ABSTRACT

Title of Thesis:FATE AND TRANSPORT OF NITROGEN AT A DEEP ROWBIOSOLIDS APPLICATION HYBRID POPLAR TREE FARM

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This study focused on a gravel mine reclamation site using biosolids in deep rows as a nutrient source and hybrid poplar trees as the stabilizing crop. Biosolids application rates of 481, 962, and 1443 dry Mg/ha and tree densities of 0, 716, and 1074 trees/ha and controls (0 dry Mg/ha – 0 trees/ha) were studied. Total nitrogen, ammonium, nitrite and nitrate in soil water samples from pan and suction lysimeters under and around the biosolids rows were evaluated. Total nitrogen was predominantly in the form of ammonium. Ammonium concentrations in more than half the samples were above 100 mg/L, reflecting the average biosolids concentration of 2,300 mg/kg. No significant differences ($\alpha = 0.05$) were determined between application rates or tree densities, but ammonium concentrations significantly decreased with distance below the biosolids row. Nitrite and nitrate nitrogen concentrations were predominantly non-detects or less than 1 mg/L, indicating that nitrification was not occurring.

TRANSPORT AND FATE OF NITROGEN AT A DEEP ROW BIOSOLIDS APPLICATION HYBRID POPLAR TREE FARM

by

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Chapter 1: Introduction

Since the advent of civilization, with increasing populations living in fixed locations, disposal and treatment of household waste has been a necessity of life. Domestic wastewater systems evolved from more rudimentary flushing systems that discharged raw waste directly into waterways to the more sophisticated wastewater treatment plants in use today. In current systems, raw sewage enters the facility; is treated through physical, chemical, and biological processes to meet regulatory requirements; and exits in two forms: 1) as effluent and 2) as sewage sludge (a.k.a., biosolids). Effluent is effectively integrated back to the environment via discharge into waterways, or in some cases by ground injection. Sewage sludge, however, poses a greater integration challenge that in many cases proves costly. It is therefore of interest to develop safe, effective, and economical means of sewage sludge disposal.

Current United States regulations for disposal are delineated in The Standards for Use of Disposal of Sewage Sludge (Title 40 of the Code of Regulations{CFR} Part 503). In addition to incineration, landfilling, and composting, these Environmental Protection Agency (EPA) regulations allow for land application of biosolids, and strongly encourage implementation of this technique for beneficial uses. Most beneficial uses consist of land application to agricultural fields and other nutrient-deficient lands to enhance growth of vegetation. In such cases, application must follow the protocols in 40 CFR part 503 to ensure that excess nutrients are not transported to surface water or leached to ground water.

Biosolids utilization in forest lands, particularly in silviculture operations, has gained increased popularity in the United States. Surface spraying, spreading and

subsurface mixing in the soil are the primary distribution techniques, with applications required each year or multiple times a year to successfully meet the nutrient needs of the trees and production goals of the operation. Because trees are not a food crop, concerns related to the potential uptake and ingestion of biosolids contaminants do not exist. Not only do the biosolids provide a nutrient source for the trees, they also build up the topsoil, reduce erosion and increase above and under ground ecosystem diversity.

An alternative land application regimen, referred to as deep row application, has been in use on private property owned and managed by the Environmental Reclamation Company, Inc. (ERCO, Inc.) since the early 1980s. This technique was established on an abandoned surface gravel mine that, prior to reclamation as a tree farm, consisted of a sand and gravel overburden underlain by a clay layer. As such, it was devoid of organic matter and subject to erosion. In concert with regulatory requirements to reclaim abandoned mine sites, ERCO devised a reclamation plan to grow hybrid poplar trees over trenches that had been filled with biosolids. The biosolids would serve as a long-term nutrient source for the fast-growing, nutrient-demanding poplars. The poplars, in turn, would provide erosion control, wildlife habitat, and potentially become a marketable product.

Deep row application has several advantages over traditional land application techniques. With deep row application, the biosolids are encased in the mine spoils such that odor from and vector attraction to the sludge is controlled. In addition, this set up hinders nitrogen volatilization and prevents biosolids runoff during storm events. The biosolids and tree root remnants from the 6-year tree cycle improve the overall quality of the soil and set the stage for more permanent ecological reclamation. The combined

advantages allow for a once-per-cycle application of biosolids at a higher rate than traditional at-surface application techniques. This decreases labor costs and allows for disposal of a larger amount of biosolids.

Of critical importance when establishing this operation was the assurance that the application of biosolids would not pose a threat to the environment. Biosolids contain nutrients that, although essential to the production of healthy crops, pose an environmental and health risk if they are applied in excess and, as they decompose to more soluble forms, leach to the groundwater or surface water. In addition, biosolids are known to contain several metals that, if concentrated, can also pose a health risk if introduced to groundwater aquifers. EPA's 503 rule allows surface application provided the biosolids contain no more than the allowed concentrations of certain metals and provided that cumulative loading does not exceed criteria. Water quality monitoring at this site was therefore a key component of permit requirements to ensure the project was environmentally sound. To address this issue, seven groundwater monitoring wells ranging in depth from 11-36.5 m (35-120 ft) were installed around the perimeter of the 36.5 ha farmed site between 1982 and 1990. Over 15 years of groundwater monitoring for nutrients, metals and biological parameters show negligible levels of pollutants.

Although the groundwater, surface water, and soil sample analyses demonstrated that the deep-row application protocols were environmentally sound, it did not provide enough detail about the mechanisms by which the nutrients in the biosolids, particularly nitrogen, were being utilized. It was clear that nitrogen had not infiltrated the groundwater flow represented by the monitoring wells, but the specific dynamics in close proximity to the biosolids rows were only theorized, and did not have quantitative data to

support such theories. To better understand the interactions amongst the soil, biosolids, and trees would require closer investigation of the local ecosystem in and around the deep rows, at 1-2 m (3-6ft) depths in the soil profile as opposed to the 11-36 m (35-120 ft) depths represented by the groundwater monitoring wells. Such information would help to 1) determine the optimal rate at which biosolids can be applied to promote the most effective poplar growth without generating excess nutrients and 2) establish the feasibility of applying this technique to other gravel spoils with the ultimate goal of 3) providing an alternate and better technique to recycle human waste.

As stated above, better understanding the fate of nitrogen will more readily provide for application of this technique to other gravel sites with similar characteristics. This is of particular importance in the Baltimore-Washington area, which produces approximately 188,000 dry Mg (207,000 dry tons) of biosolids each year and has over 2230 ha (5,500 acres) of land permitted for sand and gravel mining (Kays et al., 1999). This tree farming technique could therefore prove to be a viable solution for the reclamation of mines in concert with biosolids disposal, with the added bonus of enhancing carbon sequestration in the trees and producing a marketable wood crop.

To date, studies at the ERCO tree farm have indicated that this deep-row application technique is a favorable alternative for biosolids recycling. Tree growth, however, has not proved optimal, with diameter sizes being less than anticipated after the 6-9 year growth cycle. This sub optimal growth is the combined effect of nitrogen deficiencies and excessive tree densities. Consequently, the current study was planned to evaluate the following factors: 1) hybrid poplar planting at lower densities (to promote increased tree diameters) and 2) several biosolids application rates at levels comparable

to and higher than the standard procedure in use at the ERCO tree farm. These conditions may increase chances for nutrient leaching into the soil should the timing and amount of nutrient release exceed the poplar tree uptake rate and microbial immobilization activity. Counteracting this concern, however, is the stipulation that conditions within the deep row provide a wet anaerobic environment that facilitates denitrification, eliminating the potential for percolation though the soil. In addition, because this site is protected by a natural layer (or layers) of clay soil, vertical water flow from the trenches, along with the accompanying excess nitrates, would be impeded. This rationale must, however, be clearly and consistently demonstrated.

The focus of this study is to evaluate nitrogen fate and transport occurring in close proximity to the biosolids rows, with particular emphasis on the fate of nitrate, a soluble form of nitrogen linked to both health and environmental concerns. Although phosphorus dynamics as well as tree production and associated nutrient content is also a focus of the overall experiment, these parameters are beyond the scope of this master's thesis.

Chapter 2: Review of Literature

Documented records regarding the utilization of sewage sludge as fertilizer dates back to the 1500s in Germany, where sewage was used on croplands. Under the Federal Water Pollution Control Acts of 1972, land application of sewage sludge was recognized as a protocol for disposal, provided the disposal was managed in accordance with the applicable regulations. In conjunction with this recognition, experts from the EPA, United States Department of Agriculture (USDA), and National Land Grant Universities pooled their resources to form a Coordinating Committee on Environmental Quality that developed a subcommittee on Recycling Efforts of Sludges on Land. This subcommittee evaluated research that had been conducted on the pros and cons of sewage sludge application to provide guidance on the most appropriate protocols for use. This increased interest, along with the ongoing buildup of sewage sludge at wastewater treatment plants, sparked a series of research projects that evaluated the impacts of sewage sludge application to land (Lue-Hing, et al., 1992).

The Nitrogen Cycle

In order to understand the implications of sewage sludge disposal techniques and associated scientific studies, the nitrogen cycle must be understood. Nitrogen is one of the most important nutrients for plant growth. Only certain water-soluble inorganic forms, including ammonium (NH_4^+) and nitrate (NO_3^-), can be absorbed by higher plants. In sewage sludge, the treatment process determines the ratio of organic to inorganic forms of nitrogen. Liquid anaerobically digested sludge may contain a majority of nitrogen in the form of ammonium, with lesser amounts as organic nitrogen and negligible amounts of nitrate (EPA, 1994; Kelley, et al. 1984). In undigested lime-

stabilized biosolids, however, the majority of nitrogen present is in the form of organic nitrogen (Shepherd, 1996; Gshwind and Pietz, 1992). Several biochemical processes must therefore occur before plants benefit from this nutrient source. A depiction of the nitrogen cycle is presented in Figure 1.



Figure 1. The nitrogen cycle (Pidwirny, 2000)

Mineralization is an enzymatic process in which organic nitrogen is decomposed to inorganic forms. The first step is ammonification, in which microbes break down organic nitrogen and produce ammonia, which readily dissolves in water to form the ammonium cation (NH_4^+). This process occurs in either anaerobic or aerobic conditions and is performed by a broad group of heterotrophic organisms. Many of the organisms are thermophilic; hence optimum ammonification occurs at temperatures between 40°C and 60°C (Lewis, 1986), though it can occur at lower temperatures, albeit at a slower rate. Ammonium adsorbs to cation exchange sites; consequently, those soils with higher CEC values (e.g., clays) are more likely to inhibit percolation of ammonium than lower CEC soils (e.g., sands). This adsorption, however, depends upon the prevalence of other competing cations in the soil water; the uptake rate of ammonium by plants and microbes; and potential oxidation of ammonium as described below (Loehr, 1979).

Ammonia (NH₃, the gas) and ammonium (NH₄⁺, the cation) are in equilibrium with one another as represented by the following equation: $NH_4^+ + OH^- \leftrightarrow H_2O + NH_3^+$. Because this is an equilibrium process, anything that impacts the represented compounds will alter the balance, and drive the equation in whichever direction restores the balance. Consequently, high pH levels (by definition from higher concentrations of OH⁻ ions) as well as a decrease in water content will drive the equation to the right, and more ammonia will be produced and available to volatilize. Volatilization is impacted by contact with air and soil. If at the soil surface, more ammonia will volatilize. When placed underground in close contact with the soil, diffusion to the atmosphere is inhibited. In addition, ammonia will be adsorbed by clays and organic materials, further diminishing volatilization. Studies performed clearly demonstrate that placing biosolids in the subsurface (as opposed to the surface) significantly decreases ammonia losses (Adamsen, 1987; Brady and Weil, 2002).

The second step of mineralization is nitrification. It consists of two main sequential transformations that include: 1) the oxidation of ammonium to nitrite (NO₂⁻), typically performed by the autotrophic *Nitrosonomas* bacteria; and immediately thereafter 2) oxidation of nitrite, typically performed by *Nitrobacter* bacteria to produce nitrate. Other genera of bacteria that can perform this function do exist (e.g.,

Nitrosolobus and *Nitrocystis*) but, in general, the process is dominated by *Nitrosonomas* and *Nitrobacter* (Lewis, 1986). The swift transition from nitrite to nitrate usually prevents accumulation of nitrite. Nitrification is usually performed by autotrophic bacteria, which derive their energy from the oxidation of NH₄⁺ and NO₂⁻, as opposed to the oxidation of carbonaceous compounds (Haynes, 1986). Both genera of the nitrifying organisms cited (i.e., *Nitrosonomas* and *Nitrobacter*) as primarily responsible for this reaction sequence are aerobes, requiring the presence of oxygen to perform these conversions. In addition, they favor soils with no more than 60% of pore volume filled with water, need a carbon source (i.e., bicarbonates and carbon dioxide) to synthesize their cell components, and optimally perform at temperatures between 20-30°C (Brady and Weil, 2002; Lewis, 1986).

Nitrate is an anion that is not readily adsorbed to soil particles, is water soluble and therefore highly mobile. Of the forms of nitrogen described above, it presents the highest risk of leaching through the soil profile to the groundwater table. Additionally, nitrate warrants the most concern from a human health and environmental pollution perspective. Most acutely in infants and ruminant animals, ingested nitrate is reduced to nitrite, which decreases the oxygen-carrying ability of red blood cells and produces a condition known as methemoglobinemia (Brady and Weil, 2002). Consequently nitrate is a regulated pollutant in drinking water with a Maximum Contaminant Level (MCL) of 10 mg/L for NO₃-N (EPA, 1994).

Nitrate also can have a pronounced impact on aquatic systems. An influx of nitrate promotes algal blooms that, upon dying, are decomposed by oxygen-demanding bacteria. Exponential growth and decay results in exponential demand and depletion of

oxygen. Hypoxic conditions result that are toxic to many forms of aquatic life. Proliferation of this cycle can expand these inhospitable zones on a yearly basis, rendering once productive waters lifeless (Brady and Weil, 2002).

A number of studies have been conducted to determine the most important factors impacting mineralization. Wang, et al. (2003) performed a laboratory incubation study in which two different types of biosolids (anaerobically digested and dewatered sludge; liquid stabilized sludge from an autothermal thermophillic aerobic digestion) were mixed with two representative soils (a stony silt loam and a sandy volcanic soil) and incubated at two different temperatures (10°C and 20°C). As expected, mineralization rates were significantly greater at the higher temperature. A greater percentage of the organic nitrogen was mineralized in the aerobic biosolids and, overall, mineralization occurred sooner and more rapidly in the sandy volcanic soil. Wang reasoned that the lower pH of the silt loam (4.5 vs. 5.4 for the sandy soil) might have inhibited the microbes.

Another study focusing on predicting mineralization rates determined that the standard classification of biosolids by treatment processes (e.g., primary, aerobically digested, anaerobically digested, and composted biosolids) was not a reliable differentiating factor to use for mineralization impacts unless extensive stabilization had occurred (Gilmour, 2003). Instead, it was more appropriate to evaluate the organic and inorganic N content combined with the decomposability of the biosolids (which would be greater for unstabilized biosolids, regardless of the treatment process). A broader scope of factors was considered by Er, et al. (2004), who modeled factors impacting mineralization through regression analyses. Variables considered included: biosolid type, biosolid organic N content, biosolid application rate, biosolid carbon to nitrogen (C:N)

ratio, soil organic N content, soil pH, time, and temperature. The most relevant factors elicited from this analysis were biosolid application rate, biosolid C:N ratio, and temperature.

