Direct observations of aluminosilicate weathering in the hyporheic zone of an Antarctic Dry Valley stream

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Abstract—This study focused on chemical weathering and bacterial ecology in the hyporheic zone of Green Creek, a McMurdo Dry Valley (Antarctica) stream. An in situ microcosm approach was used to observe dissolution features on the basal-plane surface of muscovite mica. Four mica chips were buried in December 1999 and dug up 39 d later. Atomic force microscopy (AFM) of the basal-plane surfaces revealed small, anhedral ~10-A-deep etch pits covering ~4% of the surfaces, from which an approximate basal-plane dissolution rate of 8.3 x 10^-18 mol muscovite cm^-2 s^-1 was calculated (on the basis of the geometric surface area) for the study period. This is an integrated initial dissolution rate on a fresh surface exposed for a relatively brief period over the austral summer and should not be compared directly to other long-term field rates. The observation of weathering features on mica agrees with previous stream- and watershed-scale studies in the Dry Valleys, which have demonstrated that weathering occurs where liquid water is present, despite the cold temperatures.

AFM imaging of mica surfaces revealed biofilms including numerous small (<1 μm long), rounded, oblong bacteria. The AFM observations agreed well with X-ray photoelectron microscopy results showing increased organic C and N. Bacteriologic analysis of the hyporheic zone sediments also revealed <1-μm-long bacteria. α-Proteobacteria were observed, consistent with the oligotrophic conditions of the hyporheic zone. Nitrate-reducing bacteria were found, in agreement with a previous tracer test at Green Creek that suggested nitrate reduction occurs in the hyporheic zone. The results of this study thus provide direct evidence of dynamic geochemical and microbial processes in the hyporheic zone of a Dry Valley stream despite the extreme conditions; such processes were inferred previously from stream-scale hydrogeochemical studies. Copyright © 2002 Elsevier Science Ltd

1. INTRODUCTION

The McMurdo Dry Valleys are among the coldest and driest environments on Earth. Climatic conditions in these polar deserts are harsh, with a mean annual temperature of ~20°C and an annual precipitation of <10 cm yr^-1, all of which is snow and most of which is quickly lost to sublimation (Bromley, 1985). A large body of evidence has suggested that weathering rates tend to increase with increasing temperature, precipitation, or both (e.g., Holland, 1978; Velbel, 1993; Bluth and Kump, 1994; White and Blum, 1995; Berner et al., 1998; White et al., 1999), although a host of other factors, such as rates of physical removal, can also be important in the field (e.g., Stallard and Edmond, 1987; Bluth and Kump, 1994; Kump et al., 2000). Considering the cold, dry climate, one would expect minimal weathering rates in the Dry Valleys. Other factors, such as pH and biologic activity, also suggest minimal weathering rates. Dry Valley stream waters tend to be close to neutral pH (Green et al., 1988; McKnight et al., 1993), and dissolution rates of aluminosilicates are generally orders of magnitude slower under near-neutral conditions than at either low or high pH (e.g., Blum and Stillings, 1995). In addition to the lack of terrestrial vegetation in the Dry Valleys, the extremely dry soils (with gravimetric moisture contents typically <1%; Bockheim, 1997) have limited microbial activity (Vishniac, 1993).

Yet a growing body of geochemical evidence, including isotopic evidence, suggests that chemical weathering does occur in the Dry Valleys, where and when liquid water exists (e.g., Jones and Faure, 1978; Green and Canfield, 1984; Green et al., 1988; Lyons and Mayewski, 1993; Blum et al., 1997; Nezat et al., 2001 Gooseff et al., unpublished data). Although there is little precipitation, glacial meltwater flows through stream channels during the austral summer and interacts with unconsolidated alluvium in the hyporheic zone adjacent to each stream. The high permeability of this alluvium permits rapid exchange rates with the main channel, as compared with typical temperate streams (Runkel et al., 1998). Weathering within the hyporheic zone provides solutes to the stream. Examination of the dissolved load suggests that chemical weathering is an active process (Lyons et al., 1997; Nezat et al., 2001). Longer streams have higher solute concentrations, and solute concentrations typically increase longitudinally in streams, suggesting that exchange with the hyporheic zone introduces ions released by weathering to the stream water (Chin, 1993).

Other lines of evidence also indicate that chemical weathering does indeed occur in the Dry Valleys. First, although Claridge and Campbell (1977) showed that salts are prevalent in the Dry Valleys, hydrogeochemical modeling by Lyons et al. (1997) revealed that the stream chemistry cannot be explained by salt dissolution alone. Second, the ratios of K:Cl and Ca:Cl in lakes suggest a continental source from weathering, rather than a marine-salt source (Lyons et al., 1998); minor elements...
also suggest a weathering source. Third, Sr isotopic studies by Jones and Faure (1978) demonstrated crustal rather than marine-salt or aerosol sources for Dry Valley streams and lakes. More recently, W. B. Lyons et al. (pers. commun.) showed that \(^{87}\text{Sr}/^{86}\text{Sr}\) ratios of Dry Valley streams are consistent with the weathering of various intrusive rocks. Finally, Gibbs plots (Gibbs, 1970) of stream chemistry suggest that chemical weathering and atmospheric precipitation are both contributors to the dissolved loads of the streams (Lyons et al., 1997).

Microbiologic processes also may play important roles in weathering despite the cold temperatures, oligotrophic conditions, and limited duration of exposure to liquid water. Many forms of algae, fungi, and bacteria occur in microbial mats on the streambeds of Dry Valley streams (e.g., Broady, 1982; Vincent et al., 1993; McKnight et al., 1998). These persist in a freeze-dried state when there is no stream flow and begin growing as soon as flow begins (Vincent and Howard-Williams, 1986, 1989). The microbial community in hyporheic zone sediments has not been examined previously, and it is not known whether microbial biofilms in the hyporheic zone sediments persist through the winter or are regenerated when sediments thaw and flow begins in the austral summer. In streams, studies have generally found that abraded biofilms on the streambed are regenerated over 1 to 2 weeks (Allan, 1995). Thus, development of new biofilms would not be expected to greatly constrain microbial processes in the hyporheic zone or the role that they may play in weathering.

