WS proficiency samples.

Systems and laboratory audits along with analytical data and record review, may be performed by qualified representatives of MDNR which reserves such audit rights. The audit is conducted upon joint consent of both agencies. The report of all findings and recommendations are made promptly to the MDA. The systems audit includes areas in the laboratory immediately impacting overall quality assurance.

Assurance Officer if required or desirable. When a review indicates a need, the analysis is repeated using either the same method or an alternate method. Questionable data may result from the condition of the sample, inadequacy of the method, lack of validation, time constraints or other factors.

Any questionable data will be clearly identified and qualified. The Laboratory Quality Assurance Officer conducts periodic in-depth audits to assure compliance with the validation requirements.

3. Reporting
   Analytical data is reported according to the format(s) provided in the standard operating procedures. In addition to the analytical results, the reference for the method and quality control results are reported. Quality control results may include spike recovery, results of duplicate analyses, analysis of reagent blanks, but are not limited to these. When the compound(s) of interest is not detected in the sample(s), it is reported as such with the method detection limit. Any pertinent observations about the samples or the analytical process are also reported.

   All written reports will be sent to the MDNR Program Coordinator.

Internal Quality Control Checks

The internal quality control (QC) checks are a systematic in-house approach to ensure the production of high quality data. The objectives of these control checks are:

- To provide reliable and defensible analytical results,
- To provide a measure of the precisions and accuracy of the analytical methods,
- To monitor the accuracy and precision of the analyst,
- To identify problematic methods which can be flagged for further research,
- To detect training needs within the laboratory,
- To provide a permanent record of instrument performance which is used for validating data and projecting instrument repair or replacement needs, To monitor the effectiveness of the quality assurance program and laboratory performance and provide a basis for modifications of the quality

the designated logbook(s).

Depending on the method, a three to five point calibration curve will be used.

Analytical Procedures

1. Analytical Procedures
   All analyses for permit samples will be done according to methods approved by the Minnesota Department of Health as written in the MDA methods manual. These methods are based on approval EPA methodologies and Standard Methods for the Examination of Water and Wastewater.

   Other analyses will be done using laboratory methods based on EPA, ASTM, AOAC, etc. methodologies.

Data Analysis, Validation and Reporting

This section describes the basic procedures for data analysis, validation and reporting for this project.

1. Data Analysis
   Data analysis is performed on a batch run basis for samples analyzed using FAA and GFAA. Out of range samples are diluted manually for FAA and automatically for GFAA. Colorimetric autoanalysis usually relies on batch data analysis where confirmatory samples are then redirected to another automated method (IC) or a manual method. Manual methodology requires a sample by sample data analysis procedure, with confirmation by an alternate method if indicated. Details of data analysis are contained individual methods.

2. Validation of Results
   Validation of data is described in detail in the laboratory standard operating procedures. In most cases, data validation consists of a review of the analytical method. calculations and quality control results. Initial review is done by the analyst, and final review by the Chemistry Supervisor or a designated Senior Analyst. Certain samples or cases may be validated by the Laboratory Quality

Samples are to be properly documented, preserved, packaged, maintained under custody and transferred to the laboratory in a defensible manner. The Laboratory Information Section Supervisor should notify the MDNR Program Coordinator, appropriate MDNR Field Project Leader or Reclamation Laboratory QA Officer when problems are encountered with the quality of incoming samples or when laboratory problems arise that could affect the reliability and/or defensibility of analytical results.

4. Analysis
   A supervisor assigns the sample(s) to an analyst. After assignment, the sample custodian retrieves the sample(s) and transfers it to the analyst who completes the appropriate lines on the custody form. If the sample(s) is assigned to a different analyst, the appropriate lines in the second column of the custody form are completed by the new analyst. Similarly, the third column or even additional sheets can be used to document additional sample transfers within the laboratory. The original seal(s) should be kept with the sample(s) and maintained in a legible condition. Upon completion of the analysis, any remaining sample is placed in the appropriate storage location.