Despite the varying focus of the studies cited above, there is a general consensus that the following factors represent the more important conditions impacting the degradation of biosolids.

- The chemical composition of the decomposing material, including:
 - a) Nitrogen content (inorganic vs. organic and relative concentrations):
 Some studies have indicated that the presence of inorganic forms of nitrogen act as a primer and facilitate more rapid mineralization (Haynes, 1986). High concentrations of NH₄⁺, however, may inhibit nitrification (Brady and Weil, 2002; Nielsen and Revsbech, 1998).
 - b) The C:N ratio: A low C:N ratio (< 20) will promote rapid bacterial growth and mineralization, due to the high amount of nitrogen present. This surplus nitrogen will exceed the nutritional requirements of the microbes, and the decomposition products (NH₄⁺ and NO₃⁻) will be available in soil solution. The microbial activity will level off in correlation with the decreased availability of carbon. A high C:N ratio (>25) also will prompt an initial surge in microbial activity, but this surge will be depressed once the microbes consume the nitrogen. At this point, nitrogen will be immobilized in the microbes and unavailable in the soil solution. The microbes population will stagnate and nitrogen will not

become available until this population dies and decomposes (Haynes, 1986; Brady and Weil, 2002).

- c) The types of carbon in the biosolids: Easily decomposed fatty acids, amino acids, simple sugars, and starches will initiate faster, more intense mineralization (Sylvis Environmental, 2000). Conversely, lignin decomposes more slowly and may override the impact of nitrogen mineralization by facilitating the synthesis of stable, nitrogen-containing humic polymers (Haynes, 1986).
- d) Moisture Content of the Biosolids and Surrounding Soil: Dry soils with <10-20% of their pore space filled with water are inhospitable to most microbes under consideration. The heterotrophic organisms responsible for ammonification can tolerate a wider range of moisture content, particularly on the upper end of the scale, enabling decomposition in waterlogged conditions. The more select group of nitrifying bacteria operates in a narrower window, with optimum performance when 50-60% of pore space is filled with water. Above 70% water content, nitrification decreases significantly. Some studies have shown that alternate drying and wetting conditions promote mineralization. The wetting process promotes release and movement of organic compounds that serve as an energy source. Nitrification occurs as the soil conditions enter the most favorable water contents. As the soil dries, microbes die and the nitrogen cycle begins anew (Sylvis Environmental, 2000; Haynes, 1986; Lewis, 1986).

- e) Aeration of the Biosolids and Surrounding Soil: Aeration complements the moisture content. As stated above, ammonification can occur in the absence of oxygen, but nitrification is an aerobic process.
- f) Temperature: Although microbes can operate at temperatures as low as 0°C, optimum temperatures for ammonification are in the thermophillic range of 45-60°C and optimum temperatures for nitrification are in the mesophillic range of 20-35°C (Brady and Weil, 2002; Lewis, 1986; Sylvis Environmental, 2000).
- g) pH of the Biosolids and Surrounding Soil: Neutral to slightly basic pHs foster the most effective decomposition. The microbes responsible for nitrification are more sensitive to acidic conditions than ammonification, though research has shown that nitrification can occur, although at diminished rates, at pH conditions as low as 4.0 (Lewis, 1986; Sylvis Environmental, 2000).
- h) Soil Type: Sandy soils drain easily and are less susceptible to waterlogged conditions. Increasing concentrations of clay impart a more significant water holding capacity that can lead to sustained saturated conditions. In addition, the higher CEC capacity of clay soils results in adsorption of organic materials and ammonium, which can limit their availability to microorganisms.

The converse of mineralization is immobilization, in which ammonium or nitrate is complexed into an organic form via biotic or abiotic means. Both mineralization and immobilization processes occur simultaneously, as microbe populations grow and die,

and rates are dependent upon the composition of the soil (Haynes, 1986; Lewis, 1986). Those factors that most influence immobilization include: the carbon to nitrogen ratio, with a C:N above 25 leading to a higher immobilization; the inorganic form present, with microbes favoring NH_4^+ over NO_3^- ; competition between microbial populations and plants; and those physical and chemical properties that impact the microbial population dynamics, as described above.

Denitrification refers to those processes in which nitrate ions are converted to gaseous forms of nitrogen {e.g., nitric oxide gas (NO_2^+) , nitrous oxide gas (N_2O^+) , and dinitrogen gas (N_2) . The order of conversion is as follows: $NO_3^- \rightarrow NO_2^- \rightarrow NO$ (gas) \rightarrow N₂O (gas) \rightarrow N₂ (gas). In this sequence of reactions, which typically occur under oxygen-depleted conditions, nitrogen, as opposed to oxygen, acts as the terminal electron acceptor. The majority of bacteria performing this function are facultative anaerobes that can be either heterotrophs (i.e., obtain their energy and carbon from oxidation of organic compounds) or autotrophs (i.e., obtain their energy and carbon from carbon dioxide or carbonates). Some organisms are capable of catalyzing the entire sequence of reactions; others can only initiate specific steps. Typical conditions include a mixed community of bacteria performing different functions (McEldowney, et al., 1993). Required environmental conditions include: the presence of nitrate; low soil air content (<10%); temperatures between 2-50°C (with an optimum range of 25-35°C); a pH optimally between 7-8 (though some bacteria are capable of denitrifying under more acidic conditions); and an appropriate energy source (i.e., organic carbon) (Oertel and Nicklow, 2003; Brady and Weil, 2002; Barber, 1995).

Land Application of Sewage Sludge

Land application of sewage sludge to improve soil conditions, enhance crop production, improve silviculture operations, and reclaim mined land has been extensively studied. Sludge is either applied 1) on the surface, 2) by disking or plowing into the soil to a prescribed depth (usually no more than 15 cm) or 3) via injection underneath the surface. Nitrogen requirements of the crop and background soil concentration dictate application rates, with seasonal or yearly applications of the sludge often being performed. Site and crop specific management are the key to optimizing growth while preventing nitrogen loss from the system (Ritter and Bergstrom, 2001; EPA, 1994; Outwater, 1994; Granato and Pietz, 1992).

Numerous examples of nitrate leaching under biosolids-amended agricultural land have been reported in the literature (Ritter and Bergstrom, 2001; Shepherd, 1996; Clapp, et al., 1994). In these studies, the timing and rate of application, type of sludge used, nutrient demands of the crop, and soil conditions influenced the loss of nutrients. Often, a majority of the leaching could have been prevented through more careful management. Evanylo (2003) evaluated the impacts of biosolids application at two different times of the year (winter and spring) and at three different application rates bracketing the agronomic rate of corn crops planted at experimental sites in Virginia. Results showed that leaching loss of nitrogen (as nitrate) was: greater in the winter than in the summer; greater in coarser (sandier) soils than finer textured (higher silt and clay content) soils; and was more pronounced during periods of higher rainfall.

Currie et al (2003) monitored nitrogen mineralization and leaching after application of lime-stabilized biosolids to soybean fields. Results indicated that a surplus

of nitrogen was available in the soil because the soybeans continued to fix nitrogen. Despite this, nitrate concentrations in the groundwater were below 10 mg/L, indicating the possibility of denitrification.

Lee (2004) evaluated the impact of three different field management practices on soil nitrate distribution in clay soils that were amended with biosolids and planted with wheat. Biosolids were applied to exceed the agronomic rate. One management practice consisted of leaving the field fallow for a year followed by cropping with wheat on an annual basis; the second immediately cropped the wheat and continued to do so on an annual basis; the third was the same as the second, except commercial fertilizer was applied in addition to the biosolids. Results from soil samples collected two years after biosolids application showed that the maximum nitrate content in the soil was directly related to the amount of biosolids applied. In addition, the fallow treatment had a higher concentration of nitrate deeper in the soil profile than the other treatments, indicating that more leaching occurred in the absence of wheat crops. For all treatments, nitrate decreased significantly past depths of 100cm. Because clay soils tend to hold moisture longer than sandy soils (i.e., they do not drain as easily), it was reasoned that conditions were likely appropriate for denitrification to occur at these depths.

Mitchell, et al (2000) evaluated the cycling of nitrogen on a small stand of Scots pine that received an application of anaerobically digested biosolids. This traditionally nutrient poor ecosystem initially responded with fluxes of nitrogen in the upper soil profile, mainly in the form of ammonium, an order of magnitude above that of the control plot. After 17 months, some nitrate leaching was observed, but all were below 10 mg/L, demonstrating an effective use of biosolids that results in minimal leaching of nitrogen.
Other studies that demonstrate the ability to minimize nitrate leaching have been performed on land reclamation projects. Larger scale reclamation operations presented by Van Ham, et al. (2000), Sopper (1993) and Lue-Hing (1992) show that with appropriate biosolids type, application rates, and conditions, nitrogen from the biosolids can be preserved and recycled in the upper layers of the soil profile. A reclamation project in British Columbia (Van Ham, et al., 2000) transformed nutrient depleted gravel mines into self-sustaining tracts of vegetation that increased the environmental quality of the site. The vegetation not only enhanced the aesthetic and ecological value of the site, but actually reduced nitrogen movement that previously migrated to a nearby aquifer. When properly used, biosolids are an environmentally safe and effective nutrient source that greatly improves soil condition, optimizes crop production, and enhances the soil and land ecosystem into which it is introduced.

Trenching of Sewage Sludge

The majority of land application is in the form of surface spreading or subsurface incorporation, both of which evenly spread the biosolids across the parcel being fertilized. Trenching, on the other hand, refers to filling excavated rows with large volumes of sludge that are subsequently covered with overburden. This technique was studied in the 1970s and focused on the entrenchment of sewage sludge as a disposal option, as opposed to reintegration of biosolids as a beneficial reuse protocol. An added benefit (though not the primary objective of these biosolids disposal efforts) was the reintroduction of nutrients into the land, particularly land that had been over farmed.

Walker (1974) summarized the results of studies conducted on sewage sludge from the Blue Plains Wastewater Treatment Plant that services the Washington, D.C. metro area. In this study, dewatered raw-limed sludge was applied to trenches 0.6 m wide by 0.6 - 1.2 m deep. A variety of crops such as fescue, alfalfa, rye and trees were grown. Underground and surface drainage water, as well as groundwater from the site was monitored. Results from 19 months of data gathering demonstrated that entrenchment prevented contamination of surface water, promoted slow nitrogen release, and created an unfavorable environment for pathogens. An increase in nitrate levels was observed in the soil under the trenches and in subsurface drainage water, but not in groundwater samples. No metals movement was observed in the substrate. Increases in chloride were observed in groundwater samples, but this was the only migration of significance.

Nineteen months after entrenchment, sludge dewatered from the top down and between one-fifth to one-half of the trench progressed from its original 20% solids gelatinous mass to a peat-like consistency. The rate at which weathering occurred depended on the type of sludge used (e.g., digested sludges degraded faster than rawlimed sludges) and the extent of plant root penetration. This study indicated that entrenchment was a suitable procedure, but longer-term studies were recommended to determine the full effect of this practice.

Similar research was conducted by Sikora, et al. (1978) over a four-year period (1972-1976) to evaluate water quality at a sludge entrenchment site consisting of sandy soils with an underlying clay layer. In this study, lime-stabilized sludge was placed in $0.6m \ge 0.6m$ trenches and covered with 0.15 - 0.30 m of subsoil. Crops and fescue were

grown over the entrenchment area, though again vegetation was a secondary consideration. Water samples were collected from drainage tile lines, a catchment pond, and monitoring wells within and around the trenched plot. These studies showed a peak in chloride levels 18 months after entrenchment and a peak in nitrate concentration a year after the chloride peak (i.e., 30 months after entrenchment). Nitrate concentrations were below the EPA MCL of 10 mg/L nitrate-N in wells above and below the trench plot. Though a high nitrate concentration of 60 mg/L occurred during November 1974 in one well within the trench plot, most concentrations (>85%) were below 10 mg/L. Tile drains exhibited a high nitrate-N concentration of 32 mg/L. Other observations of note were that metals did not migrate and pathogens were significantly reduced.

Sikora et al. (1980) further evaluated the trenching technique with particular focus on the dynamics within and below the trench over a four-year period (1974–1978). Observations included an analysis of the original sludge sample and then the progression of the sludge starting at 22 months after entrenchment. Results showed the following patterns:

- After 22 months of entrenchment, the top portion of the sludge 5-20 cm (2-8 inches) from the top of the trench had dried out and was densely penetrated with roots. The middle and bottom portions of the trench did not dewater until 49 months after entrenchment. After this four-year period, the entire trench contents appeared to have stabilized. Similar to Walker's observations, dewatering occurred from the top down.
- A majority of chloride leached through the trench within the first year of application. Chloride, a water-soluble anion commonly found in biosolids,

does not interact chemically with most soils and provides an indication of water flow and maximum leaching potential through the biosolids and soil profile. The first reading at day 655 showed that chloride concentration was highest in the bottom of the trench, moderate in the middle of the trench, and lowest in the top of the trench. At the inception of this experiment, chloride originally present in the biosolids already had migrated through the trench.

- Organic nitrogen and ammonium leached through the soil profile.
 Distribution patterns at the beginning of the experiment (day 655) were similar to that of chloride. Ammonium in particular was present at much higher concentrations in the bottom of the trench compared to the middle and top. After 4 years, concentrations below the trenches returned to low or background levels for both parameters.
- Nitrate, an anion with the same water-soluble properties and leaching potential as chloride, exhibited a pattern different from chloride and ammonium. At day 655, the highest concentration was in the top of the trench, with lower levels in the middle and very low amounts at the bottom. With time, samples showed a progressive increase in the middle of the trench that eclipsed the top of the trench at day 998. This progression of nitrate concentration is consistent with the conditions in the trench at these dates. The production of nitrate via mineralization of ammonium requires an aerobic environment, which only existed in the top of the trench at the beginning of the experiment. Subsequent dewatering of the trench fostered conditions for additional mineralization to occur deeper in the trench.

Also important to note is that once produced, nitrate will either 1) be taken up by plants or microorganisms or 2) leach further down the trench with the water flow and/or 3) undergo denitrification. The fact that nitrate concentrations do not correspond to the timing patterns exhibited by the equally water soluble chloride indicates that 1) nitrate production via mineralization was delayed for months after biosolids entrenchment and 2) once produced, though some nitrate may have leached to the bottom of the trench, the waterlogged, anaerobic conditions were optimal for denitrification. This theory is supported by the fact that concentrations in the bottom of the trench did not reach the levels in the upper portions. Additionally, concentrations in the soil below the trenches, though elevated for a time to a maximum of 54 mg/kg, decreased to low levels (2-6 mg/kg) by the end of the experiment.

This and subsequent evaluations of the entrenchment technique (Sikora, et al., 1982; Sikora and Colacicco, 1980) led to the conclusion that contamination of the groundwater could occur dependent upon the soil characteristics and depth to the groundwater table. Experiments provided evidence, however, that recharge would likely dilute the nutrients. Consequently, the specific characteristics of an individual site would need to be evaluated to determine if groundwater contamination posed too much risk for this technique. It is important to note, however, that these experiments did not attempt to utilize a deep-rooted crop or plant a specific crop density that could reach and utilize the nutrient reservoir supplied by the biosolids.