Organic acids may also enhance weathering rates. The biologic activity of the microbial mats on the streambed results in a flux of dissolved organic carbon (DOC) to the stream water. This biologic activity is reflected in the DOC concentrations of streams. Downes et al. (1986) reported DOC concentrations from 2 to 6 mg C L\(^{-1}\) in Canada Stream in the Lake Fryxell basin. Aiken et al. (1996) found that DOC concentrations of streams feeding Lake Fryxell varied from 0.2 to 9.7 mg C L\(^{-1}\) with higher values in streams with more abundant algal mats. Chemical characterization of this DOC showed that humic substances comprised only \(\sim 10\%\). This suggests that most of the DOC is fresh leachate of the microbial mats and represents labile substrates to support biofilms in the hyporheic zone. Both studies (Downes et al., 1986; Aiken et al., 1996) showed that DOC concentrations were higher at the start of summer, when algal mats and the streambed were first wetted.

In research reported herein, we applied an in situ microcosm approach to observe dissolution features and to directly measure initial weathering rates of muscovite mica in the hyporheic zone of Green Creek, a first-order stream that drains the tongue of the Canada Glacier and discharges into Lake Fryxell in Taylor Valley. Freshly cleaved pieces of muscovite mica were buried in reaction vessels for \(~6\) weeks, after which they were dug up and returned to the laboratory for surface chemical and microtopographic analysis by atomic force microscopy (AFM) and X-ray photoelectron spectroscopy (XPS). We chose to use muscovite mica because it can be freshly cleaved to produce relatively flat basal-plane surfaces such that even subtle weathering features formed over a short reaction time can be discerned. This was crucial because environmental considerations in the Dry Valleys precluded a long-term (multiyear) experiment. In addition, we characterized the bacterial community within the hyporheic zone by means of a variety of approaches.

As discussed below, results of this study confirmed that aluminosilicate weathering occurs in the hyporheic zone in the austral summer and that there is substantial microbial colonization of mineral surfaces. The study results also shed light on the role of the hyporheic zone in the N cycle in the Dry Valleys.

2. EXPERIMENTAL

2.1. Description of the Study Site

The McMurdo Dry Valleys are a group of so-called desert oases clustered near the coast of Antarctica in the general region of McMurdo Sound. In addition to the cold, dry climate, strong katabatic winds produce a desiccating effect, enhance erosion, and are the agents of sediment transport. Green et al. (1993) review the local geology as related to the lake geochemistry. Basement geology includes schist, hornfels, and marble metasediments; granite intrusions; and lamprophyre and granitic dykes. These basement rocks are unconformably overlain by the Beacon sandstones, with dolerite sills cutting through both.

Dry Valley streams carry glacial meltwater. Given that permafrost begins only \(~0.5\) to \(~1\) m below the soil, there is no shallow groundwater system available to contribute base flow to the stream. Despite the lack of a shallow groundwater system, stream water interacts with alluvium in the hyporheic zone. This is a zone adjacent to and underneath the stream where water flows in a downstream direction, interacting with water in the main channel. Runkel et al. (1998) showed that the high permeability of the alluvium results in rapid exchange rates with the main stream channel.

Stream waters in the Dry Valleys tend to be relatively dilute, with near-neutral pH (Green et al., 1988). By use of the terminology of Gibbs (1970), Green et al. (1988) showed that Dry Valley stream composition was consistent with influence by atmospheric precipitation (marine aerosols) and rock weathering reactions (or salts derived from rock weathering) rather than by evaporation and crystallization.

Taylor Valley extends from the Polar Plateau to the Ross Sea. The valley is bordered on the north by the Asgard Range and on the south by the Kukri Hills. The study site (Fig. 1) is located along Green Creek (Fig. 2), which drains the Canada Glacier and flows a distance of 1.2 km along unconsolidated alluvium into Lake Fryxell. Bockheim (1997) characterized this alluvium as mostly sand-sized particles interbedded with large cobbles and boulders consisting primary of gneiss, diorite, and schist. From 1990 to 1995, the annual streamflow of Green Creek varied from 28 to 330 × 10\(^3\) m\(^3\), and the estimated contribution to Lake...
direct observations of aluminosilicate weathering

Fryxell ranged from 7 to 18% of total streamflow into the lake (Conovitz et al., 1998). The primary source of water in the stream is glacial melt, which begins in late November to mid-December and ends in mid-January to early February (Conovitz et al., 1998). Hence, the stream typically only flows from 4 to 10 weeks, at most, during the austral summer.

At the headwaters of Green Creek, meltwater runs off the glacier into a series of ponds. Stream water then proceeds through a broad, flat area strewn with cobbles and small boulders. The stream then follows a more distinct channel, with a shallow slope, for ~200 m. The creek becomes steep for ~100 m. The Green Creek stream gauge is located in this region. Our experiment was located 5 m upstream from the stream gauge, on the left-hand bank (Fig. 1).

2.2. Previous Investigations at Green Creek

Green Creek has been the subject of ongoing hydrobiogeochemical investigations by McKnight and colleagues since 1990. In December 1990, a stream gauge was installed in Green Creek with continuous monitoring of streamflow and conductivity during the period of flow. In January 1994, as part of the McMurdo Dry Valleys Long-Term Ecological Research Project, a network of 16 stream ecosystem monitoring sites was established in the McMurdo Dry Valleys, including establishment of a permanent transect and sampling of algal mats and water quality at Green Creek. The distribution of algal mats was mapped (Alger et al., 1997). Most of Green Creek was found to have a wide (5 to 10 m) flat streambed covered with extensive orange algal mats; black mats dominated by Nostoc spp. exist along the margins.

Average major ion and nutrient concentrations at the outlet of Green Creek, based on data from 1993 to 1997, are provided in Table 1. Water quality data from the hyporheic zone, January 2000, are listed in Table 2. As shown in these tables, Green Creek water tends to be dilute, with near-neutral pH. The average pH for Green Creek surface-water samples collected during the 1999 to 2000 sampling period was 7.6 (n = 28, SD = 0.5). The average pH for hyporheic waters samples was 7.7 (n = 28, SD = 0.6).

2.3. In Situ Microcosms

Mineral-containing reaction vessels (i.e., in situ microcosms) were introduced into the hyporheic zone 5 m upstream from the gauging station at Green Creek to monitor changes in mineral surface microtopography and surface composition as related to chemical weathering. A similar approach has been used to monitor weathering at various temperate zone locations (e.g., Bennett et al., 1996; Nugent et al., 1998).