Calibration Procedures and References

1. Field Equipment Calibration
   None

2. Laboratory Calibration
   Each instrument used routinely in the laboratory should be monitored, calibrated, and maintained. Specifications for instrument maintenance, calibration and monitoring are described in manufacturer's manuals, in analytical methods, and/or appropriate standard operating procedures. If an instrument malfunctions, or if improper sensitivity, resolution and/or reproducibility is detected, corrective action is necessary before analyses are attempted. Any corrective action taken will be documented in the appropriate instrument manual.

   Analytical standards used to prepare calibration or standard solutions are obtained from the National Institute for Standards and Technology (NIST), EPA, USDA, FDA or other reliable sources. Stock standard solution(s) are prepared as specified in the SOP. All inform on their preparation is recorded in

Field Sampling Requirements

1. Type of Samples to be Collected.
   Aqueous samples will be colleted.

2. Field Sampling Requirements: NA

3. NPDES samples will require chain of custody and proper preservation as required for permit samples. This is required in the QA plan approved by Minnesota Department of Health.

4. Preservation
   All metals samples will be preserved with ultra pure nitric acid. Samples requiring refrigeration (storage at 4°C ± 2°) will be shipped on ice or cool packs to the MDA laboratory.

Sample Custody Requirements

1. Transportation of Samples from Field to Laboratory
   Regulator samples will either be shipped by State contract courier or hand delivered by Minerals personnel to MDA within 2 working days.
   Permit samples will wither be shipped by State contract courier or hand delivered by Minerals personnel to MDA within 2 working days of shipment. The samples will be sent on ice.

2. Notification Procedure
   MDA will be notified by the MDNR Program Coordinator or MDNR QA Officer when *Permit* samples are being shipped. MDNR will also alert MDA when "non regular" samples are being shipped.

3. Sample Log-in Procedure
   Upon receipt of the sample(s), the sample custodian inspects the shipping container(s), the sample(s), the official seal(s), and documentation related to the sample(s) and other records. If accepted for analysis, the sample(s) are entered by the sample custodian into the sample logbook, database and assigned a unique laboratory number.

2. Parameter List, Matrix Type, Required Action Limits, Method Detection Limits

   Parameters
   Metals, sulfates and nutrients.

   Matrices
   Aqueous and Solids

   Required Action Limit
   Required action limits will be determined by the MDNR personnel prior to the analysis of samples by MDA. Action limits will be communicated to the Laboratory by the Minerals Reclamation Laboratory QA Officer.

   Method Detection Limit
   Method detection limits are determined by the laboratory following guidelines defined in EPA CFR 40 Part 136, Appendix B. Reporting limits are based on the lab MDLs and requirements for the program.

3. Laboratory Methods
   The laboratory will follow methods based on EPA methodologies and Standard Methods for the Examination of Water and Wastewater.

4. Samples
   4.1 Required Turn-Around Time for Analysis
   "Regular" parameters: 30 days after MDA receipt.
   "Permit" parameters within the stated time listed in the MPCA permit.

5. Quality Control Samples
   5.1 Field Blanks: One blank for every 50 samples of each experiment.
   5.2 Laboratory QC requirements and minimum volume of sample needed:
      - Metals- 60 mL
      - Sulfates- 60 mL
   5.3 Blind Set Points: One submitted with every box of samples.

Attachment A5.1.

Minnesota Department of Agriculture
Quality Assurance Program

Quality Assurance Objectives

Precision, accuracy, completeness, data comparability and sample representativeness are necessary attributes to ensure that analytical data are reliable, scientifically sound, and defensible. Each analytical result or set of results generated for this project should be fully defensible in any legal action, whether administrative, civil or criminal.

