Controlling Mine Drainage Problems-
New Approaches for Waste Rock Stockpiles

A Final Report to the Minerals Coordinating Committee

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Kim Lapakko
David Antonson
Andrea Johnson

Minnesota Department of Natural Resources
Division of Lands and Minerals
500 Lafayette Road, Box 45
St. Paul, MN 55155-4045

TABLE OF CONTENTS

TABLE OF CONTENTS (continued)

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDICES

1. Solids composition of rock from prediction bins and limestone addition tanks
2. Field precipitation, flow and reaction conditions
3. Drainage quality and concentration vs. time from prediction bins and limestone addition tank
4. Cumulative mass release for prediction bins and limestone addition tanks
5. Quality assurance program

0. EXECUTIVE SUMMARY

Minerals Coordinating Committee (MCC) funding was used to initiate field experiments to 1) conduct field scale predictive tests on Archean greenstone waste rock samples and 2) investigate the feasibility of adding fine grained limestone to acid generating Archean greenstone waste rock to control acid release with drainage from the rock.

Four field test piles containing greenstone rock (38 cubic meters) with sulfur contents of 0.02%, 0.20%, 0.39% and 0.67% were constructed from 21 July to 19 September 2000. 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%. The carbon dioxide content of all piles increased as particle size decreased, indicating any acid-neutralizing carbonate minerals were concentrated in the fine fractions.

There was no conclusive evidence indicating that oxygen was substantially depleted within the piles. Drainage volumes in 2002 ranged from 73 to 85 percent of the input precipitation, which was the only input to the piles. Drainage quality was determined between pile construction and 31 December 2001. Drainage pH values ranged from 7.3 to 8.8 and concentrations of trace metals (Cu, Ni, Co, Zn) were typically less than 0.02 mg/L. 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. Over time these minerals, in some piles, may be depleted or rendered unreactive. At this time the rate of acid production may exceed the rate of acid neutralization by the remaining minerals and, consequently, drainage will acidify.

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 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. After the first full year of operation, drainage from the controls remains neutral and drainage quality has shown little dependence on the amount of limestone added. Drainage pH ranged from 7.5 to 8.5 and, along with rates of sulfate, calcium and magnesium release, showed no strong correlation with limestone loading. Concentrations of alkalinity did tend to increase with limestone loading.

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 containing less than 0.16% sulfur produced drainage pH values above 6.0, a common water quality standard, and samples containing more than 0.16% sulfur 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.
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. Mine Waste Dissolution

3.1.1. 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).

3.1.2. 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.

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

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

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.

3.2. 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 *Thiobacillus 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 percent sulfide) containing 1.4 percent 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 al1997) 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. Analysis 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 for the coarse sand fraction to 2.6 to 4.7 m^2/g 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. 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). 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).

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 $\geq$ 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.

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

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 higher concentrations (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 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. This suggests 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, the sulfur content of the -100 fractions tended to be higher than that of the coarser fractions (Table A1.12). For the 0.20% and 0.67% sulfur piles, in contrast, sulfur content tended to decrease as particle size decreased. 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 average. In fact, almost all fractions finer than 0.25 inches had CO_2 contents at least three times the flow weighted mean. 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.

5.1.3. Waste Rock Drainage

5.1.3.1. Flow

The four test piles were constructed late in 2000, and the periods of flow were roughly two to four months. Flow through 20 November was used for calculations, although a small amount of flow occurred subsequently (Attachment A2.1). Due to equipment problems flow was not measured until November 1 (piles 1-3) or 9 (pile 4), 2000, roughly one month of the two to four month record for that year. 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). In 2001, the piles flowed from the end of April to the middle of December. The length of flow period was reflected in the annual flows, which ranged from roughly 3700 to 7000 liters in 2000 and from 17,500 to 20,400 liters in 2001 (Table 5).

Yield coefficients were calculated for one- to three-week periods after flow meters were installed in 2000, from 2 August - 28 November 2002 (the approximate period of unmeasured flow in 2000), and for the entire field 2001 season (April - December). The yield coefficients for all piles in 2000 and for piles 1,3 and 4 in 2001 ranged from 0.50 to 0.63. These yields are in good agreement with a range of 0.44 - 0.58 reported for an earlier field study conducted by the MN DNR (Eger and Lapakko 1985). The yield coefficient for pile 2 from August to November 2002 was higher (0.81), as were coefficients for the entire 2002 field season (0.73 to 0.85).

At this time it is not clear why the yield coefficients for the entire 2002 field season were higher than would be expected. Since the values from August to November, 2002 were in fairly good agreement with values from 2000 and the earlier field study (Eger and Lapakko 1985), they were selected for estimation of unmeasured flow in 2000 (appendix 2, attachment A2.2.).

5.1.3.2. Oxygen and Temperature Profiles

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. 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 trends were similar in all piles. Using data from pile 4 as representative, temperatures decreased from about 21°C in August 2000 to about -1°C in March 2001. Values then rose to near 21°C in July and dropped to around 2°C in December (Table 4, p. 3). The high and low temperatures for the lower sampling port were roughly 2-4 degrees less extreme than those from the upper port.

Oxygen contents were typically reported as 16% to 21% of the gas phase (overall range 6.7%-25.5%), as compared with 21% for atmospheric oxygen. Average oxygen concentrations were examined as a function of depth in the pile (top vs bottom port for five and ten foot distances into pile), distance into pile (five vs ten foot distance for top and bottom port) and sulfur content (top port five feet into pile, top port 10 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 four of six cases, average oxygen concentrations at the bottom of the pile were lower than at the top. There was no dependence on depth into the pile, and oxygen concentrations decreased as sulfur content increased in three of four cases. Considering the data collected to date and the problems with oxygen measurements, there is no conclusive evidence suggesting that extensive oxygen depletion has occurred within the piles.

5.1.3.3. Drainage Quality

Between 14 August and 20 November 2000 three to six drainage quality samples were collected from each bin and eleven samples from each bin during the 2001 field season. Drainage pH values ranged from 7.3 to 8.8 and alkalinities from roughly 35 to 100 mg/L as $CaCO_3$. Both values tended to peak during the summer of September 2002 (Figures 19-22, Table A3.1-A3.4). Sulfate concentrations ranged from 20 to 400 mg/L, with the lowest values from the 0.02% S rock and the highest values from the 0.67% S rock. All piles exhibited a sulfate concentration peak in 2000, perhaps due to the release of oxidation products accumulated after the rock was blasted, followed by a decline. In 2001 sulfate concentrations from the two lower sulfur piles plateaued in (Figures 19, 20), and concentrations from the two higher sulfur piles steadily increased.

Calcium, magnesium, sodium and potassium concentrations peaked in 2000 and generally plateaued in 2001 at levels roughly 01-0.3 times the peak concentrations (Figures 19-22). Molar concentrations during 2001 typically decreased in the order [Ca] > [Na] > [Mg] ~ [K]. Of the trace metals (Cu, Ni, Co, Zn) determined, copper was above the detection limit (0.002 mg/L) most often, followed by cobalt, nickel and zinc. Concentrations were typically below 0.02 mg/L.