Muscovite mica was chosen as the reacting mineral on the bases of several experimental considerations: (1) it can be easily cleaved to produce fresh basal-plane surfaces; (2) the resulting surfaces are flat and hence do not require polishing that could disturb surface structure, microtopography, composition, and hence reactivity; (3) it has long been considered to be a standard for AFM imaging; and (4) previous laboratory-based AFM and XPS studies of muscovite weathering (e.g., Johnson et al., 1992) provide background for comparison. Several geochemical considerations must be brought to bear as well: (5) muscovite micas are present in the Dry Valley sediments, and Claridge and Campbell (1977) concluded that weathering of muscovite was an important source of leachable K, whereas Blum et al. (1997) found that K increased with Si in the downstream direction in a nearby Dry Valley stream; and (6) muscovite is a common aluminosilicate mineral with surface structure similar to that of many 2:1 clays, although with different surface charge properties. Because we could only bury the reaction vessels for a single austral summer, we needed to use a mineral with excellent cleavage so that even subtle weathering features would be readily discernible on the exposed surface.

Six-millimeter-scale mica samples (chips) of a single ruby mica (Fe-bearing muscovite) specimen from the Kent State University Mineralogical collection were freshly cleaved as described by Namjesnik-Dejanovic and Maurice (1997). These six chips were all taken from a single section of the specimen (i.e., we cut one small cleavage sheet into six pieces). Five samples were then placed inside individual pouches within plastic containers for transport to the field, the sixth sample was similarly packed and kept at Kent State as a blank. Reaction vessels consisted of acid-washed (in 10% HNO3) amber 15-mL high density polyethylene (HDPE) bottles with ~1-mm-diameter holes punched through the vessel’s sides and bottom. The reaction vessels were installed at two sites close to Green Creek sampling transect 3, wells A (GCT3WA, 2 m from the stream) and B (GCT3WB, 4 m from the stream) (Fig. 1). These sites were known to have possible hyporheic solution exchange with the stream, as shown by a tracer test conducted the previous summer. On the day of installation, December 14, 1999, the stream flow was very low (Fig. 3). Two GCT3WA

Table 1. Average major ion and nutrient concentrations in surface water at the outlet of Green Creek from 1993 to 1997.*

<table>
<thead>
<tr>
<th>Conductivity (µS/cm)</th>
<th>pH</th>
<th>Ca (mg/L)</th>
<th>Mg (mg/L)</th>
<th>Na (mg/L)</th>
<th>K (mg/L)</th>
<th>Cl (mg/L)</th>
<th>SO4 (mg/L)</th>
<th>NO3 (mM)</th>
<th>PO4 (µM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>47.67</td>
<td>7.57</td>
<td>5.93</td>
<td>0.86</td>
<td>2.99</td>
<td>0.91</td>
<td>4.32</td>
<td>1.55</td>
<td>0.44</td>
<td>0.13</td>
</tr>
</tbody>
</table>

* From McKnight et al. (2002).

Table 2. Major ion concentrations in hyporheic zone water in January 2000.*

<table>
<thead>
<tr>
<th>Sample</th>
<th>Conductivity (µS/cm)</th>
<th>pH</th>
<th>Ca (mg/L)</th>
<th>Mg (mg/L)</th>
<th>Na (mg/L)</th>
<th>K (mg/L)</th>
<th>Cl (mg/L)</th>
<th>SO4 (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GCT3WA 1/7/00</td>
<td>80</td>
<td>7.68</td>
<td>10.46</td>
<td>1.59</td>
<td>4.64</td>
<td>2.25</td>
<td>5.05</td>
<td>2.46</td>
</tr>
<tr>
<td>GCT3WA 1/22/00</td>
<td>218</td>
<td>NM</td>
<td>6.89</td>
<td>1.12</td>
<td>25.61</td>
<td>3.07</td>
<td>42.12</td>
<td>2.54</td>
</tr>
<tr>
<td>GCT3WB 1/22/00</td>
<td>208</td>
<td>8.65</td>
<td>19.37</td>
<td>4.21</td>
<td>22.24</td>
<td>4.70</td>
<td>28.61</td>
<td>8.65</td>
</tr>
</tbody>
</table>

*Sampling locations shown in Figure 1. NM = not measurable.
vessels, each containing one mica chip, were placed at a depth of 16.5 cm, and two GCT3WB vessels, each containing one mica chip, were placed at a depth of 19.5 cm. The fifth mica chip was kept in its original pouch as a control. Installation depths were dictated by the depth to permafrost. One reaction vessel at each location was gently filled with sediment from the site; the other vessel was not. Because of the fairly large diameter of the holes, some sediment eventually entered the other vessels. The reaction vessels were tied to a stake before backfilling so that they could be easily retrieved later. Samples of sediment collected while digging the holes at each site were placed in clean plastic bags for microbiologic and mineralogic analysis. All samples that they could be easily retrieved later. Samples of sediment collected while digging the holes at each site were placed in clean plastic bags for microbiologic and mineralogic analysis. All samples were analyzed in ultrahigh vacuum. Data acquisition times were highly variable; the low concentrations of some components on the sample surface, minimizing lateral frictional forces during scanning. This decreases the probability of damage of soft and easily deformable materials. However, TMAFM does not provide atomic-scale resolution.

Height, amplitude, and phase-mode TMAFM images were collected simultaneously. Height mode images provide data in three dimensions and were used for all measurements of topographic relief (z-direction) reported here. Amplitude-mode images are similar, but they provide essentially the first derivative of the height—hence accentuating edges in the amplitude mode images. Widths of features were measured on amplitude-mode images. In phase imaging, the phase lag of the cantilever oscillation, relative to the signal sent to the cantilever driver, is monitored (Magonov et al., 1997; http://www.dl.com). The phase lag is highly sensitive to variations in material properties such as adhesion and viscoelasticity. Forsythe et al. (1998) showed that phase imaging can separately highlight organic (bacteria and exopolysaccharide attachment features) vs. inorganic (mineralogic) components of biofilms. Several AFM images were taken of each sample, after which they were analyzed by XPS, then again by AFM. AFM images were analyzed by the computer program ENVI (Environment for Visualizing Images; Research Systems) to determine the percentages of features at various heights or depths. This proved invaluable for estimation of dissolution rates.