1. Definitions
    1.1 Precision
    Whenever possible, a minimum of one duplicate sample should be run in order to determine precision. It is understood that in some cases there may be insufficient sample to run duplicates and therefore a determination of precision would not be possible.
    1.2 Accuracy
    Whenever possible, a minimum of one matrix spike should be run in order to determine accuracy. It is understood that in some cases there may be insufficient sample to run matrix spikes and therefore a determination of accuracy would not be possible.
    1.3 Completeness
    Should be 100% ideally. Realistically a minimum level of 90% is expected.
    1.4 Comparability
    Should be ensured by adherence to method protocols.
    1.5 Representativeness
    Should be ensured by adherence to standard laboratory sub-sampling protocols. The nature of the material being sampled must be taken into account when subsampling.

The precision and accuracy of each method is dependent on the sample matrix and analyte concentration. Therefore, for these types of analyses, the matrix and concentration determine the values of precision and accuracy (bias) which are acceptable.

Table A5.1. DNR laboratory instrument Quality Control.

Date	Distilled S.C.[1]		Distilled pH	pH vs.	pH	Calibrate	Eh reference	Clean pH Probes
	M-2[2]	M-3[2]		O-720A[3]	O-720[3]	SC meters	Check	
08/14/00	0.4	0.3	6.99	8.69	8.74	YES	YES	EDTA
08/21/00	0.4	0.4	7.11	5.35	5.39	YES	YES	EDTA
08/28/00	0.4	0.4	6.13	4.83	4.84	YES	YES	EDTA
09/05/00	0.5	0.5	6.03	4.19	4.13	YES	YES	EDTA
09/11/00	0.4	0.5	6.05	8.57	8.61	YES	YES	EDTA
09/18/00	0.6	0.6	6.03	5.31	5.36	YES	YES	EDTA
09/25/00	0.4	0.5	6.31	4.70	4.69	YES	YES	EDTA
10/02/00	0.6	0.5	6.51	4.11	4.11	YES	YES	EDTA
10/09/00	0.6	0.5	6.18	7.16	7.14	YES	YES	EDTA
10/16/00	0.6	0.6	6.48	5.36	5.39	YES	YES	EDTA
10/23/00	1.3	1.3	5.96	3.16	3.18	YES	YES	EDTA
10/30/00	0.8	0.7	6.35	4.36	4.42	YES	YES	EDTA
11/06/00	1.0	0.9	6.41	8.59	8.59	YES	YES	EDTA
11/13/00	0.7	0.6	6.73	5.36	5.33	YES	YES	EDTA
11/20/00	0.5	0.5	6.31	4.88	4.89	YES	YES	EDTA
04/09/01	0.4	0.4	6.66	5.27	5.33	YES	YES	EDTA
04/16/01	0.4	0.3	6.46	7.55	7.63	YES		EDTA
04/23/01	0.6	0.5	6.35	4.89	4.91	YES	YES	EDTA
04/30/01	0.4	0.5	6.81	6.25	6.31	YES		EDTA
05/07/01	0.6	0.6	6.42	5.33	5.37	YES	YES	EDTA
05/14/01	0.5	0.5	6.74	6.88	7.01	YES		EDTA
05/21/01	0.3	0.3	6.90	2.53	2.60	YES		HCL
05/29/01	0.4	0.5	6.63	7.07	6.93	YES		EDTA
06/04/01	0.2	0.4	7.73	5.36	5.39	YES		HCL
06/11/01	0.4	0.4	6.08			YES	YES	EDTA
06/18/01	0.6	0.4	7.28	4.71	4.72	YES		HCL
06/25/01	0.5		7.20	7.56	7.64	YES		EDTA
07/03/01	0.4	0.5				YES		EDTA
07/09/01	0.4		6.42	4.11	4.11	YES	YES	EDTA

Table A5.1. DNR laboratory instrument Quality Control.