Iron and manganese concentrations were typically below 0.3 and 0.035 mg/L, respectively, in 2000 and below 0.1 and 0.006 mg/L 2001. Nitrate concentrations were elevated in 2000, most likely due to the presence of residual blasting agents in the rock but showed a decreasing trend throughout the 2001 field season (Appendix 3, Tables A3.1-A3.4).

5.1.3.4. Chemical Release Rates

Rates of sulfate release varied during the 2001 field season, due largely to variations in flow. In particular, release was low from all piles during a very dry period from the middle of June to the beginning of August. Although rates of sulfate release tended to increase with increasing solid-phase sulfur content, this relationship was not precise. In particular, release from the 0.39% S pile was lower than would be expected and/or release from the 0.20% S pile was higher than expected. The total flow from the 0.39%S pile was about 13% lower than that from the other piles (Table 5), and this contributed to the lower sulfate release.

Retardation factors were calculated as the ratio of sulfate release rates in the field to those from samples of similar sulfur content in the laboratory. These values ranged from 0.051 to 0.48 (Table 6), 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.

Rates of calcium and magnesium release tended to be higher at the beginning of the 2001 field season. This suggests there may have been a more reactive phase containing calcium and magnesium, 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 ($CO_2 < 0.05\%$).

The calcium retardation factors were surprisingly high, ranging from 0.94 to 7.3. In contrast a range of 0.14 to 0.46 was reported 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 values 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. In contrast, retardation factors for magnesium ranged from 0.002 to 0.56. The extremely low Mg retardation factor is strongly influenced by the elevated magnesium release rate from the laboratory sample which had a siderite content of 17.9%. Values for the remaining three piles (0.041-0.56) compare favorably to the 0.053 to 0.36 range reported for Duluth Complex rock (Lapakko 1994).

5.2. Limestone Addition Tank Results

5.2.1. Introduction

Six field tanks were constructed 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 8).

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 9). 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), less than the 44% expected for pure calcite.

5.2.3. Flow

Flow from the limestone tanks ranged from 554 to 635 liters. Yield coefficients of 85% to 98% were calculated based on precipitation input of 649 liters (Table 11).

5.2.4. Drainage Quality

Each tank was sampled once in 2000 and thirteen times in 2001. Drainage pH typically ranged from 7.5 - 8.5, peaked from June to August, and was in essentially the same range for all tanks. Alkalinity ranged from approximately 25 - 130 mg/L as $CaCO_3$, tended to decrease throughout 2001, and tended to increase with increasing limestone loading (Tables A3.5 - A3.10). Sulfate concentrations typically ranged from approximately 100 - 400 mg/L, with no obvious variation with limestone loading. After elevated concentrations in the first three samples, calcium and magnesium concentrations typically ranged from 100 - 400 mg/L and 10 - 20 mg/L, respectively. 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).

5.2.5. Chemical Release Rates

Rates of chemical release were determined for sulfate, calcium and magnesium and, to determine retardation factors, were divided by those from the 0.50% S sample for weeks 60-100 in the laboratory (Lapakko et al. 2002). Sulfate release rates did not vary greatly among the various tanks, ranging from 19 to 29 μmol (kg rock week)$^{-1}$, yielding retardation factors of 0.16 to 0.25 (Table 10). Calcium release rates also fell into a fairly small range of 27 to 38 μmol (kg rock week)$^{-1}$, and appeared to be independent of limestone loading (Table 10, p. 2). Retardation factors of 4.2 to 6.0 suggested a soluble calcium phase in the rock itself, as well as the limestone added. The average Ca and CO_2 contents of the field rock were 0.39 and 0.38 percent (Table 8), 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 4.5 to 6.3 μmol (kg rock week)$^{-1}$, yielding retardation factors of 0.062 to 0.087. 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 and reporting in the 30 June 2003 report.

1. Extend data collection and interpretation through the 2002 field season.
2. Determine the chemistry of 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. More carefully measure oxygen contents within the piles and evaluate the data collected.
9. Interpret drainage quality in terms of mineral dissolution.
10. Determine the relationship between field rates of chemical release and flow.

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Table 1. Particle size distribution of rock used in field test piles bins (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	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

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	BIN 1 (0.02 %S)		BIN 2 (0.20 %S)		BIN 3 (0.39 %S)		BIN 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. 2000 and 2001 total input and output flow volumes in liters and yield coefficients.

BIN	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 %

Table 6. Page 1 of 3. Sulfate, calcium, and magnesium release rates from greenstone prediction field bins (μmol (kg rock week)$^{-1}$).

Sulfate Release					
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
Ave rate		2.13	9.02	6.12	12.4
Lab[1]	60-100	4.46	66.6[2]	120[2]	98.3
Retardation Factor[3]		0.478	0.135	0.051	0.126

[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 laboratory rates were 4.15 and 3.97.

[3] Field rate/lab rate.

Table 6. Page 2 of 3. Sulfate, calcium, and magnesium release rates from greenstone prediction field bins (μmol(kg rock week)$^{-1}$).

Calcium Release					
Date	Weeks	0.02% S	0.20% S	0.39% S	0.67% S
4/25/01	4	13.8	46.3	3.17	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	7.50	11.9
Ave rate		6.53	24.2	15.2	17.1
Lab[1]	60-100	6.79	3.32	16.1	16.8
Retardation Factor [2]		0.962	7.289	0.944	1.02

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

[2] Field rate/lab rate.

Table 6. Page 3 of 3. Sulfate, calcium, and magnesium release rates from greenstone prediction field bins (μmol(kg rock week)$^{-1}$).

Magnesium Release					
Date	Weeks	0.02% S	0.20% S	0.39% S	0.67% S
4/25/01	4	3.13	8.38	1.66	0.985
5/10/01	2	4.85	6.26	1.23	0.901
5/23/01	2	2.12	4.65	1.08	0.920
6/15/01	3	1.30	4.88	1.23	0.844
8/02/01	7	0.397	1.69	0.221	0.176
8/20/01	2	1.19	7.43	0.603	0.497
9/12/01	3	0.223	1.71	0.111	0.101
10/11/01	4	1.70	4.08	0.779	0.741
10/30/01	2	1.05	2.21	0.828	0.865
11/28/01	4	0.580	1.15	0.382	0.417
Ave rate		1.46	3.85	0.738	0.576
Lab[1]	60-100	9.68	6.83	18.0	241
Retardation Factor [2]		0.151	0.564	0.041	0.002

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

[2] Field rate/lab rate.

Table 7. Page 1 of 3. Dissolved oxygen and temperature data for field bins.

Date	0.02% Sulfur (Bin 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
12/13/01	2.0	NA	NA

Table 7. Page 2 of 3. Dissolved oxygen and temperature data for field bins.

Date	0.20% Sulfur (Bin 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
12/13/01	4.3	NA	NA	1.8	NA	NA

Date	0.39% Sulfur (Bin 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
12/13/01	4.3	NA	NA	1.7	NA	NA

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

[2] Oxygen not sampled due to obstructed tubing.

Table 7. Page 3 of 3. Dissolved oxygen and temperature data for field bins.