Use of Zero-Tension (Pan) Lysimeters and Suction Lysimeters to Collect Soil Water Samples

A number of studies have been conducted to determine differences in the chemical constituents of soil water captured by zero-tension pan lysimeters versus suction lysimeters. Barbee and Brown (1986) evaluated the ability of zero-tension pan and suction lysimeters to track chloride movement through three soils of differing texture. The suction lysimeters were not able to sample well-structured clay soils, the soil water from which was postulated to have bypassed the smaller suction lysimeters. Soil water from the clay was, however, captured by the pan lysimeters. What samples were collected by both lysimeters produced equivalent results without statistically significant differences. The authors concluded that, despite the differing soil water collection techniques of the two pieces of equipment, both were able to accurately characterize the flow of chloride, with reservations for the use of suction lysimeters in soils with high clay content.

Haines, et. al. (1982) compared nutrient concentrations collected using tension and zero-tension lysimeters. The tension lysimeters used in this experiment were plates, as opposed to cups, but operate on the same collection principle. Results for samples collected from two positions in the soil profile, one at the soil-litter interface and another 30-cm below the soil-litter interface, showed differences between the chemical constituents collected by the two types of sampling equipment. Specifically, zero-tension lysimeter results were higher than suction lysimeter results for both ammonium and nitrate. For the soil-litter interface, results for the zero-tension lysimeter were higher than the suction lysimeter by a factor of 1.5 for both ammonium and nitrate, which was not

statistically significant. At 30-cm below the soil-litter interface, results for the zerotension lysimeter were higher than the suction lysimeter by a factor of 5.1 and 3.4, respectively, which was statistically significant. Also important to note is that the zerotension lysimeters collected 7 times more water than the suction lysimeter at the upper position, but 2.1 times *less* water than the suction lysimeter at the deeper position. The authors reason that the higher concentrations in the zero-tension lysimeters were a product of a pulsed element input to saturated flow, which the zero-tension lysimeter captured more efficiently than the suction lysimeter.

In contrast, Hendershot and Courchesne (1991), found consistently lower concentrations of nitrate in zero-tension lysimeters versus suction cup tension lysimeters in a comparative assessment of the collection equipment in a sugar-maple stand. Pairs of the samplers were installed at 25 and 75 cm depths in the soil. Samples were analyzed for a number of nutrients, including ammonium and nitrate. Ammonium was present in higher concentrations in the suction lysimeter at the 25cm depth, but not enough to be statistically significant. At 75 cm, ammonium concentrations between the two lysimeters were equivalent. The absence of nitrate in the zero-tension lysimeter samples and presence in the suction lysimeter samples could not be satisfactorily explained, but was postulated to be the result of either: 1) uptake by microorganisms, which could have preferentially occurred in the zero-tension lysimeters because more suspended material (including microbes) enters the sampler with the soil water or 2) denitrification, which occurs in an anaerobic environment consistent with the saturated soil conditions required for collection by the zero-tension lysimeters. This experiment presents a scenario in which the zero-tension lysimeter collection conditions, as opposed to those of the suction

lysimeter, are more predominantly associated with chemical and biological transformations.

Yet another perspective is offered in experiments conducted by Marques, et al. (1996) at four different depths under a forest soil. Solutions collected by zero-tension plate lysimeters and ceramic-cup tension lysimeters were compared for various nutrients. One major difference to note from other studies presented (and the one conducted for this thesis) is that the suction cup lysimeters were placed under constant suction, as opposed to a limited time period of suction. Tension lysimeter solutions contained higher concentrations of nitrate and ammonium across all depths. The authors concluded that the two types of equipment represented the soil water differently. Zero-tension lysimeters collected the flux solution governed by gravitational forces, which had a shorter residence time in the soil and was primarily related to chemical and biological processes occurring in the upper soil horizon, after which the swift vertical migration would inhibit interaction of the solution with the soil. Tension lysimeters, however, collect fixed phase soil water that more closely represents longer-term biogeochemical processes throughout the soil profile including mineralization, ion-exchange, mineral weathering and ion uptake. For this reason, the breakdown products of organic nitrogen were more prevalent in the tension lysimeter solutions.

From all of these studies, it is apparent that, with the exception of the known fact that zero-tension lysimeters capture saturated flow and suction lyismeters capture both saturated and more predominantly unsaturated flow, no one physical, chemical, or biological process can be selectively linked to either collection apparatus. Rather, the soil water collected by either of these lysimeters is a product of the specific

circumstances governing the experimental set up and environmental conditions. Using both types of sampling equipment does, however, ensure that a comprehensive representation of the soil solution in the soil profile will be obtained.

Hybrid Poplar Trees and Their Use With Pollution Management

The genus *Populus* includes those trees commonly referred to as poplars, aspen, and cottonwood. They are part of the botanical family *Salicaceae*, which also includes willow trees. Hybrid poplars are crosses of two different species that are often developed to enhance desirable traits, such as hardiness, nutrient uptake, or salinity tolerance. Clones are a group of genetically identical plants that result from vegetative production of a single tree.

Hybrid poplars are well known for their high water uptake and transpiration rates and have been used for the containment and remediation of nutrients, explosives such as TNT, trichloroethylene, and a variety of other organics (Pivetz, 2001; Newman, et al., 1999; Burken and Schnoor, 1998). Specific studies evaluating groundwater capture and hydrologic flow have recorded water use between 1.2 and 25 gallons/day/tree (Ferro, et al., 2001). Other studies in which root growth was directed to an aquifer 25 feet below the surface estimated even higher uptake rates between 8-50 gallons/tree/day dependent upon the month and age of the tree. (Quinn, et al., 2001). Such high water use supports the potential to provide a large degree of leachate containment, though results vary according to the specific site characteristics, density of trees planted, and climatic conditions.

Licht (1990) evaluated the effectiveness of poplar tree buffer strips to control nonpoint source pollution, particularly nitrogen. He concluded that hybrid poplars 1)

naturally form extensive rooting systems that can be further enhanced using deep planting techniques; 2) significantly reduce nitrate concentrations in the soil profile as well as in near-surface groundwater from 90 mg/L levels to 2 mg/L (well below the drinking water MCL of 10 mg/L), and 3) are capable of surviving in both waterlogged and drought conditions.

Haycock and Pinay (1993) performed a comparison of nitrate reduction in grass and poplar vegetated riparian buffer strips in winter months and found that the poplar zone exhibited 99% retention of nitrate compared to 84% retention in the grass zone. Though active plant uptake of nitrate was reasoned to be low in the dormant season, the high carbon contribution of the poplar trees at deeper levels in the soil likely provided a better substrate for denitrifying microbes.

O'Neil and Gordon (1994) performed a controlled bench study in which an artificial riparian zone was created using Carolina poplars. The experimental chambers were fertilized with nitrate solutions, irrigated, and the leachate was collected on a weekly basis. The plots with trees removed a significantly larger amount of nitrate than the control plots and provided further evidence that poplar trees are capable of removing nitrate from soil water over time.

In summary, characteristics that favor use of hybrid poplar trees in nutrient recycling and land reclamation activities include:

• They are nutrient demanding, with an average uptake range of 91-163 kg (200-360 lbs) of nitrogen per acre per year (National Agroforestry Center, 2000), and the ability to utilize as much as 225 kg (500 lbs) of nitrogen per acre per year as estimated from other studies (Murray, 2003).

- They are phreatophytes, will extend roots to the capillary fringe, and can survive periods with their roots in the saturated zone of an aquifer
- The fibrous nature of the roots enables penetration of both highly permeable and less permeable soils.
- Impressive growth rates produce large amounts of biomass that act as a significant carbon sink.
- They are hardy, with high survival rates and can withstand high planting densities.

Studies performed at the ERCO Tree Farm on over 11 clones have demonstrated that the OP376 variety (a *Populus deltoides* x *P. nigra* clone) is the overall best performer, exhibiting superior survival and growth in Maryland sites (Kays, 2002).

Research at the ERCO Site

Techniques implemented at the ERCO Tree Farm represent a confluence of trenching, reclamation of mine spoils, and poplar tree cultivation. Research conducted at ERCO prior to this thesis experiment has focused on 1) groundwater monitoring, 2) nitrogen budgets and 3) hybrid poplar growth and survival. Pepperman (1995) performed a review of data collected over the course of operations at the ERCO Tree Farm. Evaluation of soils collected during well drilling and placement of test pits provided an overview of the geological stratification at the site. General observations included:

- Sand, gravel and some clay comprised the upper surface to depths of 0.91-1.22 m (3-4 ft).
- 2) Silts with clay and traces of sand were present between 0.91-2.44 m (3-8 ft).
- 3) Clays dominated depths from 2.44-5.49 m (8-18 ft).

 Depths of 5.49-24.4 m (18-80 ft) consisted of fine sand, some clay and a little silt.

The overriding conclusion was that a slowly permeable layer exists below the remnants of the mining operation. This layer is situated at a depth below that of the biosolids rows.

Seven groundwater wells installed up gradient, down gradient, and within the site provide information on background levels of pollutants and groundwater conditions after placement of biosolids. A background sample collected in 1982 had a nitrate-N concentration of 1.5 mg/L and pH of 7.8. Twelve years of subsequent monitoring after biosolids application showed the following trends.

- Little change in overall water quality
- No increase in chloride concentration. As previously explained, chloride is a good indicator of water flow from the biosolids. This demonstrates that water leaching from the biosolids is not percolating to the aquifer from which the groundwater samples are collected.
- Nitrate concentrations were mostly nondetects. The two highest readings of 1.5 mg/L and 1.9 mg/L came from the same well, with the 1.5 mg/L reading occurring prior to application of biosolids (i.e., it represented background levels).
- Metal concentrations, with particular focus on lead and cadmium, were near or below detection limits. None of the detects exceeded the drinking water MCLs.
- Fecal coliform levels were generally low, with some increased readings in November and August.

Pepperman also evaluated the nitrogen balance in this farming operation. Inputs consist of the biosolids, atmospheric deposition, leaf litter and background soil concentration. Outputs and/or storage vehicles include: the poplar trees; storage in the soil matrix; losses as leachate; and gaseous losses through volatilization and denitrification. The greatest challenge in estimating the balance was determining an accurate degradation rate for the biosolids. Most quantitative information on degradation comes from land application practices. The deep row technique, however, creates a unique environment that hinders mineralization for the following reasons: 1) temperatures in the deep rows are lower than those near the surface, such that microbial activity is slower; 2) until tree roots permeate the soil and the biosolids begin to dewater, oxygen, which is necessary for nitrification, will be scarce; and 3) the high pH of the limed biosolids, along with the accompanying high salt concentrations, are adverse environments for some of the microbes that perform these nitrogen conversions.

Using information derived from the literature and ongoing studies, including information on less than optimal growth of trees at the ERCO farm, Pepperman determined that the permitted rate of application was at least 25% less than that necessary for the specific operations at the ERCO site. Results of this evaluation and other studies has led to the testing of increased application rates performed as part of this thesis project.

As noted in the prior section on hybrid poplars, other studies at the ERCO tree farm tested the survival and growth of 11 different hybrids, and determined that the OP367 variety was the most appropriate for use at the farm (Kays, 2002).

Chapter 3: Objectives

Primary objectives are as follows:

- Evaluate the fate and transport of nitrate in biosolids and the surrounding soil profile over time.
- Develop an overview of the water quality associated with the new crop of poplar trees that are being planted at a lower density with higher biosolids application rates through the analysis of soil leachate and soil water samples.

Chapter 4: Methods and Materials

Site Location and Characteristics

The ERCO Tree Farm is a privately-owned tract of land in Brandywine, Maryland, situated on the southern edge of Prince George's county (see Figure 2). The former sand and gravel mine spans approximately 49 ha (122 acres) and has been subjected to reclamation efforts since 1983.





The general technique employed at the ERCO site consists of applying biosolids in deep rows (approximately 0.76m deep x 1.0m wide) at a rate of either 383 or 658 dry Mg/ha (171 or 294 dry tons/acre). Rows are dug with a backhoe and approximately ³/₄ of the trench depth is filled with biosolids. The row is then covered with backfill from the subsequently dug row to produce an overburden cap approximately 0.3-0.6 m deep, effectively sealing the biosolids underground. In the spring, hybrid poplar stem cuttings are planted on the treated field. This one-time bulk application of biosolids acts as a nutrient source for the 6-year growing cycle, at which point the trees are harvested (Kays et al., 1999). Following harvest, the cycle is repeated, with new biosolids rows perpendicular to the prior rotation, facilitating a long-term operation that can ultimately produce a viable, permanent ecosystem.

Approximately 36.5 ha (90 acres) of the gravel spoil is actively farmed at the ERCO site, with each 4.05-ha (10-acre) parcel in different phases of production. Earlier rotations of tree crops were planted at densities as high as 1215 - 2430 trees/ha (3000 – 6000 trees/acre), which resulted in crowding and stunted tree growth. Beginning in 2000, however, crops of 202 trees/ha (500 trees/acre) have been planted in an attempt to produce a more marketable wood product.

The site is topographically characterized as a plateau with steep forested banks that fall away to a stream incision. Vegetated berms 0.6 - 0.9 m (2-3 ft) high surround the plateaued areas to control runoff, and runoff is routed to four detention ponds. Seven monitoring wells ranging in depth from 6.1-30 m (20-100 ft) were installed around the perimeter and within the plateaued area from 1982-1990.

Background groundwater and soil samples were collected prior to the application of biosolids to establish baseline conditions. During and subsequent to biosolids application, groundwater was evaluated from the monitoring wells on a biannual basis. Between 1988 and 1998, samples were collected and tested for: fecal coliform, pH, color, chloride, turbidity, total residue, ammonia, nitrite, nitrate, total alkalinity, hardness, sulfate, arsenic, barium, cadmium, chromium, copper, fluoride, iron, lead, manganese, mercury, nickel, selenium, silver, sodium, and zinc. Surface water samples also were collected from creeks upstream and downstream of the site as were soil samples before and after the biosolids application process. Of particular interest were the fecal coliform,

chloride, nitrate, cadmium and lead results, due to their potential presence in biosolids and possible adverse health and environmental impacts.

Analytical results demonstrated that the pollutants were not present in appreciable quantities in the water samples {i.e., concentrations were either nondetects, below the EPA drinking water maximum contaminant levels (MCLs), and/or below the Cumulative Pollutant Loading Rates as specified in 40 CFR 503}. These results, collected over 10 years of the farming operation, definitively indicated that the tree farm was not having adverse effects on the water supplies of the area (Pepperman, 1995). Based on this well-established trend of low metals concentrations, the Maryland Department of Environment (MDE), the regulatory authority overseeing ERCO's permit, stipulated that metals and some of the wet chemistry parameters no longer needed to be determined. Subsequent groundwater monitoring has focused on the shorter list of parameters and continues to date under the conditions of the current permit. Overall, results continue to demonstrate that groundwater sources have not been adversely impacted by this beneficial reuse operation.

Mining activities have destroyed and removed any semblance of an organized soil profile. What remains are the mining spoils and an underlying 1.5-21.3 m (5-70 ft) clayey layer. A more specific description of the soil presented in Wilson and Fleck (1990), which evaluated soil borings in Prince George's County close to the Tree Farm site, is as follows.

The uppermost layer, which was removed during mining operations in the 1960s and 70s, is described as Pliocene Upland Deposits. These deposits, which ranged from 6.1-15 m (20-50 ft) thick, consist of silty, fine to very course sand and gravels, as

well as some yellow or orange silty clays. Though a majority of this layer was mined, remains of these deposits still exist throughout the farm.