### 2.5. XPS Analysis

After initial AFM analysis of all mica samples, they were analyzed by X-ray photoelectron spectroscopy (XPS). XPS is a surface-sensitive analytical technique used for evaluation of elemental and chemical state information on solid samples. The sampling depth is to ~10 nm. Samples are analyzed in ultrahigh vacuum. Data acquisition times were highly variable; the low concentrations of some components on the sample surface, minimizing lateral frictional forces during scanning. This decreases the probability of damage of soft and easily deformable materials. However, TMAFM does not provide atomic-scale resolution.

Height, amplitude, and phase-mode TMAFM images were collected simultaneously. Height mode images provide data in three dimensions and were used for all measurements of topographic relief (z-direction) reported here. Amplitude-mode images are similar, but they provide essentially the first derivative of the height—hence accentuating edges in the amplitude mode images. Widths of features were measured on amplitude-mode images. In phase imaging, the phase lag of the cantilever oscillation, relative to the signal sent to the cantilever driver, is monitored (Magonov et al., 1997; http://www.dl.com). The phase lag is highly sensitive to variations in material properties such as adhesion and viscoelasticity. Forsythe et al. (1998) showed that phase imaging can separately highlight organic (bacteria and exopolysaccharide attachment features) vs. inorganic (mineralogic) components of biofilms. Several AFM images were taken of each sample, after which they were analyzed by XPS, then again by AFM. AFM images were analyzed by the computer program ENVI (Environment for Visualizing Images; Research Systems) to determine the percentages of features at various heights or depths. This proved invaluable for estimation of dissolution rates.

### 2.6. Bacteriology Methods

Sediment samples for bacterial cultivation (5 to 9 g dry weight) were mixed with 6 mL of 0.1% sodium pyrophosphate and sonicated for 5 min (Branson 2210) to dislodge bacteria. From each sample, four plates each of two types of media (standard methods agar [SMA] and R2A) were inoculated. SMA (BBL), also known as plate count agar, is used for the isolation of bacteria from water and other sources (Atlas, 1993) and contains casein, yeast extract, and glucose. R2A (BBL) is used to enumerate bacteria in water samples (Atlas 1993) and contains yeast extract, casein, glucose, starch, pyruvate, and other compounds. Sample remaining after plate inoculation was preserved with 0.2% formalin, and the number of bacteria was determined by epifluorescent microscopy after staining with 4',6-diamidino-2-phenylindole (Porter and Feig, 1980).

Two plates of each type from each sample were incubated at 23°C, and two other plates of each type from each sample were incubated at 4°C. Colony-forming units (CFU) of bacteria were enumerated after

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### Table 3. Summary of mica sample handling.

<table>
<thead>
<tr>
<th>Mica chip</th>
<th>Location</th>
<th>Burial depth (ft)</th>
<th>With sediment?</th>
<th>DI rinsed?</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>GCT3WA</td>
<td>0.55</td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td>2</td>
<td>GCT3WB</td>
<td>0.65</td>
<td>No</td>
<td>No</td>
</tr>
<tr>
<td>3</td>
<td>GCT3WA</td>
<td>0.55</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>4</td>
<td>GCT3WB</td>
<td>0.65</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>5</td>
<td>Controla</td>
<td>No</td>
<td>No</td>
<td>No</td>
</tr>
</tbody>
</table>

*aThe control was transported but not buried.*
48 h for the 23°C plates and for the 4°C plates, after 10 and 40 d. From each sample, colonies with different morphologies were streaked for isolation. Isolates were frozen at −70°C to preserve their features and subjected to various standard tests.

Isolates were stained with Gram stain, and cell morphology and Gram stain responses were noted. Isolates were subjected to the oxidase (which detects the presence of cytochrome c) and the catalase (which detects catalase, which breaks down hydrogen peroxide) test. Nitrate reduction was also examined. Organic compound use was examined by testing for starch hydrolysis and protein degradation (based on gelatin liquefaction). Fermentation of sucrose, lactose, and glucose was also determined. Isolates were also identified with the aide of API Rapid NFT test strips (BioMerieux) following the manufacturer’s instructions.

Bacterial isolates were also examined by fluorescent in situ hybridization (FISH); in this method, fluorochrome-labeled, taxon-specific oligonucleotide probes are hybridized to rRNA inside intact bacterial cells. Briefly, cultures grown in nutrient broth were mixed with 0.5X phosphate-buffered saline (7.6 g NaCl, 1.9 g Na₂HPO₄):8% paraformaldehyde and then subjected to FISH following the method of Lemke et al. (1997). A probe specific for the α-Proteobacteria, a major group of aquatic bacteria developed by Manz et al. (1992), was used; the hybridization and washing temperature was 54°C. Positive and negative controls consisted of α-proteobacteria and members of other proteobacteria groups from the American Type Culture Collection.

3. RESULTS

3.1. Results of Physical Measurements at Green Creek

Stream water flow rate, water temperature, and specific conductance (sc) were measured continuously at 15-min intervals at the Green Creek stream gauge (Fig. 1) throughout the 6-week flow season. Figure 3 illustrates the hydrograph and sc measurements. The hydrograph shown in Figure 3 is typical for Dry Valley streams, showing intermittent high flows interspersed with more moderate flow and some periods of no measurable flow. 1999 to 2000 was a low to moderate flow year compared with the previous seven flow seasons of data. The sc profile for the season is also typical of Dry Valley streams. High values at the beginning of the flow season mark the flushing out of atmospherically deposited salts and other material that may have been deposited in the streambed by winter winds or left over from the previous season. Once this flush is completed, lower sc values prevail throughout the remaining majority of the flow season.

Stream water temperatures measured at the gauging station were generally in the single digits (°C), reaching as high as −10°C in late December 1999 (Fig. 4). Subsurface waters in the hyporheic zone were considerably colder, never rising to more than 2°C at either measuring location (Fig. 4). Subsurface-water temperatures in the hyporheic zone fluctuate primarily in response to solar heating of the sediment surfaces. Interactions with exchanging stream water also influence temperature. Subsurface-water temperatures at GCT3WB were generally higher than at GCT3WA, and the maximum temperature was also slightly higher (2°C at GCT3WB vs. 1.5°C at GCT3WA).