Date	0.67% Sulfur (Bin 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
12/13/01	3.9	NA	NA	0.9	NA	NA

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

[2] Oxygen not sampled due to obstructed tubing.

Table 8. 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 9. 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 10. 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	21.270	16.30	15.769	15.16	18.98	15.49
5/10/01	2	17.305	8.52	8.019	8.87	7.24	8.01
5/23/01	2	34.127	35.64	29.849	43.30	36.40	33.15
6/15/01	3	51.506	36.00	32.316	44.78	41.83	33.93
8/02/01	7	9.613	6.47	9.768	13.74	13.10	7.11
8/20/01	2	32.117	32.83	27.982	36.47	39.30	32.84
9/12/01	3	9.747	9.72	4.989	9.31	9.10	9.40
9/28/01	2	30.867	30.54	26.727	36.89	35.82	28.35
10/11/01	2	63.456	57.04	45.617	66.48	62.98	51.42
10/30/01	2	43.850	28.25	20.568	39.81	39.34	36.46
11/26/01	4	44.813	37.73	20.207	32.13	32.68	26.75
Average Rate		29.055	23.765	19.446	27.613	27.068	22.096

Table 10. 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	65.11	34.93	42.48	40.23	42.94	43.16
5/10/01	2	37.51	12.94	19.86	21.52	12.39	15.90
5/23/01	2	62.68	49.96	47.38	54.78	49.96	51.97
6/15/01	3	57.39	42.33	42.71	54.99	52.23	45.16
8/02/01	7	12.23	8.42	12.00	17.95	14.35	8.67
8/20/01	2	32.29	33.86	28.99	42.88	42.73	35.68
9/12/01	3	10.42	9.75	5.53	8.98	9.17	9.50
9/28/01	2	32.51	28.90	27.41	36.95	36.13	27.55
10/11/01	2	62.26	53.42	49.67	66.29	63.78	49.73
10/30/01	2	43.18	25.70	25.30	38.55	38.45	34.31
11/26/01	4	43.89	35.54	20.95	31.84	32.98	25.70
Average Rate		38.361	27.472	26.658	34.173	32.674	28.132

Table 10. 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	10.433	6.274	7.773	7.173	8.303	8.510
5/10/01	2	6.515	2.115	3.600	3.853	2.437	3.129
5/23/01	2	9.743	8.169	7.890	9.040	9.294	10.113
6/15/01	3	8.602	6.411	7.033	8.405	9.079	7.878
8/02/01	7	1.692	1.273	2.015	2.769	2.725	1.594
8/20/01	2	4.399	4.974	4.949	6.212	7.733	6.402
9/12/01	3	1.498	1.493	0.954	1.441	1.737	1.813
9/28/01	2	4.783	4.650	4.974	5.873	7.333	5.705
10/11/01	2	9.454	8.687	9.180	10.485	12.921	10.319
10/30/01	2	6.606	4.402	4.615	6.485	8.383	7.365
11/26/01	4	6.912	6.136	4.190	5.383	7.049	5.513
Average Rate		5.895	4.493	4.737	5.547	6.337	5.527

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

TANK	RATIO	INPUT FLOW VOLUME	OUTPUT FLOW VOLUME	YIELD COEFFICIENT
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 %

Figure 19. Drainage quality vs. time for the 0.02% S prediction field bin (#1).

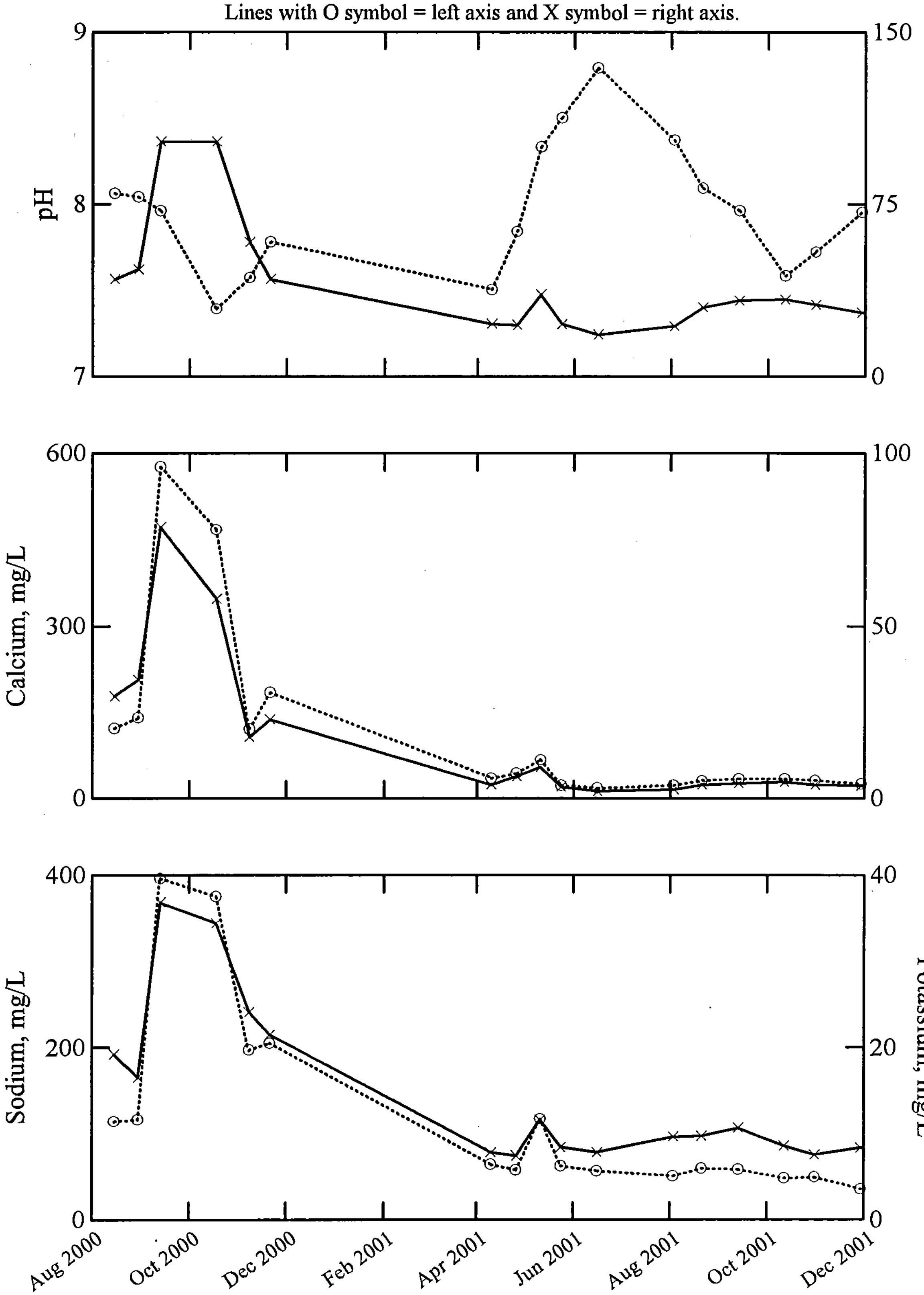


Figure 20. Drainage quality vs. time for the 0.20% S prediction field bin (#2).