- The next layer down, which is what is now predominantly at the surface of the graded farming areas, is the lower Miocene Calvert Formation. These marine shelf environment deposits are a micaceous, clayey silt approximately 27-30 m (90-100 ft) deep.
- Underneath the Miocene is the lower Eocene Nanjemoy Formation, which consists
 of fine to medium glauconite-bearing sands and ranges from 27-38 m (90 125 ft) in
 thickness.
- Underneath all of the above is the Marlboro Clay Formation, a hydrologically confining unit between 4.6-9.1 m (15-30 ft) deep.
- Multiple aquifers are located below the Marlboro Clay Formation.

On-site soil sampling from well drilling and trenching activities have delineated a more site-specific geological stratification pattern, which was presented by Pepperman (1995) and is summarized in the literature review above. One of the more important conclusions of this evaluation was that the site contained a confining, very slowly permeable layer situated below the deepest biosolids row depth (i.e., deeper than 0.8m) that would significantly hinder leachate flow to groundwater. This is consistent with the findings of the Maryland Geologic Survey described above.

Experimental Design

This section provides a general overview of the experimental setup. Details regarding implementation of each facet are provided in subsequent sections. The 1.2-ha (3-acre) research site is located within a 3.65-ha (9-acre) parcel in the southeast corner of

the farm. The entire 3.65-ha (9-acre) parcel has been subject to a prior round of biosolids application and tree cultivation under ERCO's standard farming conditions. The 1.2-ha (3-acre) experimental site was partitioned into three blocks based on a north-south gradient of changing soil composition and slope. For this experiment, three biosolids application rates of 481, 962, and 1443 dry Mg/ha (215, 430, 645 dry tons/acre), which provided approximately 19,650, 39,300, and 58,900 kg N/ha (17,400, 34,800, and 52,000 lbs N/acre), respectively and three tree densities of 0, 716, and 1074 trees/ha (0, 290, and 435 trees/acre) were tested. Each biosolids application rate/tree density combination was replicated three times. Three controls, positioned on the west end of each block, contained no biosolids or trees. Biosolids application rates were randomly assigned but, due to logistical considerations, tree densities were not.

A total of 30 different subplots resulted from this set up with the layout representing a split-block design. Each subplot extends approximately 22m (72 ft) in an east-west direction and either 32m (105 ft), 21.3m (70 ft), or 10.7m (35 ft) in a north-south direction to accommodate the tree densities of 0, 716, and 1074 trees/ha (0, 290, and 435 trees/acre), respectively. Within each subplot containing trees, an outer perimeter of two rows of trees (6.1m, or 20ft) was designated as a buffer to isolate treatments and potential edge effects. All soil water collection equipment was installed in the inner rectangle delineated by this outer perimeter. The general experimental layout is depicted in Figures 3 and 4.



Figure 3. Experimental layout.



Figure 4. Experimental layout showing biosolids rows and subplot IDs.

A set of sampling equipment designed to capture soil water under and around the deep rows was installed in each of the 30 subplots. Each set of equipment consisted of the following:

 One pan lysimeter installed 0.3m (12 inches) under a deep row to collect leachate from saturated flow transport due to gravimetric forces. Pan lysimeters were installed as biosolids rows were being filled. A graphical depiction is provided in Figure 5.



Figure 5. Schematic of pan lysimeter layout.

Five suction lysimeters installed under and around the deep row to collect soil water either flowing past due to gravimetric forces or, more predominantly, held in the soil profile by matric forces. Three of the lysimeters were positioned 0.15m (6 in), 0.30m (12 in), and 0.60m (24 in) underneath the row

to capture vertical flow. The other two were positioned to capture lateral flow, one at 0.15 m (6 in) and the other at 0.30m (12 in) from the side of the deep row in the soil profile. Both were positioned at a depth equal to that of the bottom of the trench. The lateral flow lysimeters were installed around the same biosolids row. Suction lysimeters were installed after all biosolids rows were filled and the field was leveled. Figure 6 shows the positions of the lysimeters relative to the biosolids row, though it is important to note that not all of the lysimeters were installed under the same row. For more details regarding the requirements for suction lysimeter location, see the forthcoming section on suction lysimeter installation.

When installing pan lysimeters, soil core samples were collected at and above the pan installation depth to evaluate hydraulic conductivity of the soil. In addition, on a monthly basis as biosolids rows were being filled, a composite sample of the biosolids was collected upon delivery at the farm and analyzed for macro-and micro- nutrients as well as basic soil properties.

A rain gauge was installed at the farm and rainfall data were collected over the course of the project to evaluate overall atmospheric input of water as well as isolated storm events.



Figure 6. Schematic of suction lysimeter layout.

Biosolids Characteristics and Application

Biosolids currently used at the ERCO Tree Farm are from the Blue Plains Wastewater Treatment Plant in Washington, D.C. These are dewatered, lime-stabilized sludges that, although categorized as Class B biosolids, have markedly low metals concentrations. Lime stabilization is the addition of calcium oxide (CaO—quicklime) or calcium hydroxide (Ca [OH]₂—hydrated lime) to sludge to elevate the pH to a level for an appropriate period of time to inactivate microorganisms. Lime reacts with water to produce hydroxides that, in appropriate amounts, elevate the pH to 11 or 12, creating an environment inhospitable to the microbes. In addition, when quicklime (CaO) is used, an exothermic reaction occurs that elevates the temperature to fatal levels in the biosolids, further insuring the destruction of pathogens (EPA, 2000).

During the design stage of the research project, several samples of the dewatered, lime-stabilized biosolids were collected upon drop off at the tree farm and showed, on a wet weight basis, an organic nitrogen concentration of 1.16% (11,600 mg/kg), total phosphorus content of 0.38% (3800 mg/kg), pH values between 11-12, and percent solids content of 20-25%. Ammonia volatilization was evident from the distinct odor exiting the biosolids pile. Throughout construction of the deep rows, biosolids samples were collected on a monthly basis to assess physical and chemical properties over time.

Deep rows were constructed in a north-south direction (perpendicular to the prior set of deep rows) on 1.8–2.0 m (6-6.5 ft) centers with a width of 1.07 m (42 inches) and a total trench depth of either 0.61 m (24 inches), 0.94m (37 inches), or 1.24 m (49 inches), dependent upon the required application rate. With these dimensions, rows were separated by approximately 0.93m of gravel spoils. Rows were filled with biosolids to a depth of either 0.32 m (12.5 inches), 0.64 m (25 inches), or 0.96 m (37.5 inches) to achieve application rates of 1694, 3388, and 5082 wet Mg/ha (757, 1515, and 2277 wet tons/acre). With an average percent solids of 28%, this converts to 481, 962, and 1443 dry Mg/ha (215, 430, and 645 dry tons/acre). These biosolids loading rates resulted in total nitrogen applications of approximately 19,650, 39,300, and 58,900 kg N/ha (17,418, 34,837, and 52,255 lbs N/acre).

As biosolids were applied to the appropriate depths, they were covered with an initial layer of overburden. Then, as the next row was dug, the excavated overburden was placed on top of the previously filled row. This excavated layer, combined with the

initial layer of overburden, produced a cap over the biosolids approximately 0.46 - 0.76 m (1.5-2.5 ft) thick, effectively sealing the biosolids within the mine spoils. This process is shown in the pictures below (Figures 7-10).



Figure 7. Digging a deep row.



Figure 8. Offloading biosolids.



Figure 9. Pushing biosolids into a deep row.



Figure 10. Covering biosolids with overburden.

The basis of the experimental application rates were a combined evaluation of 1) the standard application rate of 383 dry Mg/ha (171 dry tons/acre) used at the tree farm 2) the demonstration plot application rate of 658 dry Mg/ha (294 dry tons/acre) used at the tree farm, 3) studies on the foliar nutrient content of the trees at the farm as well as 4) nitrogen mass balance estimates for the operation. With past total nitrogen contents of approximately 3.5% (dry weight), the standard 383 dry Mg/ha (171 dry tons/acre) biosolids application rate provided approximately 13,400 kg N/ha (12,000 lbs N/acre) and the 658 dry Mg/ha (294 dry tons/acre) rate provided approximately 23,070 kg N/ha (20,600 lbs N/acre). Nitrogen budget evaluations and foliar nutrient data collected from past rotations indicated that trees at the farm were not being supplied with enough nitrogen (Pepperman, 1995). After four to six years of growth, foliar nitrogen concentrations dropped below the optimal 3.5% level and in some plots foliar concentrations diminished to the point that trees were considered nutrient deficient.

Consequently the biosolids rates used in this experiment were designed to test rates similar to and greater than those in operation at the farm. Initial design planned for application rates of 20,160, 40,320, and 60,480 kg N/ha (18,000, 36,000 and 54,000 lbs N/acre). Slightly lower rates of 19,650, 39,300, and 58,954 kg N/ha were actually applied once it was determined that the substrate was too unstable to incorporate 12 biosolids rows within a 21.3m subplot width, and 11 rows were instead applied.

The biosolids distribution required for a given application rate is dependent upon the dimensions of the deep rows and how closely spaced the deep rows can be placed, among other factors. The procedure used to determine the amount of biosolids needed for each application rate is itemized below, with detailed calculations following in table format.

Note that each set of calculations is presented twice; first in S.I. Units, followed by a second table with the same information in *non*-S.I. Units to facilitate comparison to other studies.

- Based on the wet weight nitrogen content of the biosolids being used, calculate the amount of biosolids (wet weight, Mg/ha) required for each given kg N/ha application rate (see Table 1 for SI units and Table 2 for non-SI units).
- 2) Given a subplot with a set length and width, and given a set number of biosolids rows that can be incorporated into the subplot, determine the amount of biosolids (wet weight, Mg/ha) that needs to be placed into each row of a given length to meet the application rate (see Table 3 for SI units and Table 4 for non-SI units).
- Given a set length and width for each row, determine how many wet Mg of biosolids will be included for each unit depth (i.e., 1 cm) of a row.
- 4) Based on the calculations performed in Steps 2 and 3, determine the depth of biosolids needed in each row, regardless of row length, for each application rate (see Table 5 for SI units and Table 6 for non-SI units). Note: It is important to determine the required depth of biosolids independent of row length because the length of the subplots will be different depending upon the tree density being used.
- 5) Based on the actual depths of biosolids applied and the number of biosolids rows used in each subplot, back-calculate the actual application rate used in this experiment (see Table 7 for SI units and Table 8 for non-SI units).

rate design									
Required	% N	Mg N/wet Mg	kg N/wet Mg Biosolids	Wet Mg/ha needed for					
N loading	(wet	Biosolids	= (0.0116*1000kg N/Mg	required N loading rate					
rate (kg	weight)	$=(1.16 \div 100)$	N)	$= \{ kg N/ha required \div$					
N/ha)				kg N/wet Mg biosolids}					
20,160	1.16	0.0116	11.6	$20,160 \div 11.6 = 1738$					
40,320	1.16	0.0116	11.6	$40,320 \div 11.6 = 3476$					
60,480	1.16	0.0116	11.6	$60,480 \div 11.6 = 5214$					
N=total nitrogen; ha = hectare; Mg = megagram = 1000 kg = metric tonne									

Table 1. Determination of biosolids application rates needed to meet nitrogen loading rate design (S.I. Units).

Table 2. Determination of biosolids application rates needed to meet nitrogen loading rate design (non-S.I. Units).

Required	% N	Tons N/WT Biosolids	lbs N/WT Biosolids =	WT/Acre Needed for			
N loading	(wet	$=(1.16 \div 100)$	(0.0116*2000lbs/ton)	required N loading rate			
rate (lbs	weight)			= {lbs N/acre required ÷			
N/acre)				lbs N/WT biosolids}			
18,000	1.16	0.0116	23	$18,000 \div 23 = 783$			
36,000	1.16	0.0116	23	$36,000 \div 23 = 1565$			
54,000	1.16	0.0116	23	$54,000 \div 23 = 2348$			
lbs = pounds; N=total nitrogen; WT = wet tons (i.e., U.S. Ton = 2000 lbs)							

With these wet Mg/ha (and wet tons/acre) values and a subplot area 21.9m (72 ft) wide by 21.3m (70 ft) long, the wet Mg needed per 21.3m row based on a set number of rows can be estimated (see Table 3 for data in S.I. units and Table 4 for non-S.I. units). Although the original plan was to install 12 biosolids rows within each 21.9m (72ft) width plot, it also was recognized that soil stability issues might require wider row spacing. For this reason, the calculations below consider both 11 and 12 rows per 21.9m plot width.

Units).								
Required	Wet Mg/ha	21.3m x	Sample	Wet Mg/Plot =	Number of	Wet Mg		
Ν	needed for	21.9m	Plot Area	(wet	21.3m Rows	Needed Per		
loading	required N	Sample	(ha) =	Mg/ha*ha/Plot)	Anticipated	21.3m Row		
rate (kg	loading rate	Plot Area	$m^{2}*1.0E^{-4}$		per 21.9m	(wet Mg/Plot ÷		
N/ha)		(m^2)	ha/m ²		width plot	#rows/plot)		
20,460	1738	468	0.0468	81.3	11 or 12	7.4 or 6.8		
40,320	3476	468	0.0468	162.7	11 or 12	14.8 or 13.6		
60,480	5214	468	0.0468	244	11 or 12	22.2 or 20.4		
N=total nitrogen; ha = hectare; Mg = megagram = 1000 kg = metric tonne								

Table 3. Determination of wet Mg of biosolids required per 21.3m (70 ft) row (S.I. Unite)

e mesji								
Required	Wet	70' x 72'	Sample Plot	Wet Tons/Plot =	Number of	Wet Tons		
N loading	Tons/acre	Sample	Area (acres)	(WT/acre*acres/Plot)	70' Rows	Needed Per		
Rate (lbs	needed for	Plot Area	=ft ² *22.9E ⁻⁶		Anticipated	70' Row		
N/acre)	required N	(ft^2)	acres/ft ²		per 72'			
	loading rate				width plot			
18,000	783	5040	0.1157	90.6	11 or 12	8.2 or 7.5		
36,000	1565	5040	0.1157	181.0	11 or 12	16.5 or 15.1		
54,000	2348	5040	0.1157	271.6	11 or 12	24.7 or 22.6		
lbs = pounds; N=total nitrogen; WT = wet tons								

Table 4. Determination of wet tons of biosolids required per 70-foot row (non-S.I. Units).

The next step is to determine the number of wet Mg that each cm depth of the trench can hold. From this calculation, the required depth of biosolids needed for each application rate can be determined. With a trench width of 1.067m, length of 21.3m, and 0.01m-unit depth, the unit volume is 0.2272m³. This volume can be converted to a weight measurement with the following conversion factors (University of Missouri Extension, 2006; EPA, 1995; Knute, 1986).

Given: 1 $\text{ft}^3 = 62.4 \text{ lbs of biosolids}$ (Note: this assumes the biosolids density is similar to that of water. Biosolids density estimates range from approximately $62.4 - 75 \text{ lbs/ft}^3$, dependent upon percent moisture and other factors.)