3.2. Results of XPS Analysis

Results of XPS analysis of mica samples are presented in Table 4 and Figure 5. Results of the surface elemental analysis of the blank (mica 5) showed a deviation from the 1:1 Al:Si stoichiometry expected for a pure mica (KAl₂(Si₃O₁₀)(OH)₂).

![Figure 5](image)
micas, whereas Na:Si showed no clear trend. Relatively high Ca is characteristic of many Dry Valley streams, and Green et al. (1988) determined that CaCO₃ was the only simple salt that would form in Dry Valley lakes. They further noted that calcite was common in Dry Valley soils.

Three of the four mica samples showed little or no change in Al:Si, but mica 1 showed a slight depletion in Al. A number of processes such as mineral dissolution, secondary phase formation, and adhering particles can all contribute to Al:Si ratios on weathered surfaces (e.g., Johnsson et al., 1992; Nugent et al., 1998). Hence, surface Al:Si is not always a clear indicator of any specific weathering processes. Fe:Si ratios decreased slightly for all of the hyporheic zone–reacted samples, relative to the blanks. This is important because Fe is a nutrient that tends to be immobile in near-neutral pH conditions. Thus, the mica surface could potentially serve as a source of Fe for microorganisms. Kalinowski et al. (2000) and Maurice et al. (2001) showed that some species of bacteria can remove Fe from aluminosilicates. For the Fe and Al XPS data, both of which showed at most only subtle changes, it is possible that accumulation of C at the surface affected the elemental ratios if the bacteria or organic C contain different surface ratios of Fe or Al to Si. For example, Buss et al. (in press) demonstrated that removing bacteria by several different methods either slightly decreased or did not change the surface Fe/Si ratios measured by XPS on bacteria-reacted mineral substrates. We chose not to attempt to remove bacteria from our field-reacted samples because of the potential for disturbing surface composition or structure.

A trace of P was detected on one reacted mica chip, and N was observed on most of the hyporheic zone–reacted samples. Peak details suggested an organic nitrogen rather than, for example, a nitrate. Neither P nor N were detected on unreacted blanks. We did not conduct any high-resolution scans for Mg.

### 3.3. Results of AFM Imaging

AFM imaging of unreacted mica blanks showed surfaces typical for cleaved, unreacted mica (e.g., Johnsson et al., 1992): mostly flat (at the nanometer scale) surfaces with occasional adhering particles and cleavage steps. The adhering particles were generally in the range of tens to hundreds of nanometers. We did not observe any major changes in surface microtopog-

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**Table 4. Elemental concentrations (% atomic) on mica samples determined by XPS.**

<table>
<thead>
<tr>
<th>Mica sample</th>
<th>Al</th>
<th>Ca</th>
<th>C</th>
<th>Cl</th>
<th>F</th>
<th>Fe</th>
<th>N</th>
<th>O</th>
<th>K</th>
<th>Si</th>
<th>Na</th>
<th>P</th>
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<tbody>
<tr>
<td>1</td>
<td>11.0</td>
<td>0.34</td>
<td>18.3</td>
<td>BD</td>
<td>1.1</td>
<td>1.4</td>
<td>0.39</td>
<td>50.3</td>
<td>3.3</td>
<td>13.5</td>
<td>0.40</td>
<td>BD</td>
</tr>
<tr>
<td>2</td>
<td>11.5</td>
<td>0.26</td>
<td>17.1</td>
<td>BD</td>
<td>1.1</td>
<td>1.3</td>
<td>0.42</td>
<td>51.0</td>
<td>3.2</td>
<td>13.9</td>
<td>0.36</td>
<td>BD</td>
</tr>
<tr>
<td>3</td>
<td>11.0</td>
<td>0.73</td>
<td>20.1</td>
<td>0.25</td>
<td>1.1</td>
<td>1.3</td>
<td>0.17</td>
<td>49.2</td>
<td>3.3</td>
<td>12.1</td>
<td>0.62</td>
<td>BD</td>
</tr>
<tr>
<td>4</td>
<td>11.5</td>
<td>0.54</td>
<td>18.0</td>
<td>0.11</td>
<td>1.2</td>
<td>1.3</td>
<td>0.25</td>
<td>50.4</td>
<td>3.5</td>
<td>12.4</td>
<td>0.72</td>
<td>BD</td>
</tr>
<tr>
<td>5</td>
<td>11.9</td>
<td>0.34</td>
<td>12.9</td>
<td>BD</td>
<td>1.2</td>
<td>1.5</td>
<td>0.27</td>
<td>54.6</td>
<td>3.5</td>
<td>13.3</td>
<td>0.56</td>
<td>BD</td>
</tr>
</tbody>
</table>

*Each sample was analyzed in two 300 × 700–μm regions; results for each of these two regions are presented separately. Micas 1 to 4 were reacted in the hyporheic zone; mica 5 was an unreacted blank. BD = below detection.*
raphy after XPS analysis of the samples, although the density of ultrafine particles increased slightly.

In addition to cleavage steps and small particulates, several other surface microtopographic features were observed on samples buried in the hyporheic zone; examples of salient features are shown in Figures 6 to 9. (1) Numerous clusters of shallow pits that formed on muscovite surfaces reacted in the hyporheic zone. The pits are ~10 Å deep and are thus about the depth of one tetrahedral–octahedral–tetrahedral structural layer in muscovite. Several pits are pointed out with arrows.

Fig. 6. TMAFM height mode image in air of small, anhedral pits that formed on muscovite surfaces reacted in the hyporheic zone. The pits are ~10 Å deep and are thus about the depth of one tetrahedral–octahedral–tetrahedral structural layer in muscovite. Several pits are pointed out with arrows.

Fig. 7. TMAFM height mode image of mica 1 showing blocky particles that are likely either precipitated salts, adhering particles from the hyporheic zone, or both.

In addition to cleavage steps and small particulates, several other surface microtopographic features were observed on samples buried in the hyporheic zone; examples of salient features are shown in Figures 6 to 9. (1) Numerous clusters of shallow pits that formed on muscovite surfaces reacted in the hyporheic zone. The pits are ~10 Å deep and are thus about the depth of one tetrahedral–octahedral–tetrahedral structural layer in muscovite. Several pits are pointed out with arrows.