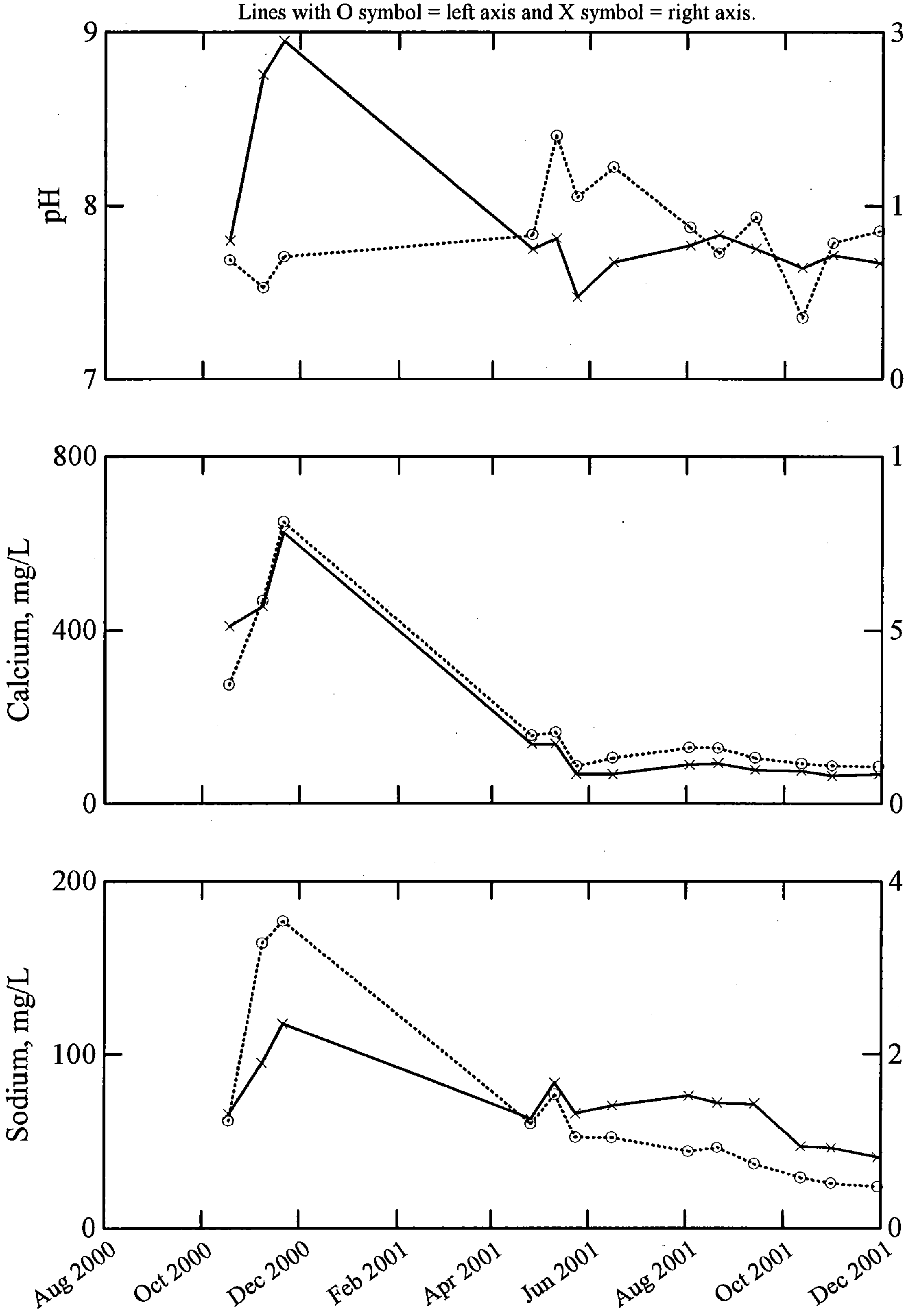


Figure 21. Drainage quality vs. time for the 0.39% S prediction field bin (#3).

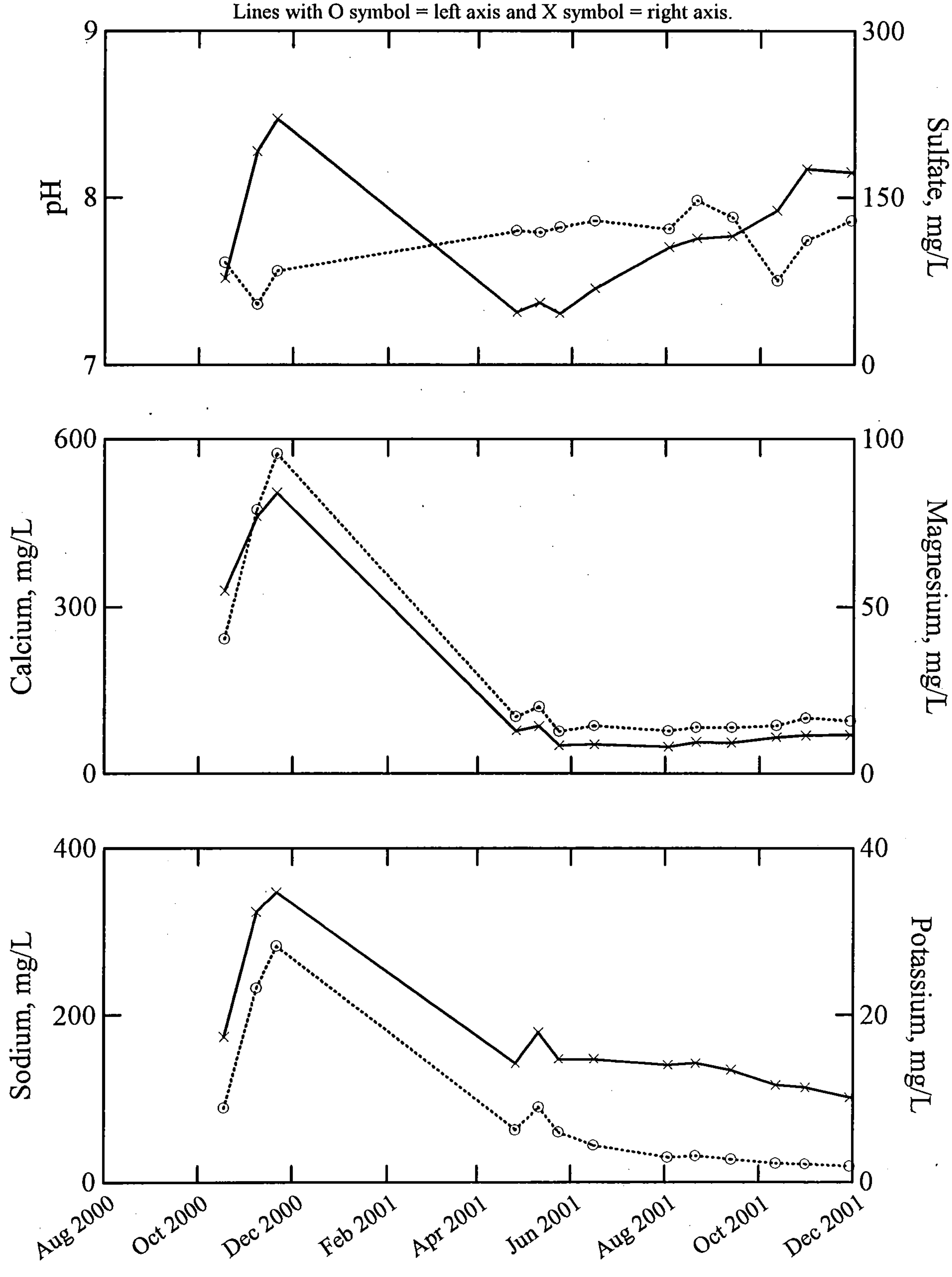


Figure 22. Drainage quality vs. time for the 0.67% S prediction field bin (#4).