Given: $1 \text{ ft}^3 = 0.0283 \text{ m}^3$

Then: $0.0283 \text{ m}^3 = 62.4 \text{ lbs of biosolids}$

Then: $1 \text{ m}^3 = 2,205 \text{ lbs of biosolids}$

Given: 1 Mg = 2,205 lbs

Then: $1 \text{ m}^3 \text{ biosolids} = 1 \text{ Mg biosolids}$

Using the 0.2272m³ volume determined above for a one-cm depth of a biosolids row:

 $0.2272m^3 *1$ Mg biosolids/m³ biosolids = 0.2272 Mg biosolids per cm of a 21.3m length row.

Using the wet tons needed per 21.3m row (from the last column of Table 3) for each of the three application rates, the depth of biosolids in each row (regardless of row length) can be determined as shown in Table 5.

	Wet Mg Needed	Wet Mg Biosolids	Cm of biosolids needed =
	per 21.3m row	per 1cm unit depth	Wet Mg needed ÷ Wet Mg per cm
		of 21.3m length row	
12 rows	6.8	0.2272	$6.8 \div 0.2272 = 30.0$
	13.6	0.2272	$13.6 \div 0.2272 = 59.8$
	20.4	0.2272	$20.4 \div 0.2272 = 89.8$
11 rows	7.4	0.2272	$7.4 \div 0.2272 = 32.7$
	14.8	0.2272	$14.8 \div 0.2272 = 65.3$
	22.2	0.2272	$22.2 \div 0.2272 = 98.0$

Table 5. Determination of cm of biosolids needed per application rate (S.I. Units).

To determine this same information in non-S.I. units, a one-inch biosolids depth is used as the standard unit. With a trench width of 3.5ft, length of 70ft and 0.0833ft (1-inch) unit depth, the unit volume is 20.42ft³. This volume can be converted to a weight measurement with the conversion factors listed previously.

Given: $1 \text{ ft}^3 = 62.4 \text{ lbs of biosolids}$ Then: $20.42 \text{ ft}^3 * 62.4 \text{ lbs biosolids/ ft}^3 = 1274 \text{ lbs biosolids}$ per 1-inch depth of a 70ft row. 1274 lbs biosolids * 1Ton/2000lbs = 0.637 wet tons biosolidsper 1-inch depth of a 70 ft row. Using the wet tons needed per 70ft row (from the last column of Table 4)

for each of the three application rates, the depth of the biosolids in each row (regardless of row length) can be determined as shown in Table 6.

	Wet Tons Needed per	Wet Tons Biosolids	Inches of biosolids needed =
	70ft row	per 1-inch unit	WT needed ÷ WT per inch
		depth of 70ft row	
12 rows	7.5	0.637	$7.5 \div 0.637 = 11.8$
	15.1	0.637	$15.1 \div 0.637 = 23.7$
	22.6	0.637	$22.6 \div 0.637 = 35.5$
11 rows	8.2	0.637	8.2 ÷ 0.637 = 12.9
	16.5	0.637	$16.5 \div 0.637 = 25.8$
	24.7	0.637	$24.7 \div 0.637 = 38.7$

Table 6. Determination of inches of biosolids needed per application rate (non-S.I. Units).

Although it was originally anticipated that 12 rows could be installed within each 21.9m (72ft) subplot width, once installation commenced, it proved to difficult to maintain stability between rows at this density. Consequently 11 rows were installed within each 21.9m subplot width. Actual depths of biosolids installed, and the adjusted actual nitrogen application rates are provided in Tables 7 and 8 below.

А	В	С	D	Е	F	G	Н
Actual	Wet Mg	Wet Mg	# Rows	Back	Subplot	Back	Back
Depth	Biosolids	per	per	calculated	Area	calculated	calculated
Used	per cm of	21.9m	subplot	Wet	(ha/subplot)	Wet Mg/ha	kg N/ha
(cm)	21.9m row	Row =		Mg/subplot		= E/F	= 11.6*G
		A*B		= C*D			
31.7	0.2272	7.20	11	79.4	0.0468	1694	19,650
63.5	0.2272	14.39	11	158.7	0.0468	3388	39,300
95.2	0.2272	21.59	11	238.1	0.0468	5082	58,954

Table 7. Back calculation of kg N/ha applied (S.I. Units).

Table 8. Back calculation of lbs N/acre applied (non-S.I. Units).

А	В	С	D	Е	F	G	Н
Actual	Wet Tons	Wet	#	Back	Subplot	Back	Back
Depth	Biosolids	Tons per	Rows	calculated	Area	calculated	calculated
Used	per inch of	70ft	per	Wet	(acres/subpl	Wet	lbs N/acre
(in.)	70 ft row	Row =	subplo	Tons/subplot	ot)	Tons/acre	= 23*G
		A*B	t	= C*D		= E/F	
12.5	0.64	7.96	11	79.4	0.1157	757	17,418
25.0	0.64	15.93	11	158.7	0.1157	1515	34,837
37.5	0.64	23.89	11	238.1	0.1157	2272	52,255

The average percent solids in the biosolids over the course of application averaged 28.4%. The Wet Mg/ha (wet tons/acre) specified in Tables 7 and 8 produces application rates of 481, 962, and 1443 dry Mg/ha (215, 430, 645 dry tons/acre). Nitrogen content averaged 1.16% wet weight, consistent with values obtained during the design phase of the experiment.

Planting of Hybrid Poplar Clones

Due to an extremely wet spring, planting was delayed from the usual April/May schedule until June 2003. Unrooted cuttings of the OP367 cultivar of hybrid poplar clone (*Populus deltoides* x *Populus nigra*) were obtained from Broadacres Nursery in Hubbard, Oregon. When refrigerated, they can be maintained in a dormant state until planting time. Cuttings were prepared for planting by removing them from refrigerated conditions and soaking in water for several hours. Trees were planted on 3m x 3m (10 ft x 10 ft) spacing for 435 trees/acre and 3m x 4.6 m (10 ft x 15 ft) spacing for 290 trees/acre. Each experimental subplot extends approximately 22m (72 ft) in an east-west direction and either 32m (105 ft), 21.3m (70 ft), or 10.7m (35 ft) in a north south direction for the 435, 290 and 0 trees/acre densities, respectively (see Figure 4).

The standard planting technique used at ERCO consists of attaching a subsoiling bar to a bulldozer and etching a set of 0.3m deep lines that are 3m (10 feet) apart and parallel to one another in one direction of the field, followed by etching a similar set of lines perpendicular to the first set. This creates a square grid over the field. At the intersection of the lines, a tree cutting is planted by hand, resulting in 3m x 3m spacing (i.e., 1074 trees/ha or 435 trees/acre). Tree rows are not intentionally situated directly over the biosolids rows, though in some instances this may occur by coincidence. Over 20 years

of experience at the ERCO site definitively shows that tree roots will naturally grow towards the water and nutrient source. Consequently, positioning trees over the biosolids would incorporate an unnecessary layer of planning into the planting process. In addition, it would only be possible if the tree row spacing were the same as the biosolids row spacing.

Because some of the soil water sampling equipment had been installed prior to planting, it was not possible to use a tractor or bulldozer to cultivate the field with a subsoiling bar as described above. Instead, rows were delineated by hand with a tape measure and the planting locations were marked with spray paint. A dibble bar was used to create a hole for the approximately 0.3m long cutting. Between 2/3 to 3/4 of the cutting length was placed in the hole, which was then packed with dirt to seal out air and create close contact between the surface of the tree cutting and the soil. Figures 11 - 14 depict the planting procedure.



Figure 11. Laborer with dibble bar.


Figure 12. Dibble hole and poplar cutting.



Figure 13. Cutting in ground.



Figure 14. Cutting with initial leaf growth.

In summer 2003 and spring 2004, pre-emergent herbicides were applied in 3-foot strips along each side of the trees to reduce competition from weeds. In spring 2004, tree mortality was assessed. Any trees that had died were removed and replaced with new cuttings.

Pan Lysimeter Installation

Pan lysimeters were assembled at the University of Maryland Biological Resources Engineering machine shop. Pans were constructed from stainless steel sheets (ASTM-176-B7) to form a container with a square, open top and sloping underside to deliver collected soil water to one point underneath the pan. Stainless steel sheets were welded to the interior of the pan across the length and width to provide structural support. A cast stainless steel threaded T-fitting was attached under the deep end of the bottom of the pan to provide an access port for the sampling line positioned below the bottom of the pan. A stainless steel wire mesh lid (304 stainless steel 0.120 inch diameter, woven plain square weave, 2x2 inch mesh, 0.380 inch opening) was placed over the top of the pan to provide additional structural support. Nylon window screening was sewn on top of the wire mesh lid to filter out smaller particulates.

Prior to installation, pans and screens were cleaned by wiping them down with isopropyl rubbing alcohol, followed by a distilled water rinse and then a deionized water rinse. Pan lysimeters were determined to be capable of holding approximately 10 liters of liquid. Overall shape and dimensions of the pan (not drawn to scale) are shown in Figures 15 and 16. Pictures of the pans are provided in Figures 17 and 18.



Figure 15. Front view of pan lysimeter.



Figure 16. Side view of pan lysimeter.



Figure 17. Pan lysimeter top view.



Figure 18. Underside of pan lysimeter and protective wire mesh lid (on left).

Pan lysimeters were installed between July 2002 and March 2003, concurrent with the construction of biosolids rows at the research plot. Details of each installation process are provided in Appendix 1. Once the requisite length of biosolids row was complete, a measuring wheel was used to determine the appropriate section of the row under which the pan needed to be located (i.e., the interior zone of the subplot, inside the buffer perimeter as discussed in the Experimental Design section). An installation trench was dug using a backhoe to a depth sufficient for workers to comfortably stand while drilling 0.3m below the bottom of the biosolids row. The installation trench was dug parallel to the biosolids row as close to it as possible without compromising the integrity of the installation trench wall (approximately 0.61m (2 feet)).

The specific depth of the biosolids row at the installation location was determined by sinking a steel T-handled point bar into the biosolids row until it reached the bottom of the row. The overburden covering the biosolids row was loosened from digging and was easily penetrated, as was the biosolids. The resistance to movement upon reaching the bottom of the biosolids row/mine spoil interface, however, was markedly higher than when the point bar was sliding through the biosolids. The bar depth at this interface was marked, measured, and used as the basis for calculating pan installation depth. These measurements also served as a comprehensive quality control check for the biosolids row construction process, and demonstrated good agreement with the designed depths.

For the control subplots, the depth of installation was the same as the design depth for the lowest application rate. Using a designed row depth of 0.61m, plus the 0.30m distance between the bottom of the row and the pan, pans were installed at a depth of 0.91m under the surface.

As stated in the Experimental Design section, the research plot and surrounding acreage had previously received an application of biosolids under the standard ERCO regimen. Consequently, old biosolids rows ran perpendicular to (though not necessarily at the same depth as) the new rows being laid out for this experiment. The old biosolids rows had been subject to years of dewatering, microbial conversion, and tree root infiltration. In this decomposed state, they often served as preferential flow paths for subsurface flow. Dependent upon the application rate and corresponding depth of the new row, the old rows could be either above, at the same level as, or below the vertical position of the pan. It was therefore important to horizontally position each pan lysimeter

equidistant from the two bracketing old rows to ensure the pans captured flow migrating from and around the new biosolids rows. Figure 19 graphically depicts this concept.



Figure 19. Cross-sectional view of installation trench wall.

Upon determining the pan installation depth with regard to the new biosolids row and horizontal pan position with regard to old biosolids rows, a rectangle 0.56m wide x 0.15m tall was etched into the installation trench wall to guide drilling. Drilling distance into the trench wall was calculated as the distance between the installation trench wall and biosolids trench wall (usually 0.6m) plus the length of the pan lysimeter (0.53m), for a total drilling distance of approximately 1.13m. Basic installation steps are as follows.

- Drill the pan installation cavity. The drill used was a Milwaukee 350 rpm, ³/₄ inch Super Hole Shooter with pipe handle (Catalog No. 1854-1). A generator provided the electrical power. The original drill bit used was a modified bulb and plant auger with 6.35cm (2.5 inch) diameter flights and 61cm (24 inch) length. This broke within a minute of drilling. Trailer anchors with 15.24cm (6 inch) flights were then used. The end of the anchors with the bolt bracket was cut off so it would fit in the drill bit. This modified auger proved much more sturdy and could withstand the drilling pressure. Prior to subsequent installations, teeth were welded onto the first flight in the auger to give it more cutting power.
- Clean out and shape the cavity using shovels to fit the dimensions of the pan lysimeter.
- Test the pan fit by sliding the pan into the cavity and ensuring that the ceiling wall of the cavity allows for a flat, level placement of the top of the pan.
- Rinse the pan to remove any mine spoils and other particulates that may have entered the pan while out in the field. Fit the wire mesh and window screening (which was previously sewn to the wire mesh) into the top lip of the pan lysimeter. Collect an equipment blank on the pan and screens by rinsing with deionized water. Permanently plug the bottom opening of the pan with a 2.66cm (³/₄ inch) threaded PVC plug using PVC primer and glue. Screw another

2.66cm (¾ inch) threaded PVC plug into the sampling portal to temporarily cover the opening.

- Slide the pan into the cavity until it reaches the back wall. In some installations, a layer of playground sand was placed on the bottom of the cavity to facilitate sliding of the pan into the cavity.
- One person would hold the top of the pan flush against the ceiling of the cavity while another person would use the previously removed cavity contents to tightly repack the cavity and hold the pan in place.
- Once the back end of the pan was secure enough to remain flush against the ceiling without assistance, the plug was removed from the sampling portal and permanently replaced by gluing a 2.66 cm (³/₄ inch) threaded PVC coupling. A length of 2.66cm (³/₄ inch) straight PVC pipe measured to reach a couple of inches from the installation trench wall was then glued into the PVC coupling. The pipe was propped level with packed mine spoils (and in some cases a crushed soda can) to ensure it did not have a downward slope in the direction of the trench wall.
- A 2.66cm (¾ inch) 45° PVC elbow joint was glued to the end of the PVC pipe.
- Another, usually smaller length of straight PVC pipe was cut and glued to the other end of the 45° elbow. Another 45° elbow was glued to the other end of the shorter PVC pipe. At this point, the PVC pipe and connections reached out of the installation cavity next to the wall, and the second 45° elbow was pointing straight up.

- A final 3.05m (10 ft) straight PVC pipe was glued to the second 45° elbow to provide a continuous, protected watertight conduit through which sampling line could be threaded into the pan lysimeter for sample collection.
- A 2.66cm (¾ inch) PVC cap was placed on top of the pipe to close the pipe network.
- The remainder of the pan installation opening was tightly repacked with the native soil that had been drilled out to make the opening. The remainder of the installation trench was then refilled and tightly packed with the original contents.
- Soil core samples were collected along the installation trench wall (or along the opposite side of the installation trench if space constraints necessitated). One core was collected in line with the pan lysimeter location; one core was collected 30cm (12 inches) above the first soil core; the third core was collected 60 cm above (24 inches) above the first soil core. These were used to evaluate soil properties, specifically hydraulic conductivity.
- The installation trench was filled with the previously excavated mine spoils. As the trench was refilled, the ground was tamped with both the backhoe bucket and a hand tamper to ensure the soil around the opening of the pan cavity would not be looser than the surrounding soil.

A pictorial layout of the pan installation is provided in Figures 20-25. While some installations were completed with relative ease within 4 hours, others proved more problematic. In some cases, heavy subsurface water flow into the trench from the old biosolids rows flooded the bottom of the installation trench. Submersible pumps were

installed to clear out the water, though regardless installations were more complicated given the wet conditions. In other instances, soil instability and texture made drilling difficult. In a few cases, the integrity of the pan lysimeter cavity or the installation trench could not be maintained during the drilling process and the site had to be abandoned, at which point a new installation location was determined under a different biosolids row within the subplot. Figures 26-29 depict the pan positions as well as the suction lysimeter positions at the experimental site.