Fig. 8. (a) TMAFM amplitude mode image of mica 4 showing a single oblong-shaped bacterium (~300 nm long) on the surface of hyporheic zone–reacted mica. The size and shape of this bacterium matches those observed in hyporheic zone sediments by epifluorescence microscopy. (b) TMAFM phase mode image of biofilm on muscovite, with several bacteria (~400 nm long) highlighted in the box. The top edge of the film is highly linear, which suggests that it is running along the edge of a step (lower left to upper right) on the mica surface.

(~10 Å deep), flat-bottomed, anhedral pits (Fig. 6) appeared, similar to those described by Johnsson et al. (1992) after mica dissolution in deionized water. (2) In some locations on the mica surfaces, large irregular or blocky particles were observed. They were likely either mineral precipitates, including salts, or adhering particles from the hyporheic sediments (Fig. 7).
Surfaces contained extensive films that were easily deformed by the AFM tip and that showed strong phase contrast and therefore were likely organic in nature (Figs. 8 and 9). The structures shown in Figure 9 were similar morphologically to adsorbed humic substances imaged previously in our laboratory, with pores indicative of film rupture (Namjesnik-Dejanovic and Maurice, 1997). However, morphologic similarity is not sufficient to identify this material. Small (several hundred nanometers long), oblong bacteria were observed in many locations on sample surfaces (Figs. 8 and 9). These bacteria often were associated with organic films; hence, extensive biofilms were present in some areas. There were no noticeable differences between mica 2, which was not rinsed with deionized water, and the other three hyporheic zone samples. The bacteria often were observed lined up along step edges on the mica surface (images not shown).

Fig. 9. (a) TMAFM height mode image of mica 1 showing a thin film present along the left side of the image. A portion of this film, to the right of ~2.0 mm and above ~1.4 mm, was partially eroded by a prior smaller area scan. The film is likely an organic deposit, perhaps of humic substances. The circular structures are likely caused by film rupture on drying, as commonly observed when humic substances dry on a mica surface (Namjesnik-Dejanovic and Maurice, 1997). The full z-range scale from black to white is 20 nm for this image. Horizontal streaks are an imaging artifact. (b) Phase-mode image taken simultaneously with (a). The organic film shows different phase contrast than the underlying mica surface, consistent with different viscoelastic properties—that is, the organic material is softer and “stickier.” This is also consistent with the ease by which it was eroded during small-region scans. (c) Amplitude mode image taken simultaneously with (a) and (b).
3.4. Results of Microbiologic Analysis

In agreement with AFM imaging of reacted micas, bacteriologic analysis of the hyporheic zone sediments revealed small (<1 μm long) bacteria. Because the samples were not preserved in the field, the microscopic examination was limited. The total number of bacteria was higher at site GCT3WA (the site nearest the stream) than at site GCT3WB; however, the numbers were similar on the two collection dates (Table 5). Because these samples were not preserved in the field but only on arrival at Kent State University, we do not know how the values changed after sample collection.

The number of CFU was consistently higher at site GCT3WA (near stream) than site GCT3WB (further from stream), regardless of media type or incubation temperature. At both sites, CFU were considerably higher in December than in January. This is despite the fact that the December sampling was at the beginning of the season, when temperatures were just beginning to warm. December daily average air temperatures measured at a meteorological station located ~1 km from the experimental site were higher (~1.6°C) than those for January (~−2.84°C) during the course of this experiment. We do not have water temperatures for the period in December directly leading up to the sampling day (December 14, 1999), so we cannot compare specific changes in water temperature. The differences in CFU likely result from changes in nutrient concentrations. As discussed above, DOC concentrations are generally higher at the start of the austral summer and decrease thereafter (Downes et al., 1986; Aiken et al., 1996).

All of the bacteria were gram negative (Table 6). Optical microscopy showed rounded, rod-shaped cells several hundred nanometers (<1 μm) in length. The oxidase test was used to distinguish among groups of bacteria on the basis of cytochrome oxidase activity (i.e., the ability to produce oxidase enzymes). Oxidase-positive organisms include most pseudomonads, which have a complete electron transport chain based on the presence of the final cytochrome oxidase. Oxidase-negative organisms include some environmental bacteria, such as Acinetobacter. More oxidase-positive isolates were obtained in December, compared with January, at both sites. As expected for gram-negative bacteria, the bacteria at both sites were 100% catalase positive from both sampling rounds. Nitrate reduction is a test to determine whether bacteria can use nitrate as a terminal electron acceptor. Nitrate reduction was common for all samples but was greater at the far site (GCT3WB) than the near site (GCT3WA) and was also greater in January than in December. In terms of organic compound use, starch hydrolysis was uncommon. Sucrose, glucose, and lactose fermentation occurred for many isolates, but the fermentation ability was rather weak compared with typical fermenters.

Examination of bacteria via FISH using an α-Proteobacteria probe revealed that ~46% of the bacterial isolates hybridized the probe, suggesting that they are in the α-Proteobacteria group. This group of bacteria contains many oligotrophs. Also, some bacteria were identified as Pseudomonas or Sphingomonas on the basis of API test strip results.

4. DISCUSSION

4.1. Chemical Weathering in the Hyporheic Zone

Development of numerous small, shallow, anhedral etch pits with a depth appropriate for the unit cell of muscovite (~10 Å) indicated that dissolution occurred during weathering in the hyporheic zone. The pit morphology was similar to that observed by Johnsson et al. (1992) after muscovite dissolution in deionized water at room temperature. Because AFM provides three-dimensional data, we could calculate the dissolution rate over the 39-d microcosm experiment. On the basis of measurements from 10 of the highest-quality AFM images, we calculated that etch pits covered an average of ~4% of the reacted mica surfaces. By using an etch pit depth of 10 Å, we therefore estimated a dissolution rate of 8.3 \times 10^{-18} \text{ mol muscovite cm}^{-2} \text{ s}^{-1} on the basis of geometric surface area. This rate should be considered to be an approximation because of the small sampling size. Moreover, it is an integrated rate over the study period (austral summer) and should be considered to be an initial rate because of the relatively short duration of the field experiment. If the dissolution is stoichiometric, then the Si dissolution rate would be 2.5 \times 10^{-17} \text{ mol cm}^{-2} \text{ s}^{-1}; non-stoichiometric dissolution could lead to somewhat slower or faster Si rates.