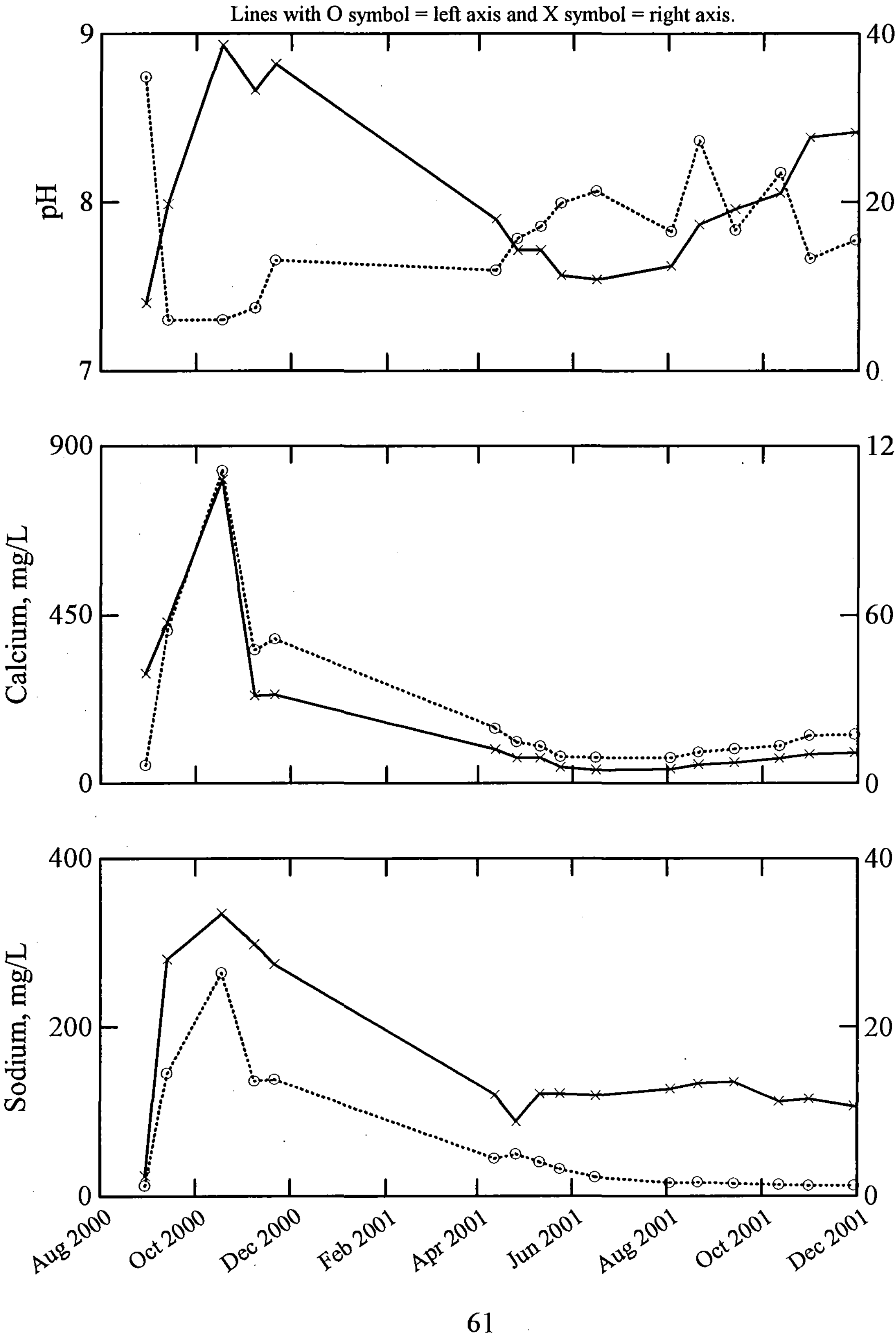


Figure 23. Drainage quality vs. time for the limestone addition control tank (#1).