Date	Distilled S.C.[1]		Distilled pH	pH vs.	pH	Calibrate	Eh reference	Clean pH Probes
	M-2[2]	M-3[2]		O-720A[3]	O-720[3]	SC meters	Check	
07/16/01	0.4	0.3	6.41	7.47	7.56	YES		EDTA
07/23/01	0.4	0.5	6.23	6.33	6.36	YES		HCL
07/30/01	0.5	0.5	6.27	3.49	3.42	YES		EDTA
08/06/01	0.6	0.6	6.45	5.34	5.38	YES	YES	HCL
08/14/01		0.4	6.37	7.53	7.59	YES		HCL
08/20/01	0.6	0.6	7.43			YES		HCL
08/27/01	0.4	0.4	6.20	7.64	7.64	YES		EDTA
09/04/01		0.4	7.22	5.09	5.07	YES	YES	HCL
09/10/01	0.4	0.3	6.09	7.01	7.03	YES		EDTA
09/17/01	0.4	0.4	6.77	5.61	5.69	YES		HCL
09/25/01	0.5	0.5	6.05	7.07	7.06	YES		EDTA
10/01/01	0.6	0.3	5.73	5.93	5.96	YES		
10/15/01	0.4	0.4	6.80	5.10	5.15	YES		EDTA
10/22/01	0.4	0.4	6.62	6.99	7.01	YES		HCL
10/29/01	0.4	0.4	7.57	5.41	5.43	YES	YES	EDTA
04/29/02	.5, .4	0.2	6.59	8.53	8.57	YES		HCL
05/06/02	.3, .3	0.2	6.53	5.28	5.32	YES		EDTA
05/13/02	.3, .3	0.3	6.02			YES		EDTA
05/20/02	.4, .5	0.5	6.41	7.15	7.18	YES	YES	HCL
05/28/02	.6, .6	0.5	6.96	3.17	3.21	YES		EDTA
06/03/02	.6, .7	0.3	6.30	5.47	5.49	YES		HCL
06/11/02	.4, .4	0.6	6.74	7.11	7.17	YES		EDTA
06/17/02	.7, .6	0.4	6.75	5.18	5.22	YES		EDTA
07/01/02	.6, .55	0.6	6.90	9.17	9.41	YES	YES	EDTA
07/08/02	.65, .5	0.7	6.36	5.12	5.14	YES		EDTA
07/15/02	.8, .9	1.0	7.18	7.31	7.35	YES		EDTA
07/22/02	.7,.6	1	6.98	6.27	6.24	YES		EDTA
08/05/02	.7..6	0.85	6.73	6.63	6.61	YES	YES	EDTA
08/12/02	.7,.45	0.8	7.15	6.68	6.73	YES		EDTA

Table A5.1. DNR laboratory instrument Quality Control.

Date	Distilled S.C.[1]		Distilled pH	pH vs.	pH	Calibrate	Eh reference	Clean pH Probes
	M-2[2]	M-3[2]		O-720A[3]	O-720[3]	SC meters	Check	
08/19/02	.6,.75	0.85	6.44	6.79	6.74	YES		EDTA
08/26/02	0.4	0.55	6.14	6.03	6.02	YES		EDTA
09/03/02	0.4	0.55	6.76	6.4	6.38	YES		EDTA
09/09/02	0.65	0.5	6.47	7.3	7.22	YES		EDTA
09/16/02	0.5	0.5	7.32	7.2	7.19	YES		EDTA
09/23/02	.6,.4	0.5	6.5	7.29	7.28	YES		EDTA
09/30/02	.55,.4	0.5	6.86	7.67	7.73	YES	YES	EDTA
10/09/02	.5,.4	0.55	6.29	5.28	5.34	YES		EDTA
10/14/02	.45,.4	0.4	6.26	7.65	7.62	YES		EDTA
10/21/02	.4,.4	0.55	6.18	7.89	7.86	YES		EDTA
10/28/02								

[1] SC= Specific Conductance

[2] Specific Conductance Meters

[3] O720A and O720 = comparison of pH meters

[4] new RO tank = change reverse osmosis tank for distilled water systems

Table A5.2. Laboratory measurements of certified conductance setpoints(µS/cm).