Each installation trench provided insight into the varying soil profile in each of the subplots. As previously stated, the southernmost experimental area (Block 3) was characterized by higher clay content, with increasing sand and gravel present from Block 2 to Block 1. While in general this overall trend was representative, the soil profile within each installation trench was variable. Block 3 subplots had pockets of sandier soils and Block 1 subplots contained pockets of clay soils. In addition, the old decomposed biosolids rows introduced yet another soil characteristic into the profile. These observations demonstrate that soil composition varies with depth at this site, and encompasses a wide range of characteristics. All installation measurements and conditions were recorded and transferred to installation diagrams and a summary table. These pan-specific installation diagrams and the accompanying table with details about the installations are provided in Appendix 1.



Figure 20. Drill with modified trailer anchor as auger (pan and screening in background).



Figure 21. Installation trench wall with etched outline of pan installation cavity.





Figure 22. Drilling out pan outline.



Figure 24. Pan installed with attached PVC piping.

Figure 23. Drilled hole with fines for repacking.



Figure 25. Front view of PVC piping exiting pan cavity.



Figure 26. Experimental plot layout with pan and suction lysimeter positions.



Figure 27. Block 3 pan and suction lysimeter installation locations.



Figure 28. Block 2 pan and suction lysimeter installation locations.



Figure 29. Block 1 pan and suction lysimeter installation locations.

Suction Lysimeter Installation

Suction lysimeters were installed from July-August 2003, after all biosolids had been applied to the experimental site and trees had been planted. An itemized listing of installation dates and conditions is provided in Appendix 1. The pressure/vacuum soil water sampler (a.k.a., suction lysimeter) used in this experiment was obtained from Soilmoisture Equipment Corp. (product no. 1920F1L12B02M2). The sampler was 30cm (12 inch) long, with a 4.83cm (1.9 inch) outer diameter PVC body and an epoxy bonded 200kPa (2 bar) porous ceramic cup (1.1*um* pore size) on one end. The other end of the PVC body was capped and threaded with nylon compression fittings through which customized lengths of access tubes (0.64cm {0.25 inch} outer diameter polyethylene tubing) were attached. A plastic dip tube inside the sampler was attached to the underside of the cap directly under the one of the nylon fittings and extends down the PVC body into the ceramic cup.

The suction lysimeter collects soil water held in the soil profile under matric forces by pulling the water through the ceramic cup while under tension. After a requisite period of time, the tension is discontinued. The collected soil water is then purged from the sampler by applying pressure to the access tube, which pushes the collected sample out through the sample recovery tube. A diagram of the suction lysimeter is provided in Figure 30.



Figure 30. Suction lysimeter (pressure-vacuum soil water sampler).

Suction lysimeters were obtained from the vendor assembled; only the access tubes needed to be attached. Green medium density polyethylene (MDPE) tubing (0.65cm outer diameter) was used for the sampling access line and black MDPE tubing was used for the vacuum/pressure line. Approximately 2.4-3.6m lengths of the MDPE tubing were connected to each lysimeter. Neoprene tubing, cut into 10-15cm lengths was placed over the exposed end of the access lines, bent in half, and held in position with a clamping ring. This effectively sealed the access lines until pressure or tension needed to be applied. After attaching access tubes, all suction lysimeters were soaked in water for several hours to prime the ceramic cup. Pressure was then applied with a hand pump through the pressure access tube while clamping off the sample recovery tube. The entire

submerged apparatus was observed to determine if any bubbles were escaping, which would indicate the presence of an unwanted leak. Particular attention was paid to the interface of the PVC tube and ceramic cup as well as the nylon compression fittings. Leaks in the nylon compression fittings were the most common, and were eliminated by adjusting the tightness of the fittings.

As previously noted, installations occurred during the summer of 2003. Installations needed to be within the interior zone of each subplot, inside the buffer perimeter. In addition, suction lysimeters needed to be 3.05m (10 feet) from both the pan lysimeter and the former pan installation trench to prevent any interactions amongst equipment. Typically, four different biosolids rows were needed for the five suction lysimeters required per subplot. One row was used for the two lateral flow lysimeters, which were positioned on either side of a biosolids row. A separate biosolids row was then used for each of the three vertical flow lysimeters.

Lateral flow lysimeter installations required that both edges of the biosolids be delineated. One lysimeter was then installed 0.15m (6 inches) from one side and the other lysimeter was installed 0.3m (12 inches) from the other side to evaluate lateral flow over two distances. The two distances were randomly assigned to the east and west sides of the biosolids row. These lysimeters were installed on either side of the biosolids row in the mine spoils at a depth equal to that of the bottom of the biosolids row. Diagrams of the suction lysimeter positions within each subplot are provided in Figures 26-29 in the prior section on Pan Lysimeter Installation. Details regarding the individual installations are provided in tabular format in Appendix 1.

Suction lysimeters were soaked in water for several hours right before installation and tested a second time to ensure no leaks were present. The standard protocol for installation of the suction lysimeter was consistent with that provided in the 1920F1/1920F1K1 Pressure-Vacuum Soil Water Samplers Operating Instructions Manual (Soilmoisture Equipment Corporation, 1997) and is summarized below. The installation process at the site is depicted in Figures 31-46.

- Identify the appropriate area within which installations are to be performed (within inner subplot).
- Place plastic tube protectors over trees in the delineated area.
- Using a backhoe, carefully uncover between 0.3-0.6m of overburden in an east-west direction within a subplot until the top of one or two biosolids rows can be identified with a point bar. Note: Because installations took place in the summer, the experimental area contained a natural cover crop of native grasses and weeds that were dug up during this process.
- Determine the specific depth of the biosolids row at the installation location by sinking a steel T-handled point bar into the biosolids row until it reaches the bottom of the row. Evaluate whether or not an old biosolids row is underneath the new biosolids row. Given that the old biosolids rows are often less dense than the new biosolids row, the point bar will sink significantly deeper than the designated depth of the new biosolids row. Move to a different location in the biosolids row if this occurs because the suction lysimeters are not to be installed within an old row.
- If the lateral flow suction lysimeters are being installed, the east and west edges of the biosolids trench also need to be delineated using the point bar. Measure 0.15m

and 0.30m from the designated east and west side of the trench walls to identify the installation locations.

- If the vertical flow suction lysimeters are being installed, only the depth to the bottom of the biosolids row needs to be determined.
- Note: The equipment used to drill the installation hole included 1) a Little Beaver® 11 hp hydraulic earth drill with a 2-man handle as well as 2) a hand auger with a 9-10cm diameter. A level was periodically placed on top of the hydraulic drill handle to ensure a vertical hole was being drilled.
- Excavate a hole to the appropriate depth with the hydraulic drill. Periodically pull up the auger and empty the residue out of the hole. Save it for repacking the cavity.
 - Ø When installing the lateral flow lysimeters, drill in the mine spoils to adepth equal to the bottom of the biosolids row.
 - Ø When installing the vertical flow lysimeters, drill through the biosolids row and then either 0.15m, 0.30m, or 0.60m beneath the row.

Note: The excavated holes from both the mine spoils and biosolids material remained intact during the drilling and installation process and did not require additional wall support to prevent collapse. This made the installation process much simpler than originally anticipated, especially with regard to the vertical installations directly underneath the biosolids rows.

• Approximately 5-10 cm before reaching the bottom of the lysimeter cavity, switch over to a hand auger to reach the final depth and to better clean out the bottom of the cavity. Monitor depth periodically throughout the drilling process with a tape measure.

- Sift the overburden drilled from the installation cavity through a sieve to remove particles >2mm and to produce a relatively uniform backfill soil.
- Pour approximately 200mL of distilled water down the hole and, using a wooden stake (or similar implement), mix the water with backfill soil to make some mud at the bottom of hole.
- Make some mud in a bucket by mixing screened spoil that was excavated from the hole with distilled water.
- Pack the mud in the bucket around the ceramic cup of the suction lysimeter. This will create a hydraulic seal that will promote good flow of soil water through the ceramic cup.
- Taking care to keep the mudpack around the ceramic cup, lower the suction lysimeter into the installation hole. Push the lysimeter into the mud at the bottom of the hole. The mud should fill the hole slightly above the ceramic cup.
- Fill the hole around the lysimeter with more mud and backfill to between one-third to halfway up the lysimeter.
- Take approximately 3-5 handfuls of dry bentonite and deposit in the hole to make a ring around the lysimeter. The bentonite will expand as it makes contact with the mud and absorbs moisture. It will expand and form an impermeable collar around the lysimeter column.
- Backfill the hole with native soil, packing the soil while filling. When the level of
 packed backfill is just under the top cap of the lysimeter, pour more dry bentonite
 around the lysimeter to make a second bentonite plug at the top of the lysimeter.
 These bentonite plugs will seal the area around the lysimeter to prevent water from

selectively passing down the drilled hole through any fissures that could not be completely packed up. This will ensure that the soil water collected through the ceramic cup is representative of the leachate that percolated through the biosolids and soil profile to the depth of the lysimeter.

- Fill the remainder of the hole with backfill, carefully packing it around the lysimeter to prevent the creation of preferential flow paths.
- Move the lysimeter access hoses to the sides of the trench and fill in the trench with the backhoe. Tamp the trench as it is being filled.
- Use plastic cable ties to attach the exposed portions of the access tubes to wooden stakes that are securely hammered into the soil.
- After each installation, remove any remaining residue from the augers used in the drilling process.

As with the pan lysimeters, for the Control subplots, the depth of installation was the same as the design depth for the lowest application rate. Using a designed row depth of 0.61m, suction lysimeters were installed at the appropriate depths in relation to the theoretical biosolids row.



Figure 31. Removing overburden to locate biosolids rows.



Figure 32. Biosolids rows.



Figure 33. Measuring biosolids depth.



Figure 34. Drilling equipment.



Figure 35. Drilling hole.



Figure 36. Drilled hole in control subplot.



Figure 37. Laterally placed lysimeter hole (to the side of the biosolids row).



Figure 38. Vertically placed lysimeter hole (below the biosolids row).



Figure 39. Measuring depth.

Figure 40. Cleaning out bottom of hole with hand auger.



Figure 41. Sifting drilled soil.



Figure 42. Sieve used for sifting.



Figure 43. Placing mudpack around ceramic cup.



Figure 44. Pouring bentonite around lysimeter to create a watertight plug.



Figure 45. Packing fill around lysimeter.



Figure 46. Lysimeter lines placed to the side of the installation site.

Sample Collection

Soil Water Samples

On a monthly basis, soil water samples were collected from the 30 pan lysimeters and 150 suction lysimeters for testing at a water quality lab. The experimental site was divided into three sets of subplots running in a north-south direction. Each of the three sets was sampled in a given week within the same month. Within a sampling week, pan lysimeters were collected on one day and suction lysimeters were usually collected on a different day. Sample collection for pan lysimeters commenced in April 2003. Sample collection for suction lysimeters commenced in November 2003. This thesis addresses samples collected through December 2004.

Each pan lysimeter was fitted with a dedicated line of polyethylene tubing running down the inside of the PVC pipe that was connected to the pan lysimeter. Samples were drawn out of the polyethylene tubing into a 1-L filtration flask by applying suction on the arm of the flask with a vacuum hand pump. Water was withdrawn until the pan emptied or the estimated volume of the pan (i.e., 10-L) was extracted, whichever occurred first. In some instances, volumes greater than 10-L could be extracted, due to the fact that water recharged more quickly than sample could be removed. In other instances, pans did not contain leachate and a sample could not be collected. For each 1-L of volume collected, 100-mL was sub-sampled and placed in a 1-L high-density polyethylene (more container to produce a composite sample representative of the contents of the pan. This composite sample was transferred to a smaller, high-density polyethylene (HDPE) 125 or 200mL container for delivery to and processing at the laboratory. The total volume and appearance of the sample purged from the pan was recorded. Samples were stored on ice

in a cooler until delivery at the laboratory. Figures 47-52 show the pan lysimeter sample collection process.



Figure 47. Hand pump and sample collection flask for the pan lysimeter.



Figure 48. Collecting a pan lysimeter sample in the flask.



Figure 49. Pouring an aliquot of sample into the compositing container.



Figure 50. Rinsing the sample collection flask with distilled water.





Figure 51. Rinsing the flask with deionized water.

Figure 52. Rinsing the stopper and tubing with deionized water.

As previously shown in Figure 30 above, each suction lysimeter apparatus contains a dedicated pressure-vacuum access tube and discharge access tube. Approximately three to four days prior to the collection date, 60-70 centibars of suction was applied to the vacuum access tube with a hand pump while keeping the discharge access tube closed. The vacuum tube was then closed to maintain suction and draw sample from the soil matrix through the porous ceramic cup of the suction lysimeter. Sample was then recovered from the suction lysimeter by opening both lines, applying pressure to the pressure-vacuum tube, and collecting the sample that ejected from the discharge access tube in a graduated cylinder. The volume and appearance of the sample collected was recorded, and transferred into pre-labeled 125mL HDPE bottles. Samples were stored on ice in a cooler until delivery at the laboratory. Figures 53-58 show the sample collection process.



Figure 53. Hand pump next to suction lysimeter vacuum and pressure lines.

Figure 54. Applying suction to the lysimeter vacuum access tube.



Figure 55. Purging sample by applying pressure.

Figure 56. Sample collected in graduated cylinder.







Figure 58. Rinsing sampling equipment with deionized water.

Both pan and suction lysimeter samples were transported in coolers on ice to the laboratory for further processing. Samples were measured for pH and an aliquot was vacuum filtered through a 0.45um nylon membrane filter to remove particulates. A separate aliquot was preserved with sulfuric acid to pH < 2. All samples were frozen until analyzed.

Biosolids Samples

As mentioned in the experimental design section, during the construction of the biosolids rows in the experimental plot, biosolids samples were collected on a monthly basis to evaluate nutrient and other parameters over time. After being offloaded from the delivery truck, a composite sample was obtained by taking five to seven aliquots from different parts of the biosolids pile and mixing them together in a HDPE sampling container. Biosolids samples were placed on ice and frozen, then delivered to the laboratory for analysis.

Soil Core Samples

During the installation of pan lysimeters, soil core samples representative of the soil profile at and above the pan installation location were collected along the installation trench wall. Soil cores were collected in either 5.4cm diameter, 6cm length brass cylinders with beveled ends or 4.7cm diameter, 5cm length aluminum cylinders. One end of the cylinder was placed on the surface of the soil site to be collected and a wooden block was placed over the other end of the cylinder. The cylinder was driven into the soil by hammering on the wooden block. The filled cylinder was carefully removed from the soil and covered with plastic caps on both ends.

Soil cores were obtained from three different depths in the soil profile for 28 of the 30 subplots. For the remaining two subplots (subplots 3B and 2B), one and two soil cores were collected, respectively. The depth of sample collection was in relation to the position of the pan lysimeter (and therefore the depth of the biosolids row, which correlates to the application rate). Soil cores were collected at 1) the depth of the pan lysimeter installation, 2) 30 cm above the pan depth, and 3) 60 cm above the pan depth. Diagrams depicting the collection location at each installation subplot are provided in Appendix 1.