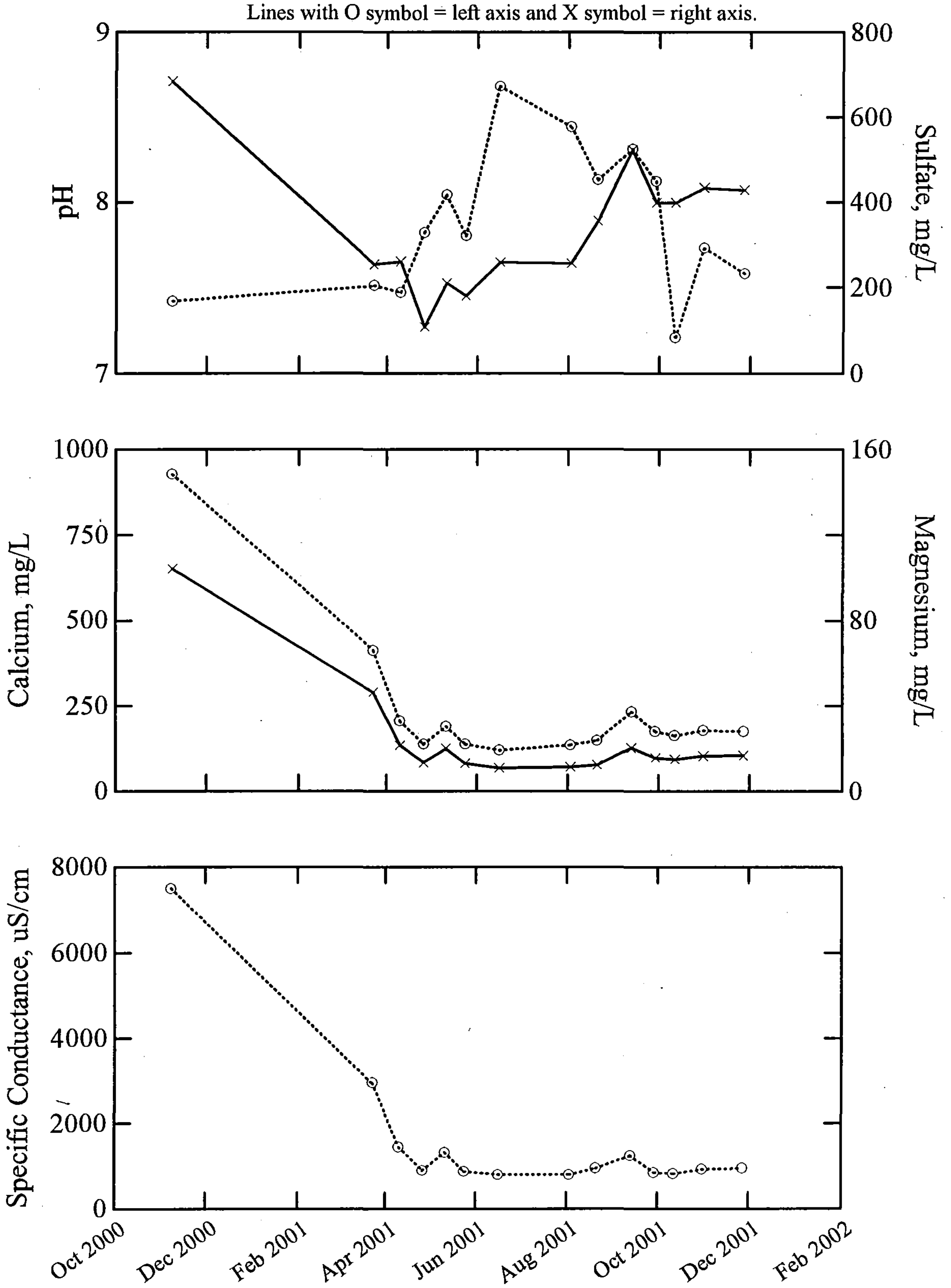
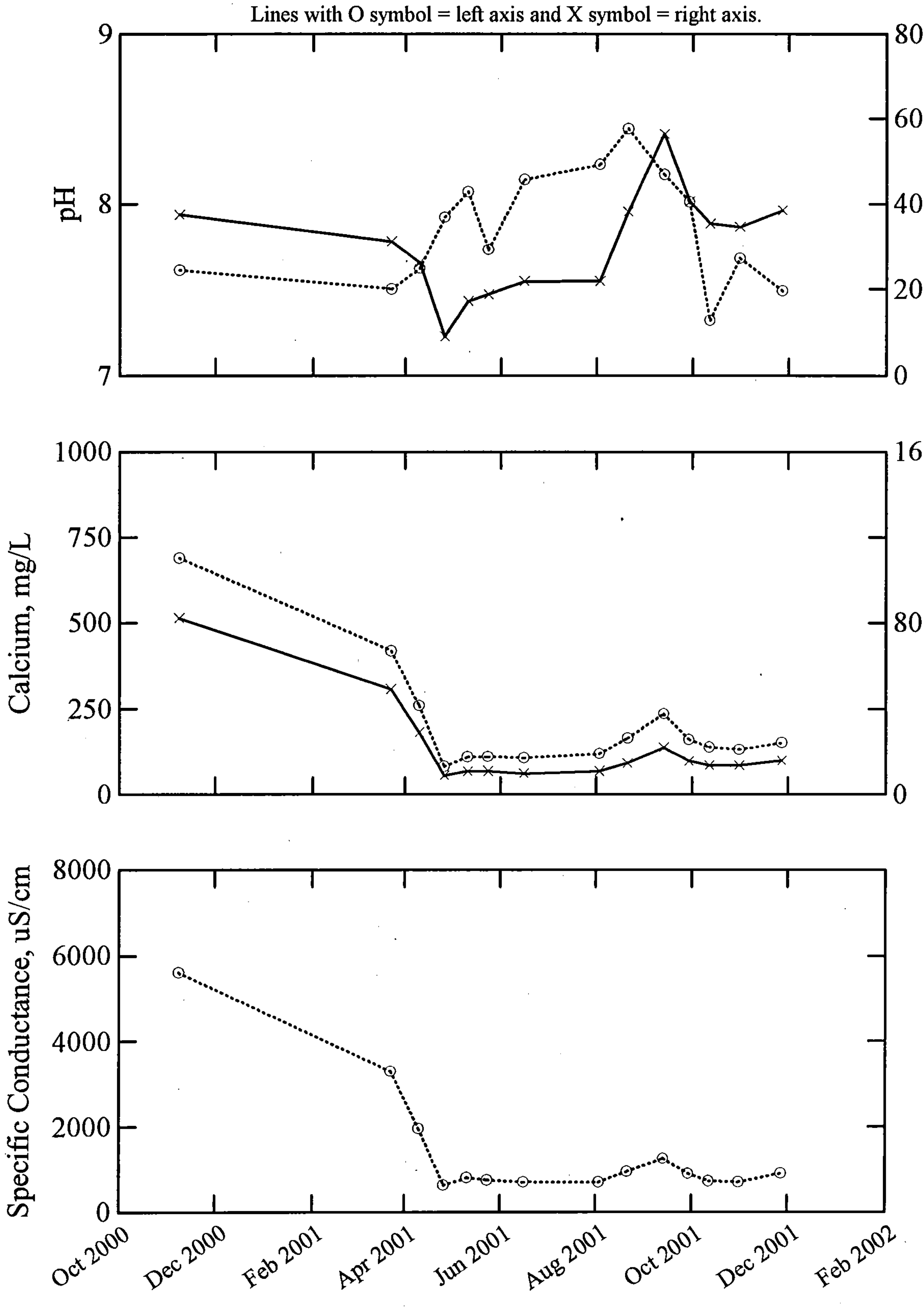


Figure 24. Drainage quality vs. time for the limestone addition control tank (#6).



Lines with O symbol = left axis and X symbol = right axis.

Figure 25. Drainage quality vs. time for the limestone addition 1:1 ratio tank (#2).