Date	Measured SC Meter 1	Measured SC Meter 2	Measured SC Meter 4	Mean SC Setpoint	95% Confidence Interval[1]
08/21/00	925	900	900	930	846-1013
09/18/00	950	925	900	930	846-1013
10/16/00	925	900	900	930	846-1013
11/13/00	925	925	925	930	846-1013
12/11/00	925	900	925	930	846-1013
04/09/01	975	900	950	930	846-1012
05/07/01	975	950	950	930	846-1012
06/04/01	880	na	920	930	846-1012
07/03/01	920	na	925	930	846-1012
08/06/01	925		950	930	846-1012
09/06/01	na		950	930	846-1012
10/29/01	925		950	930	846-1012
12/03/01	445		450	451	390-456
04/01/02	575	525	530	549	505-593
05/06/02	560	550	525	549	505-593
06/03/02	560	525	550	549	505-593
07/01/02	550	525	550	549	505-593
08/05/02	550	550	575	549	505-593
09/04/02		550	575	549	505-593
09/30/02	875	850	850	843	773-913
11/04/02	850	800	850	843	773-913

Table A5.3. Laboratory measurements of certified alkalinity setpoints(mg/L $CaCO_3$).

Date	Measured alkalinity	Measured alk (duplicate)	Setpoint alkalinity	95% Confidence Interval[1]
08/21/00	55	50	54.3	48.1-60.4
09/18/00	55	50	54.3	48.1-60.4
10/16/00	55	55	54.3	48.1-60.4
11/13/00	55	55	54.3	48.1-60.4
12/11/00	52.5		54.3	48.1-60.4
04/09/01	54		54.3	48.1-60.4
05/07/01	54		54.3	48.1-60.4
06/04/01	55	52	54.3	48.1-60.4
07/03/01	57		54.3	48.1-60.4
08/06/01	55		54.3	48.1-60.4
10/29/01	53		54.3	48.1-60.4
12/03/01	37		36.2	31-41.4
04/02/02	37		36.2	31-41.4
05/06/02	50		52.5	46.4-58.6
06/03/02	50		52.5	46.5-58.6
07/01/02	50		52.5	46.4-58.6
08/05/02	50		52.5	46.4-58.6
09/04/02	50	50	52.5	46.4-58.6
09/30/02	22		22.7	18.3-27.1
11/04/02	22		22.7	18.3-27.1

Table A5.4. Laboratory measurements of certified pH setpoints.

Date	Measured pH Orion 720	Measured pH Orion 720A	Mean Setpoint pH	95% Confidence Interval[1]

Table A5.4. Laboratory measurements of certified pH setpoints.

Date	Measured pH Orion 720	Measured pH Orion 720A	Mean Setpoint pH	95% Confidence Interval[1]
08/21/00	5.39	5.35	5.14	4.66-5.63
09/18/00	5.36	5.31	5.14	4.66-5.63
10/16/00	5.39	5.36	5.14	4.66-5.63
11/13/00	5.33	5.36	5.14	4.66-5.63
12/11/00	5.34	5.36	5.14	4.66-5.63
04/09/01	5.33	5.27	5.14	4.66-5.63
05/07/01	5.37	5.33	5.14	4.66-5.63
06/04/01	5.39	5.36	5.14	4.66-5.63
07/03/01	5.43	5.39	5.14	4.66-5.63
08/06/01	5.38	5.34	5.14	4.66-5.63
09/06/01	5.36	5.34	5.14	4.66-5.63
10/29/01	5.43	5.41	5.14	4.66-5.63
12/03/01	5.49	5.46	5.14	4.66-5.63
04/02/02	5.33	5.33	5.14	4.66-5.63
05/06/02	5.32	5.28	5.14	4.66-5.63
06/03/02	5.49	5.47	5.14	4.66-5.63
07/01/02	NA			
08/05/02	6.38	6.37	6.35	6.19-6.50
09/04/02	6.38	6.40	6.35	6.19-6.50
09/30/02	6.36	6.33	6.35	6.19-6.50
11/04/02	6.36	6.37	6.35	6.19-6.50