Rain Gauge Data

A tipping bucket rain gauge connected to a HOBO[®] Event data logger from Onset Computer Corporation was installed on an open rooftop of a trailer adjacent to the west side of the experimental plot. This site was equal to or higher than all other trailers in near proximity and devoid of trees. On a biweekly basis, data from the rain gauge was downloaded in accordance with the protocols for the Onset Computer Corporation's

Boxcar[®] software. Each event recorded by the logger was equivalent to 0.025cm (0.01inch) of rainfall, and was associated with a specific date and time. Rainfall event files from the data logger were imported to excel spreadsheets, converted to cm of rainfall and summed to determine daily totals.

The rain gauge was physically inspected on a monthly basis to ensure all components were unencumbered by insects, spider webs, or debris and that all parts were in working order. When temperatures were below freezing, the heating component of the rain gauge was turned on to ensure any frozen precipitation would be melted and properly recorded *Laboratory Analysis of Soil Water, Biosolids and Soil Samples*

Soil Water Samples

Pan and suction lysimeter samples were transported to the laboratory after collection. Samples were analyzed for pH on a Fisher Scientific accumet Basic AB15 pH meter. An aliquot of sample was vacuum filtered through a 0.45*um* pore size nylon membrane filter (Whatman part no. 7404-004) and frozen until analyzed. Original, unfiltered aliquots were frozen and placed in storage. Filtered samples were analyzed for total nitrogen, ammonium, nitrite, and nitrate. With the exception of some nitrate and nitrate analyses noted below, all analyses were performed by the Appalachian Laboratory at the University of Maryland Center for Environmental Studies in Frostburg, MD. Analytical methods/protocols used included the following.

- Total nitrogen: Standard Methods, Method 4500-N B. In-Line UV/Persulfate
 Digestion and Oxidation with Flow Injection Analysis (APHA, 1998)
- Ammonium nitrogen: Lachet QuickChem Method 10-107-06-3-D, Revision
 Date August 26, 2003 (Sodium salicylate –based method).

• Nitrite/nitrate:

a) Methods for Chemical Analysis of Water and Wastes (MCAWW)
 Method 353.2 Determination of Nitrate-Nitrite Nitrogen by
 Automated Colorimetry (using a Lachet Quick Chem 8000 Flow
 Injection Analyzer) (EPA, 1983). Both nitrite and nitrite+nitrate are
 determined; nitrate is then mathematically calculated as the
 difference.

OR

b) Bran and Luebbe Method 696E-82W (nitrite) and 696F-82W (nitrite+nitrate). These methods are based on Methods 4500-NO₂ B. and 4500-NO₃ H, respectively, from Standard Methods for the Examination of Water and Wastewater (APHA, 1998). Nitrate is mathematically calculated as the difference between Nitrite+nitrate and nitrite.

Note: The Braun and Luebbe method was used for samples collected prior to March 2004, which were analyzed at the University of Maryland's Water Quality Laboratory in the Biological Resources Engineering Department in College Park, MD. Samples collected during and after March 2004 were analyzed using MCAWW Method 353.2 by the Appalachian Laboratory at the University of Maryland Center for Environmental Studies in Frostburg, MD.
Biosolids Samples

Biosolids samples collected on a monthly basis during set up of the experimental plot were delivered to the University of Maryland's Maryland Cooperative Extension Laboratory in College Park, MD. Analytical methods/protocols used included the following.

- A sample aliquot is analyzed for moisture content.
- <u>Ammonium nitrogen:</u> A representative fresh (not dried) aliquot is distilled using MgO (Association of Official Analytical Chemists {AOAC} Section #2.057.
- For all remaining analyses, a sample aliquot is dried at 80°C and ground in a Wiley Mill to pass through a 20 Mesh sieve.
- <u>Organic nitrogen:</u> Leco CHN combustion determination (Campbell, C.R. 1992. In Plant analysis reference procedures for the southern region of the U.S. Southern Cooperative Research Ser. Bulletin 368. USDA, Washington, D.C. pp. 21-23).
- <u>Total nitrogen:</u> The sum of ammonium and organic nitrogen.
- <u>Magnesium, phosphorus, potassium and calcium:</u> Perchloric/Nitric acid digestion followed by Technicon AutoAnalyzer determination (Walsh, L.M., 1971).
- <u>Manganese, zinc, and copper:</u> Perchloric/Nitric acid digestion followed by Atomic Absorption determination (Gorsuch, 1970).
- <u>Sulfur:</u> Leco S132 combustion determination (Leco Application Bulletin 203-601-073).

Soil Core Samples

Hydraulic conductivity was determined on soil cores collected concurrent with the pan lysimeter installations. Analyses were performed in-house at the University of Maryland Biological Resources Engineering Soil Water Laboratory using an adaptation of the constant head protocol delineated in Methods of Soil Analysis (Knute, A. 1986). The protocol is based on Darcy's Law, in which:

$$q = Q/A = -k(\Delta h/\Delta l) \tag{1}$$

where:

q = hydraulic flux

Q = volumetric flow rate = volume of water flowing through core sample (V) for a given time (t) = V/t

A = cross sectional area of the core sample (cylinder). Determine from $\pi d^2/4$.

k = hydraulic conductivity

 Δh = the hydraulic head difference imposed across a sample of length "l"

{i.e., difference in height between the bottom of the Mariotte air tube (i.e.,

bottom of copper tubing) and bottom of brass soil core cylinder}

 Δl = length of the core sample (distance through which the water flows)

t = time

For these experiments, $\Delta h/\Delta l$ was approximately 10.

Solving for k = hydraulic conductivity:

$$-\mathbf{k} = (\mathbf{V}^* \Delta \mathbf{l}) / (\mathbf{A}^* \Delta \mathbf{h}^* \mathbf{t}) \tag{2}$$

In summary, a soil core sample is placed in a Tempe Cell and saturated with water from the bottom up. The Tempe Cell set up was modified to replace the ceramic disk that is normally placed in the bottom of the cell underneath the soil core with a thin, porous hydrophobic polypropylene material made by Porex Corporation. The resistance of the material is orders of magnitude less than the soil samples and hence is neglected in the hydraulic conductivity calculation.

A Mariotte reservoir is filled with water and flushed until air bubbles exit the Mariotte air tube within the reservoir. The Mariotte tube is used to deliver water to a soil column at a constant outlet pressure. Tygon tubing extending from the bottom opening of the Mariotte reservoir is filled with water in the process of flushing. This tubing is then attached to the upper opening of the Tempe Cell without introducing air bubbles to the system. A known pressure head is consequently established with the Mariotte air tube positioned at a known height above the core sample that sits in the Tempe Cell. The spigot at the bottom of the Mariotte reservoir is opened. A steady stream of water from the Mariotte reservoir flows to the Tempe Cell and through the soil core sample. The volume of water flowing through the core sample for a known amount of time is used to determine the hydraulic conductivity based on the equation provided above. The general set-up is shown in Figure 59.



Figure 59. Saturated hydraulic conductivity constant head set up.

Data Analysis

Data were statistically analyzed using analysis of variance (ANOVA) techniques (Kuehl, 2000) to evaluate trends in hydraulic conductivity with depth and location, water quality over time, and whether differences exist in water quality between biosolids application rates and tree densities. SAS 9.1. © 2002-2003 (SAS Institute, Inc., Cary, North Carolina) was used to perform these analyses.

Hydraulic conductivity and nitrogen data were statistically analyzed using analysis of variance (ANOVA) techniques with SAS statistical software (v. 9.1). Hydraulic conductivity results were examined in terms of block and depth. This factorial treatment design was evaluated using PROC Mixed. Significant fixed effects (α =0.05) were then subjected to Least Squares Means evaluation to isolate which interactions were responsible.

Water quality data from the pan and suction lysimeter soil water samples were subjected to the following procedures. The monthly measurements were averaged on a quarterly basis for each subplot. Seasonal quarters were similar, but not exact for the pan lysimeters and suction lysimeters, due to the different start dates for collection activities and the need to have a data set complete enough to successfully run through the statistical procedures. Table 9 identifies which months were consolidated into the seasonal quarters.

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Pan Lysimeter	0	Suction Lysimeter				
Month-Year	Quarter	Month-Year	Quarter*			
April-2003	1	N/A				
May-2003	1	N/A				
June-2003	2	N/A				
July-2003	2	N/A				
August-2003	2	N/A				
September-2003	3	N/A				
October-2003	3	N/A				
November-2003	3	November-2003	4			
December-2003	4	December-2003	4			
January-2004	4	January-2004	4			
February-2004	4	February-2004	4			
March-2004	5	March-2004	5			
April-2004	5	April-2004	5			
May-2004	5	May-2004	5			
June-2004	6	June-2004	6			
August-2004	6	August-2004	6			
October-2004	7	October-2004	7			
December-2004	8	December-2004	7			

Table 9. Quarterly assignments for monthly samples.

As is shown in Table 9, suction lysimeter samples could not be separated into as many quarters as the pan lysimeters for the November 2003 to December 2004 time period. Specifically, the averaging of suction lysimeter (SL) results over more months for quarters 1 and 4 was necessitated by the fact that for each subplot, 5 different positions/depths were represented by the SLs. Samples were not always present each month (or for several months) at a particular lysimeter, and data were therefore not generated during those times. If the data set was not complete enough for a particular position, statistical analyses were in some cases compromised and usable results could not be generated. This necessitated the consolidation of more of the monthly results than with the pan lysimeters.

Non-detect results were set to a value equal to 2/3 of the detection limit (Douglass, L., personal communication, 2005). Data for each analyte were then evaluated to

determine if they met the normal distribution and homogeneity of residuals assumptions of ANOVA. Upon determining that they did not, data were log transformed and again evaluated. Log transformation produced data sets that met the assumptions for ANOVA.

The split plot experimental design and collection of data over time provides a repeated measures data set best analyzed using the Mixed procedure with repeated measures analysis techniques that: 1) estimate the covariance residuals and 2) use the variance and covariance estimates to determine appropriate standard errors and test hypotheses (Douglass, 2005). Six different covariance structures were evaluated to determine which structure best described the random variances and covariances among the repeated measures. These included: compound symmetry (CS), heterogeneous compound symmetry (CSH), first-order autoregressive {AR(1)}, heterogeneous first-order autoregressive {ARH(1)}, spatial power{SP(power)}, first-order ante-dependence {ANTE(1)}, unstructured (UN) (Littel, R.C., et. al., 1996).

Upon determining the most appropriate structure for the data set (i.e., the one with the best goodness of fit measurement), the program was run to evaluate whether or not the null hypothesis was rejected. The null hypothesis and alternate hypothesis were:

 H_o = Treatment effects means and interaction effects means are equal

 H_a = Treatment effect means and/or interaction effects means are not equal

Tests of fixed effects showed which null hypotheses were rejected based on a probability level of 0.05. Those rejected null hypotheses were further evaluated by the least squares difference (LSD) procedure to compare individual treatment means. Significant differences (p < 0.05) from the LSD analysis were then studied to determine if any differences were important in the context of the experiment (Kuehl, 2000; Littel, R.C., et. al., 1996).

Chapter 5. Results and Discussion

Results and the accompanying discussion are presented in the following order:

- Biosolids analysis
- Hydraulic conductivity
- Rain gauge data
- Soil water analysis and results: overview
- Soil water results: total nitrogen and ammonium (NH₄⁺-N) data
- Soil water results: nitrite (NO_2) data
- Soil water results: nitrate (NO₃) data.

Biosolids analysis results provide information about the nutrient content applied during the course of the experimental set up. Hydraulic conductivity values offer insight to the potential flux of water throughout the experimental plot at different depths within the soil profile. Precipitation values from the rain gauge provide further information on hydrologic conditions impacting water percolation in the soil profile. Soil water results are the main focus of this thesis. These include results from the pan and suction lysimeter samples collected to ascertain nitrogen concentrations in close proximity to the biosolids rows.

Biosolids Samples

As stated in the Chapter 4, biosolids were dewatered and lime-stabilized with a pH of approximately 12. Samples were collected on a monthly basis during application to the experimental site to monitor the concentrations of macro and micronutrients. Summary results from the analysis of these samples are presented in Table 10.

Descriptive S tatistic	N (%)	NH4-N (%)	P ₂ O ₅ (%)	K2O (%)	Ca (%)	Mg (%)	S (%)	Mn (ppm)	Zn (ppm)	Cu (ppm)	Moisture (%)
Mean	1.15	0.073	0.84	0.12	3.42	0.09	0.19	49.16	111.04	58.53	71.76
Standard Deviation	0.12	0.05	0.11	0.04	1.25	0.14	0.06	16.70	38.40	11.19	3.55
Coefficient of Variation	10.57	69.66	12.78	35.49	36.67	155.85	31.73	33.96	34.58	19.11	4.94

Table 10. Biosolids analysis results (wet weight basis).

These data were reported by the laboratory on a wet weight basis. Because moisture content varies amongst biosolids, it is useful to report results on a dry weight basis to allow for comparisons with other biosolids. Conversion is performed using the formula: $C_{dry} = C_{wet}$ (100/% solids), where C = concentration of the parameter. Dry weight conversions are presented in Table 11. For reference, those results reported in percent (%) units can be converted to mg/kg units by multiplying the % value by 10,000.

Та	ab	le	1	1.	B	Biosolids	anal	lysis	resul	ts (dry	y wei	igh	t	basis).
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Descriptive Statistic	Moisture (%)	Solids (%) = 100-% M	N(%)	NH4-N (%)	P2O5 (%)	K2O (%)	Ca (%)	Mg (%)	S (%)	Mn (ppm)	Zn (ppm)	Cu (ppm)
Mean	71.76	28.24	4.12	0.27	2.99	0.41	11.94	0.31	0.66	173.55	394.20	207.42
Standard Deviation	3.55	3.55	0.43	0.22	0.36	0.14	3.39	0.43	0.17	53.57	139.15	29.54
Coefficient of												
Variation	4.94	12.56	10.37	83.19	12.16	33.89	28.42	140.58	26.42	30.87	35.30	14.24

Results demonstrated relatively consistent values over time, with magnesium (Mg) and ammonium (NH_4^+) exhibiting the highest relative variability. In both instances, the

high variability is attributed to a single outlier value. For ammonium, a markedly high value of 1.51% (15,100 mg/kg) dry weight was reported for the 3/26/2003 sample, compared to an average value of 0.27% dry weight. For magnesium, a notably high value of 2.85% (28,500 mg/kg) dry weight was reported for the second of two samples collected on 11/27/2003, compared to an average value of 0.31% dry weight. These individual sample results (as opposed to the mean values shown in this section) are presented in Appendix 2. When these outliers were removed, the coefficient of variation decreased significantly for both parameters, becoming comparable to those values reported for other parameters (see Table 12).

Table 12. Biosolids results: revised after removal of outliers (dry weight basis).

Descriptive		
Statistic	NH4-N (%)	Mg (%)
Mean	0.23	0.24
Standard Deviation	0.07	0.04
Coefficient of		
Variation	29.94	17.35

A specific explanation could not be obtained for the two unusually high values. The consistency of all other values indicates that they could be the result of a calculation or transcription error. Another possibility for the outlier ammonium value could be explained by its transient nature under certain conditions. The high pH conditions of the biosolids (pH=11-12) drive the conversion of ammonium to gaseous ammonia (NH₃). Wherever the biosolids are in contact with air, ammonia can escape into the atmosphere.