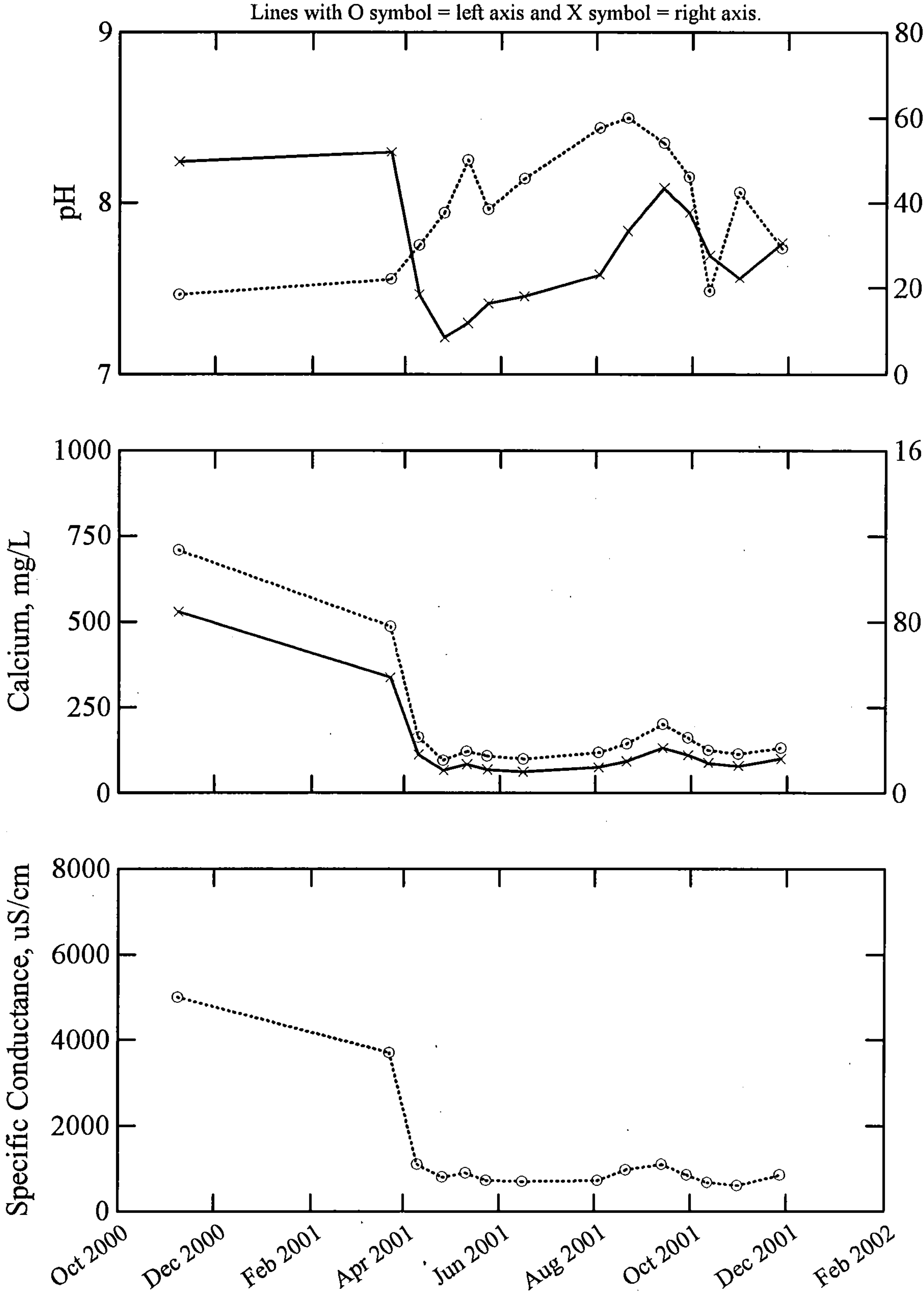


Figure 26. Drainage quality vs. time for the limestone addition 1:1 ratio tank (#5).

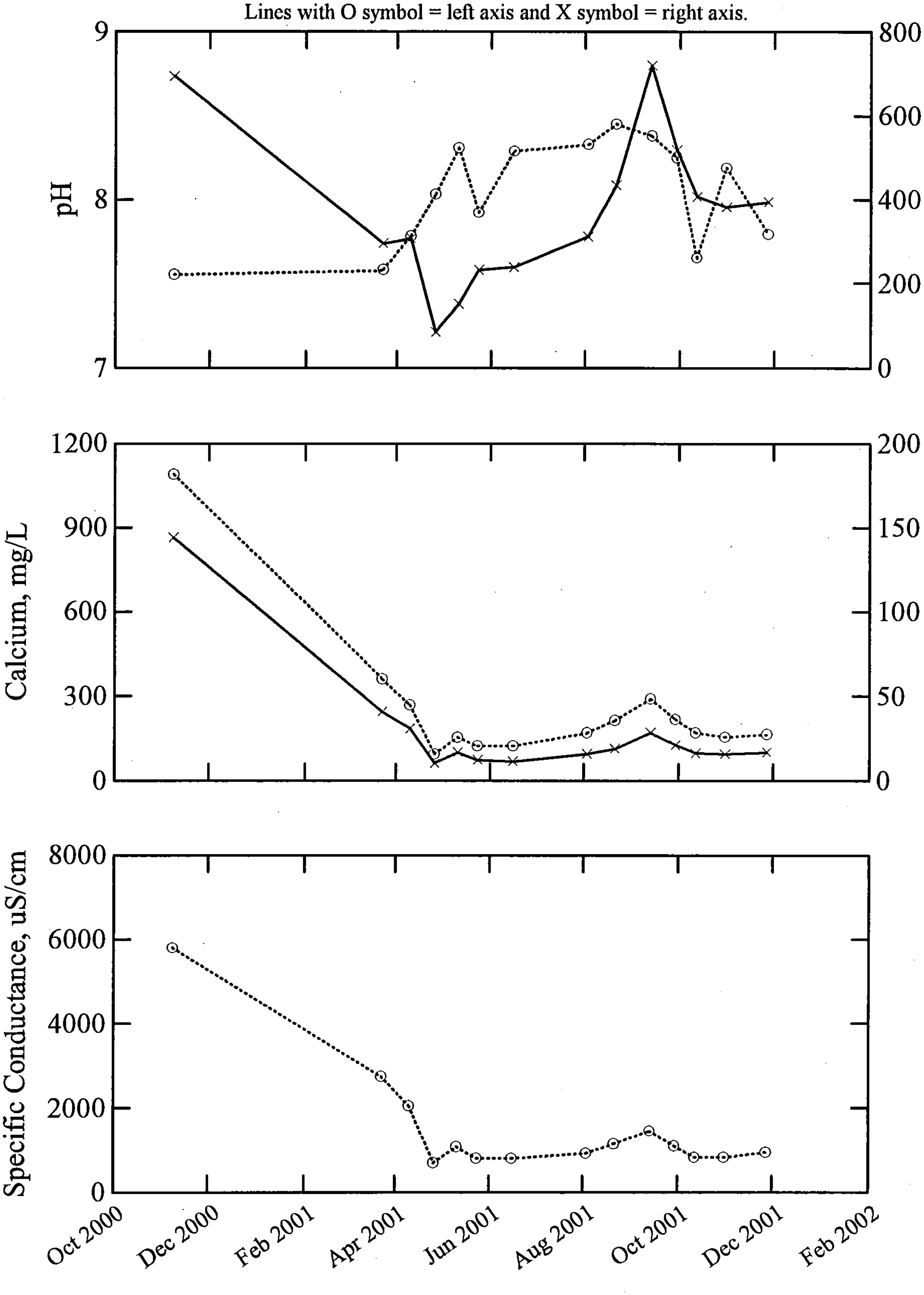


Figure 27. Drainage quality vs. time for the limestone addition 3:1 ratio tank (#3).