A number of researchers (Nickel, 1973, in Kalinowski and Schweda, 1996; Clemen and Lin, 1981; Lin and Clemency, 1981a,b; Acker and Bricker, 1992; Kalinowski and Schweda, 1996; Taylor et al., 2000) have investigated mica (muscovite, phlogopite, biotite) weathering rates in the laboratory at or around 22 to 25°C. Experiments most similar to our conditions in terms of pH and mica mineralogy (i.e., muscovite) were...
those of Nickel (1973, in Kalinowski and Schweda, 1996) and Lin and Clemency (1981a). Muscovite dissolution data of Nickel (1973, in Kalinowski and Schweda, 1996) provide a dissolution rate of 5.0 × 10⁻¹⁰ mol cm⁻² s⁻¹, assessed on the basis of Si release, and of 1.0 × 10⁻¹⁹ mol cm⁻² s⁻¹, assessed on the basis of Al release, in deionized water at pH 6 at room temperature. Lin and Clemency (1981a) measured a dissolution rate of ruby mica (Fe-bearing muscovite) at pH ~5 and room temperature of 2.4 × 10⁻¹⁷ mol muscovite cm⁻² s⁻¹.

Although our field rate appears consistent with these laboratory rates, one must be cautious not to compare the rates directly. First, the laboratory rates were for entire particles, whereas our rate was for the basal-plane surface alone, which is probably less reactive than edge sites. Second, our surface area was calculated on the basis of geometry as determined by the relatively newly developed AFM and is not equivalent to BET surface area (i.e., surface area by gas adsorption, Brunauer et al., 1938). Differences in methods of surface area measurement may translate to several orders of magnitude greater dissolution rates when normalized to geometric surface area than when normalized to BET surface area (Brantley et al., 1999). Third, our rate is at temperatures considerably below typical room temperature, and aluminosilicate dissolution rates tend to increase with increasing temperature. Fourth, our rate is based on direct observation of changes in surface microtopography rather than on inference from solution chemistry.

Hochella et al. (1999) used AFM to measure dissolution rates in deionized water of etch pits on a pretreated phlogopite basal plane. They measured a pit dissolution rate of 5.1 ± 2.1 × 10⁻¹⁴ mol cm⁻² s⁻¹. This rate is several orders of magnitude faster than the rate we measured, but it is specific to highly reactive sites.

Several researchers have investigated rates of biotite mica weathering in the field. Biotite has long been known to dissolve faster than muscovite (e.g., Birkeland, 1974). As reported in Murphy et al. (1998), determinations of field dissolution rates of biotite range from 10⁻¹⁹ to 10⁻¹⁷ mol biotite cm⁻² s⁻¹. This range includes geogenic field dissolution rates on the order of 10⁻¹⁸ to 10⁻¹⁷ mol biotite cm⁻² s⁻¹ (Velbel, 1985; Swoboda-Colberg and Drever, 1993). Our initial basal-plane geometric muscovite dissolution rate for the hyporheic zone in the austral summer is within this range. However, the rates are not directly comparable because of the short duration of our experiment. It must be remembered that our rate is only for the hyporheic zone and only for the portion of the year when liquid water is present.

The Antarctic dissolution rates can be compared with a previous in situ study of feldspar dissolution at a temperate zone site. Such a comparison is useful because both studies used AFM to calculate weathering rate. Nugent et al. (1998) calculated a feldspar dissolution rate of between 10⁻¹⁶ and 10⁻¹⁸ mol feldspar cm⁻² s⁻¹ on the basis of AFM images of feldspars buried in a State College, Pennsylvania, USA, spo- dosol for 3 yr. Considering that feldspar is likely to dissolve more rapidly than muscovite mica (e.g., Birkeland, 1974), and that sites on the basal-plane surfaces of micas are likely less reactive than are edge sites, our results are consistent with relatively rapid initial dissolution rates of mica in the hyporheic zone during the austral summer.

XPS results showed a decrease in the surface Al:Si ratio for one of the reacted mica surfaces, as well as a slight decrease in the Fe:Si ratio for all of the reacted micas. These results are suggestive of dissolution, and they are thus in agreement with the observation of etch pits.

The weathering rate calculated in this study represents the capacity of Dry Valley stream systems for initial weathering of an aluminosilicate mineral at two particular points in one stream. The hyporheic zone of any stream is inherently heterogeneous, and the initial weathering rates calculated in these experiments may not be directly applicable to other portions of the hyporheic zone along Green Creek or other Dry Valley streams. However, several previous studies have concluded that primary silicate weathering is an important process in Dry Valley streams on the basis of analysis of long-term records of water chemistry for many Dry Valley streams (Lyons et al., 1997) and on analyses of longitudinal increases in solutes in one Dry Valley stream (Gooseff et al., unpublished data). Gooseff et al. (unpublished data) computed Si denudation rates on the order of 10⁻¹⁵ to 10⁻¹⁶ mol Si cm⁻² s⁻¹ of sediment (geometric) surface area for the hyporheic zone of a Dry Valley stream. Our mica dissolution rate is one to two orders of magnitude slower, most likely because mica is one of the most unreactive of aluminosilicate phases and because sites on the basal-plane surface are less reactive than edge sites. Our study directly demonstrated that chemical weathering may occur in the hyporheic zone during the limited period of streamflow in one austral summer. Gooseff et al. (unpublished data) also observed that the rates of hyporheic exchange act as another major control on the extent to which this weathering is observed in the stream. We hypothesize that the reported relatively high weathering rates in the hyporheic zone of Dry Valley streams (Lyons et al., 1997; Nezat et al., 2001; Gooseff

Table 6. Microbiologic characterization of isolates from the Green Creek hyporheic zone sediments.