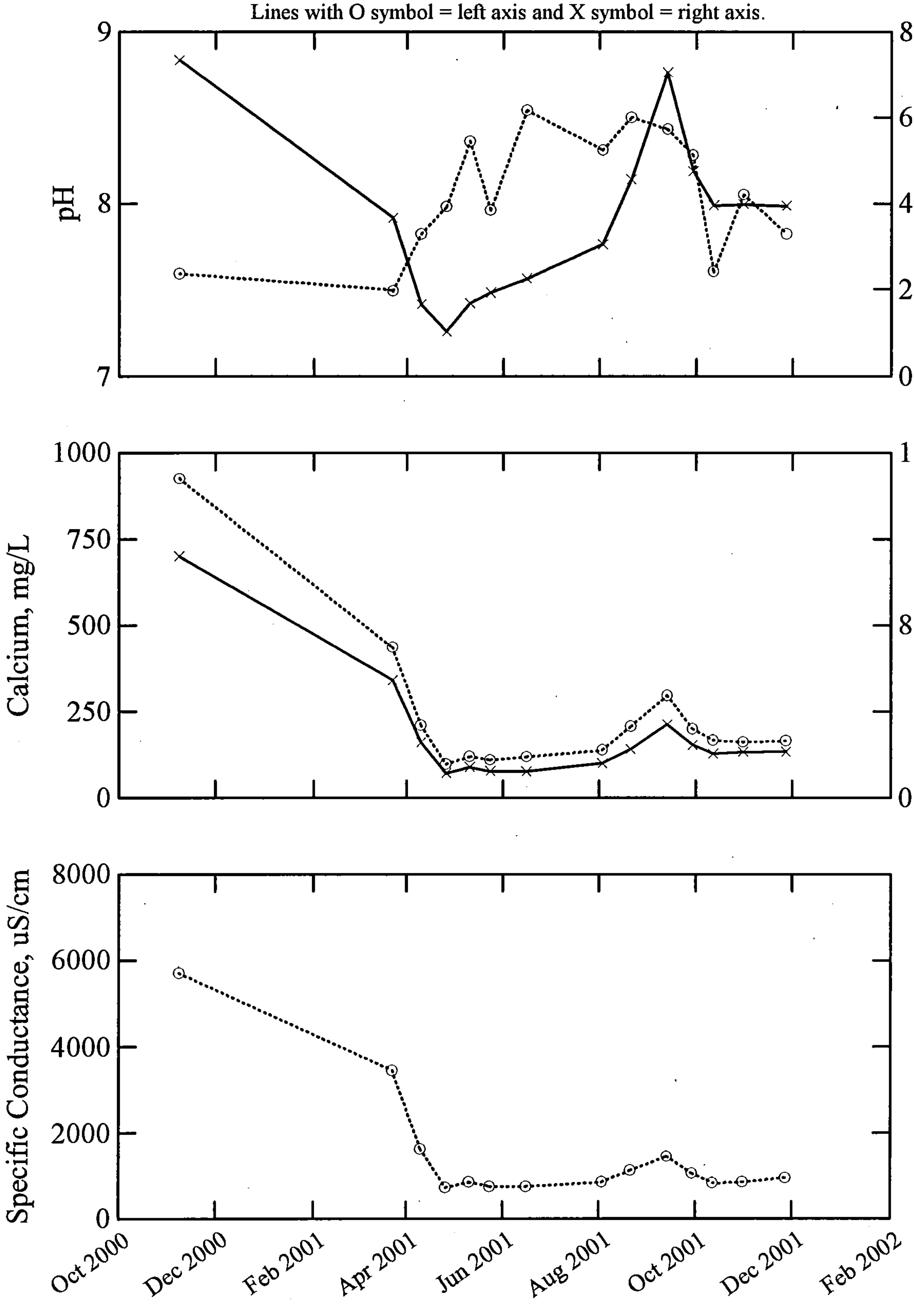
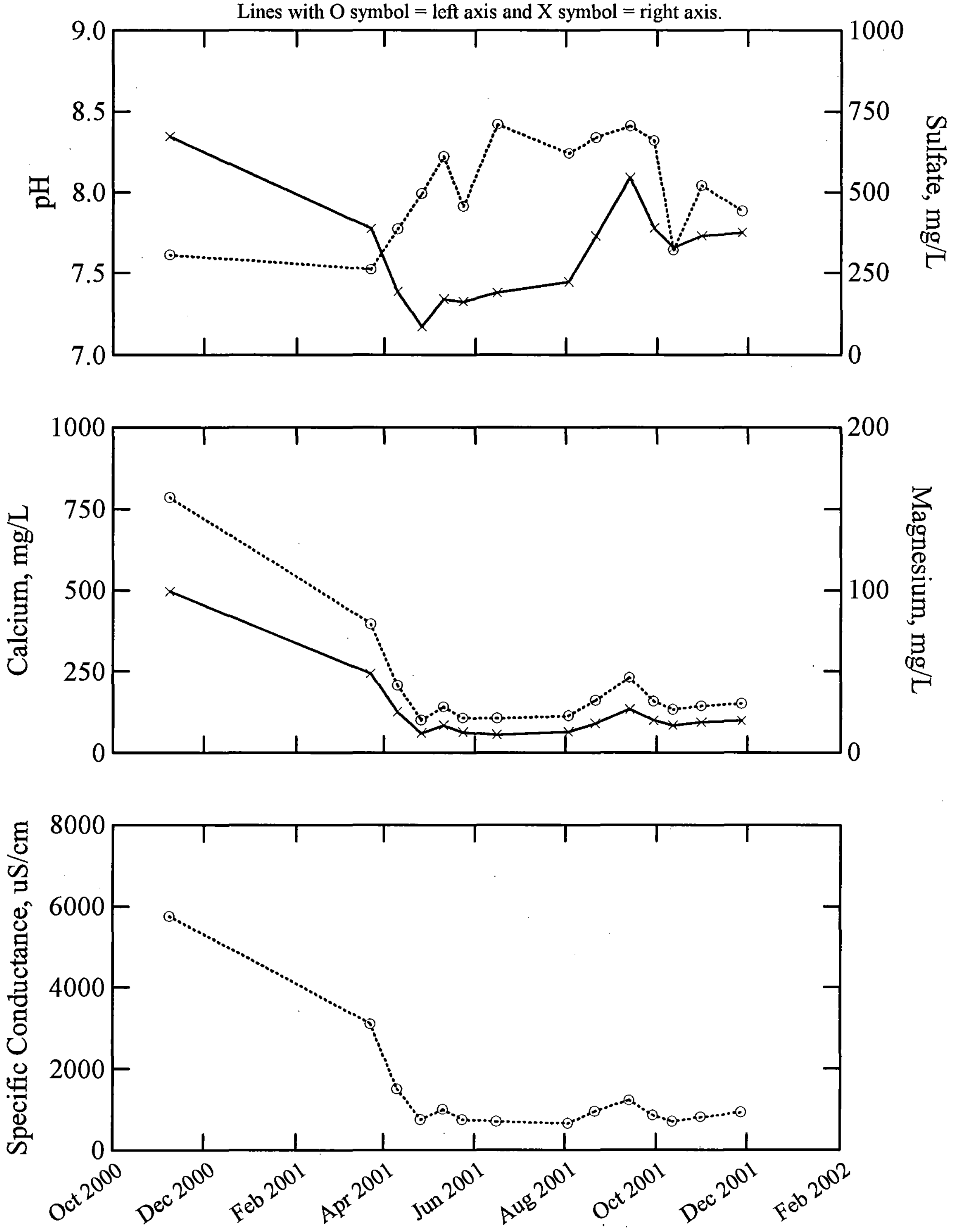


Figure 28. Drainage quality vs. time for the limestone addition 3:1 ratio tank (#4).


Lines with O symbol = left axis and X symbol = right axis.