<table>
<thead>
<tr>
<th>Feature</th>
<th>GCT3WA 12/14/1999</th>
<th>GCT3WB 12/14/1999</th>
<th>GCT3WA 01/22/2000</th>
<th>GCT3WB 01/22/2000</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total number of isolates</td>
<td>9 (100%)</td>
<td>12 (100%)</td>
<td>7 (100%)</td>
<td>15 (100%)</td>
</tr>
<tr>
<td>Cell morphology</td>
<td>Rods (100%)</td>
<td>Rods (100%)</td>
<td>Rods (100%)</td>
<td>Rods (100%)</td>
</tr>
<tr>
<td>Appropriate cell length</td>
<td>&lt;0.5 μm</td>
<td>&lt;0.5 μm</td>
<td>&lt;0.5 μm</td>
<td>&lt;0.5 μm</td>
</tr>
<tr>
<td>Gram reaction</td>
<td>100% −</td>
<td>100% −</td>
<td>100% −</td>
<td>100% −</td>
</tr>
<tr>
<td>Oxidase</td>
<td>78% +</td>
<td>58% +</td>
<td>43% +</td>
<td>47% +</td>
</tr>
<tr>
<td>Catalase</td>
<td>100% +</td>
<td>100% +</td>
<td>100% +</td>
<td>100% +</td>
</tr>
<tr>
<td>Nitrate reduction</td>
<td>55% +</td>
<td>67% +</td>
<td>86% +</td>
<td>93% +</td>
</tr>
<tr>
<td>Starch hydrolysis</td>
<td>100% −</td>
<td>92% −</td>
<td>86% −</td>
<td>93% −</td>
</tr>
<tr>
<td>Sucrose fermentation</td>
<td>67% +</td>
<td>83% +</td>
<td>43% +</td>
<td>53% +</td>
</tr>
<tr>
<td>Lactose fermentation</td>
<td>67% +</td>
<td>50% +</td>
<td>43% +</td>
<td>40% +</td>
</tr>
<tr>
<td>Glucose fermentation</td>
<td>67% +</td>
<td>50% +</td>
<td>29% +</td>
<td>40% +</td>
</tr>
</tbody>
</table>
et al., unpublished data, data herein) are likely related to the abundant supply of fresh, unweathered surfaces exposed to dilute water that flows and exchanges rapidly with water in the stream channel.

Research has shown that bacteria can significantly affect the rates of aluminosilicate dissolution, thus influencing chemical weathering processes and elemental cycling (e.g., Ehrlich, 1981, 1996; Hiebert and Bennett, 1992; Vandevivere et al., 1994; Welch and Vandevivere, 1994; Bennett et al., 1996; Ullman et al., 1996; Barker et al., 1998; Barker and Banfield, 1998; Rogers et al., 1998; Liermann et al., 2000; Maurice et al., 2001). Although we observed bacteria and organic films on the mica surfaces after reaction in the hyporheic zone, we did not notice any increase in dissolution pit density in the vicinity of the bacteria. This may be misleading, though, because some microorganisms were likely lost from the surface during rinsing and transport, and we also could not image directly beneath attached bacteria and biofilms. Hence, although the extensive and quite rapid surface colonization suggests that the bacteria may be utilizing nutrients in or on the muscovite surface, the role or roles of bacteria and biofilms in the dissolution and weathering reactions remains to be determined. It is also possible that the bacteria merely used the mica surface as a physical substrate for growth. Observation of decreased Fe:Si ratios on the reacted mica surfaces suggests that the bacteria could potentially be depleting Fe from the mica, as Fe is an important nutrient. Clearly, further studies of microbe–mineral interactions in Dry Valley hyporheic sediments are needed.

4.2. Nitrate-Reducing Bacteria: Implications for the N Cycle

The detection of nitrate-reducing bacteria has important implications for N cycling in the Dry Valleys and agrees well with results of a prior study at Green Creek. McKnight et al. (2002) investigated hyporheic zone exchange and inorganic nitrogen and phosphorus dynamics at Green Creek in late January 1995. They injected lithium chloride, sodium nitrate, and potassium phosphate into the creek. Variations of Cl over time demonstrated that substantial exchange of surface water with the hyporheic zone occurred at low flow conditions of <3 L s⁻¹. Microbial communities associated with the algal mats in the streambed were found to have significant nitrate and phosphorus uptake abilities. Moreover, production of nitrite and ammonium at sites located several hundred meters downstream from the injection site indicated that dissimilatory nitrate reduction was occurring. McKnight et al. (2002) estimated that hyporheic nitrate uptake, which represented the loss due to assimilatory and dissimilatory nitrate reduction, was 7 to 16% of the total uptake. They also suggested that microbial processes of nitrate reduction were likely important in the hyporheic zone. Our XPS-based observation of N accumulation at the mica surface, along with our AFM images showing organic adsorption, bacteria, and biofilms and our identification of nitrate-reducing bacteria in hyporheic zone sediments, thus confirm the interpretation of McKnight et al. of the tracer experiment. The activity of these bacteria and the exchange of hyporheic zone water with stream water thus can be expected to play an important role in the N flux to Lake Fryxell. Moreover, we observed more nitrate-reducing bacterial percentages at site GCT3WB, which is further removed from the stream. Water within the hyporheic zone at this site would have been in the hyporheic zone longer and the exchange at the surface is less. These conditions would be conducive to greater oxygen depletion in microzones and to increased potential for nitrate reduction.

5. CONCLUSIONS

Our observation of dissolution features on the basal-plane surfaces of mica upon weathering in the hyporheic zone is consistent with the findings of previous studies, which indicated that aluminosilicate dissolution occurs in the Dry Valleys despite the cold temperatures, scant precipitation, and lack of terrestrial vegetation. We suggest that the combination of largely unweathered and highly reactive hyporheic zone sediments, with relatively dilute glacial meltwaters and high exchange rates between the stream and the hyporheic zone, likely results in fairly rapid initial dissolution rates analogous to the rapid rates commonly observed (e.g., Rimstidt and Dove, 1986, and references therein) at the beginning of laboratory-based dissolution experiments. Our observed rates are specific to the hyporheic zone during the austral summer and cannot be extrapolated to the surrounding (dry) soils or to the full year. Analysis of bacterial ecology revealed an abundance of fairly small bacteria that rapidly formed biofilms on mineral surfaces. Overall, our results demonstrate that the hyporheic zone of a Dry Valley stream is a hydrobiogeochemically active location during the austral summer.

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