Adamsen and Sabey (1987) conducted studies in which the ammonia content of surface-applied biosolids was measured at the time of application and at various intervals thereafter. Results showed that 40% of ammonium can be lost via conversion to gaseous ammonia within 2 weeks. Because the biosolids from the ERCO study are stored in a yard at the wastewater treatment plant and moved frequently with a front-end loader, variable amounts of ammonium could be lost during storage or transport of the biosolids. Despite the ammonium and magnesium anomalies, the overriding conclusion is that biosolids of consistent composition and nutrient content were applied to the site throughout the experimental setup.

Hydraulic Conductivity

Soil core results show a wide range in saturated hydraulic conductivity from $1.40 \times 10^{-7} - 1.84 \times 10^{-2}$ cm/sec, reflecting varied soil composition, some with high clay content and others dominated by sand and gravel. This range is consistent with visual observations during equipment installations at the site. Visual observations indicated higher sand and gravel contents in Block 1, with successive transition over to higher silt and clay content through Blocks 2 and 3. Also noted, however, during equipment installations was the fact that some subplots with sandy soil at the surface had clay layers or pockets further in the soil profile. Similarly, the higher clay content surface in Block 3 would sometimes contain sandier layers and pockets at different depths. Thus, the soil composition was reflective of the extensive disturbance and mixing of overburden that would occur during excavation operations at a gravel mine.

A fresh sample of biosolids was also subjected to the hydraulic conductivity analysis. The hydraulic conductivity measured was 2.55×10^{-6} cm/sec, reflecting properties similar to silty and clay soils. If the soil surrounding the biosolids row has a higher conductivity value than the biosolids, water entering the subsoil system via precipitation will likely travel around the biosolids row. Conversely, if the soil has a lower conductivity value, water will choose the path of least resistance and percolate through the biosolids row. It is also important to note that within the biosolids row the hydraulic conductivity value will change over time as biosolids dewater and decompose. Based on observations of decomposed biosolids at the tree farm as well as the actual water flow in and around old biosolids rows, the conductivity increases as the biosolids age and the old rows serve as conduits.

To better quantify these visual observations, the hydraulic conductivity data were evaluated and subjected to statistical analyses to determine if: 1) significant differences ($\alpha = 0.05$) occurred between the three blocks at the experimental site and 2) significant differences ($\alpha = 0.05$) existed at different depths in the soil profile. Although soil cores were collected at three different depths within each subplot, because those depths varied with biosolids application rate and variances in the topography of the site, the range of depths over which soil cores were collected were separated into four different levels. This allowed for a more consistent comparison across the experimental area based on the standard datum of depth from the surface. The four depth levels consisted of: 30-60cm; 61-94cm; 95-129cm; and 130-168cm. The shallower depths were typically associated with the lowest and middle application rates; the highest depths were almost exclusively associated with the highest application rates.

The two factors under consideration were block (i.e., areas of the experimental plot with differing topographic features and soil composition) and depth. This constitutes a factorial treatment design with three levels of the block factor and four levels of the depth factor. PROC Mixed was used to perform a factorial analysis of variance. Results showed statistically significant differences ($\alpha = 0.05$) between blocks (Pr<0.0001), but not between depths or block*depth interactions. Least Squares Means evaluation showed all three blocks to be significantly different from one another (Pr <0.0031 for Blocks 1 and 2; Pr<0.0001 for Blocks 1 and 3; Pr<0.0038 for Blocks 2 and 3). These results are reflected in plots of the data as shown in Figures 60-62.



Figure 60. Hydraulic conductivity by depth (no significant differences between depths).

As can be seen from Figure 60, as depth increases, hydraulic conductivity neither increases nor decreases in a consistent trend. Higher and lower values exist at both shallow and deep locations within the soil profile. What this demonstrates is the varying nature of the soil composition within the evaluated profile depth. The subsurface stratigraphy of this region indicates that underneath the gravel and sand formations (Upland Deposits and Calvert Formation) there exist the silty clays and clayey sands of the Nanjemoy Formation followed by a confining unit of clays known as the Marlboro Clay (see Chapter 4, Site Location and Characteristics; Wilson and Fleck, 1990). It was originally reasoned that the mining operations would have removed most of the gravel and sand formations, leaving the silty clays and clays exposed at the experimental site, such that with increasing depth, a higher proportion of clays would be encountered in the soil profile.

Mining operations, however, will only remove what is economically feasible, and it is obvious from the visual inspection and soil core analyses that pockets of sand and gravel remain at the mined site, particularly in the north end of the experimental site (Block 1). The range of depths examined in this experiment encompassed the upper four meters of the soil profile (i.e., was limited to the soil profile in proximity to the biosolids rows). The relatively shallow profile considered likely did not cross over different geographic formations within each subplot considered. Furthermore, with all of the soil disturbance inherent to the mining operations, and the fact that this experimental site had previously been subjected to a round of biosolids application, significant alteration of the profile had already occurred. Were there originally a trend of increasing clay content with depth reflecting different stratigraphic regions in the upper 4m of the profile, they may have been mixed enough to render them indistinguishable.

Figures 61 and 62 show hydraulic conductivity by block. Figure 61 emphasizes the marked differences between blocks, the most notable difference belonging to Block1, with the highest overall values. Block 1 is located on the north end of the experimental plot, is approximately 10-15 feet lower in elevation than Block 3, and is characterized by high sand and gravel content. Figure 62 shows the same data, but with a log transformed scale to make the lower end of the scale more visible.



Figure 61. Hydraulic conductivity by block.



Figure 62. Hydraulic conductivity by block (logarithmic scale).

Though not a specific goal during the design of the experiment, the vast range of soil conditions encountered has expanded the scope to examine nutrient fate and transport in a much wider variety of soil types. Consequently, results will provide valuable information about whether or not this reclamation technique is environmentally feasible not only in high clay content soils, but in sandier soils as well.

To put the hydraulic conductivity measurements in perspective with the experimental layout, the average hydraulic conductivity was computed for each subplot, and the subplots were color coded to reflect ranges in values, as shown in Figure 63. Individual hydraulic conductivity results are provided in Appendix 2.



Figure 63. Experimental plot color-coded with average hydraulic conductivity categories.

Hydraulic conductivity values provide insight into the amount of time it would take for water leaching from the biosolids to reach sampling equipment. Using the results from the deep soil core from each subplot, which represent the soil conditions under the biosolids row in the vicinity of the pan and suction lysimeters, it is possible to estimate leachate travel time. Transmission of water through porous media is described using Darcy's Law:

$$q = -K dH/dx$$
(3)

where,

- q = volume flux density of water, i.e., the volume of water V passing through a unit cross sectional area A, that is perpendicular to the flow direction (L/T),dH/dx = hydraulic head gradient (L/L), and
- K = hydraulic conductivity, i.e., the ability of the conducting medium to transmit the liquid (L/T).

Hydraulic conductivity is directly proportional to flux and is dependent upon pore size, tortuosity, and fluid properties including viscosity and density. It can be used to estimate the rate of soil water flow if 1) seepage through the soil is assumed to be due to gravimetric forces alone and 2) saturated flow conditions are represented (Hillel, 1998). It also assumes that no preferential flow exists. Using the measured hydraulic conductivities and the 30 cm distance between the bottom of the biosolids row and the pan lysimeter (as well as the mid-depth vertically positioned suction lysimeter), leachate travel times were determined for each subplot and are presented in Table 13 (in subplot ID and travel time order) and Figure 64 (in travel time order).

The assumptions inherent to this analysis are not unreasonable for the study period under consideration. Past studies on biosolids trenching and deep row application show that biosolids rows actively dewater, especially during the first two years after application (Sikora, et al., 1982). Therefore, the soil directly underneath the trenches would likely become saturated. Whether or not the soil remains continuously saturated, though, would be dependent upon the rate of leaching from the biosolids versus the rate of travel through the soil. Although matric forces will be present in concert with gravimetric forces, given the documented observations of downward dewatering occurring in biosolids trenching studies, gravimetric forces likely dominate, at least initially. It is important to note that these assumptions will become less valid over time as the most intense biosolids dewatering subsides, and as tree roots infiltrate the area around the biosolids rows and actively divert gravimetric flow. The estimates presented could be skewed if preferential flow paths existed when the soil samples were collected (which would impact the hydraulic conductivity values). For example, the fissures created when the clay-dominated soil dried would serve as conduits for rainfall infiltration until the clay swelled from moisture absorption.

Conversely, the estimates may not represent future travel times, given that the hydraulic conductivy values used do not account for preferential flow paths that may develop over time. As a result, these are crude approximations of travel time. Regardless, the hydraulic conductivity values provide a basis for comparing the differences in transport over the experimental site.

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Sorted by Subplot ID							
	Time required to travel 30 cm from bottom of						
Cubulat ID	biosolids row to	Time (down)					
	lysimeter (nour)	(days)					
1A 1D	2.70	27.02					
10	2.00	0.12					
10	2.90	0.12					
1D 1E	54.02	0.02					
1E 1F	110.92	<u> </u>					
1G	3 36	0.14					
1H	2.30	1 1/					
11	21.33	0.09					
2A	113 33	4.72					
2B	92.29	3.85					
<u>2C</u>	3.80	0.16					
20 2D	194.31	8.10					
2E	9354.81	389.78					
 2F	159.89	6.66					
2G	184.49	7.69					
2H	178.59	7.44					
2I	18.14	0.76					
3A	653.59	27.23					
3B	829.06	34.54					
3C	89.72	3.74					
3D	2708.36	112.85					
3E	84.79	3.53					
3F	1544.84	64.37					
3G	50.66	2.11					
3Н	14.70	0.61					
3I	55.61	2.32					
4A (Block 3)	63.96	2.66					
4B (Block 2)	64.60	2.69					
4C (Block 1)	4.40	0.18					

Table 13. Biosolids leachate travel time calculated for each subplot.

Sorted by Travel Time								
Subplot ID	Time required to travel 30 cm from bottom of biosolids row to lysimeter (hour)	Time (days)						
1D	0.47	0.02						
11	2.12	0.02						
1A	2.78	0.12						
1C	2.90	0.12						
1G	3.36	0.14						
2C	3.80	0.16						
4C (Block 1)	4.40	0.18						
3H	14.70	0.61						
2I	18.14	0.76						
1H	27.33	1.14						
3G	50.66	2.11						
1E	54.02	2.25						
3I	55.61	2.32						
4A (Block 3)	63.96	2.66						
4B (Block 2)	64.60	2.69						
3E	84.79	3.53						
3C	89.72	3.74						
2B	92.29	3.85						
1F	110.82	4.62						
2A	113.33	4.72						
2F	159.89	6.66						
2Н	178.59	7.44						
2G	184.49	7.69						
2D	194.31	8.10						
3A	653.59	27.23						
3B	829.06	34.54						
1B	888.63	37.03						
3F	1544.84	64.37						
3D	2708.36	112.85						
2E	9354.81	389.78						

These results show a wide range in travel times, and slightly more overlap between blocks (i.e., Blocks 1, 2, and 3 as denoted by the first character in the subplot ID, with the exception of the controls, which have block designations after the ID) than was observed

in computations of average hydraulic conductivity across all depths for each subplot. This reflects the fact that some of the hydraulic conductivity values from the deepest layer (as opposed to the average of values across all layers) were lower in Block 1 than Blocks 2 and 3. The trend of notable differences between blocks, however, is still evident.



Figure 64. Leachate travel time from bottom of biosolids row to sampling equipment.

As can be more readily seen from Figure 64 above, in 80% of the subplots, biosolids leachate will reach the collection equipment within 8 days. The other 20%, however, will take a minimum of one month to travel 30 cm. Longer travel times will result in more time for diffusion, interaction with soil particulates and microbes, and other processes occurring in the soil profile, which could result in compositional changes in the leachate. Short travel times, however, may allow for intervals in which some portions of the soil are drained and left unsaturated, thereby altering the physical and chemical conditions in the soil.

Rain Gauge Data

Rain gauge data collection began in December 2002. Data were downloaded approximately every two weeks from the data logger. Routine physical inspection of the rain gauge showed it to be in good working condition over most of the course of data collection activities. Between May 12–30, 2003 and June 20-23, 2003, malfunctions resulted in the loss of data from these time periods. Values for these time periods were estimated using rain gauge data collected from three other sites in Maryland and applying the U.S. National Weather Service's inverse square distance weighting method. This technique uses the formula:

(4)

$$P_{x} = \{(1/d_{ax})^{2} * P_{a} + (1/d_{bx})^{2} * P_{b} + (1/d_{cx})^{2} * P_{c} + \dots\} / \{(1/d_{ax})^{2} + (1/d_{bx})^{2} + (1/d_{cx})^{2} \dots\}$$
where:

 P_x = estimated precipitation at gauge x,

 $P_{a,\,b\,\,o\,r\,\,c}$ = known precipitation at gauge a, b, or c, and

 $d_{ax, bx, or cx}$ = distance between rain gauge x and rain gauge a, b, or c.

On another occasion in June 2003, bird droppings had plugged the hole at the bottom of the rainwater collection funnel and, upon release of the plug, the rain collected from the prior day's storm was consequently recorded in the data logger as occurring over a much smaller time frame on the day after the actual storm event. This did not adversely impact use of the data. Monthly totals are presented in Figure 65 and a side-by-side comparison of 2003 to 2004 rainfall is provided in Figure 66.



Figure 65. Monthly rainfall totals for December 2002-December 2004.



Figure 66. Comparison of rainfall in 2003 and 2004.

From these figures, it is evident that 2003 had more precipitation than 2004, with particularly high rainfall in May and June. In 2003 this high rainfall delayed the planting of trees at the experimental site from early May until mid-June 2003. In both years, May through September were marked by greater precipitation than other months. Dependent upon surface and soil conditions as well as the intensity and duration of precipitation, rainfall can impart an intense, immediate influence on subsurface flow or, conversely, a more diffuse, delayed effect.

In those soils with higher hydraulic conductivity values (> 10^{-4} cm/sec) the travel time for infiltrating rainfall to reach sampling equipment can be rapid. The rate at which rainfall would flow directly through the biosolids row, however, would be much slower given that the measured hydraulic conductivity for the biosolids at time of application was 2.55x10⁻⁶ cm/sec. In such cases rainfall will take the path of least resistance and flow around the edges of the trench to then proceed underneath the row and into the sampling equipment. For those soils with hydraulic conductivity comparable to the biosolids, flow may be more evenly distributed amongst biosolids and surrounding soil.

As biosolids rows drain and loose moisture content, they will have the capacity to absorb more of the infiltrating rainfall. In fact, the resulting gravimetric and matric potential combined with chemical forces from the high organic content facilitates water infiltration into the biosolids rows (Sikora and Colacicco, 1980). Trenching studies conducted in the 1970s showed that gravimetric flow prevailed, and biosolids rows dewater from the top down. In fact, 19 months after biosolids placement in a row surrounded by sandy soil, the top 20% of a row of raw, limed sludge had weathered to a peat like consistency (Walker, 1974). Another trenching study conducted between 1977-

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1980 (Sikora, et al., 1982) in well-drained silt loam soils similarly found the largest amount of biosolids dewatering to occur in the first 20 months, but overall, the amount of dewatering that occurred was less than that observed in sandy soils due to the slower percolation through silty soils. Regardless, the biosolids pack dewatered from the top down.

These varying soil conditions will impact the transport of rainfall and, by association, those compounds soluble in water that will accompany the flow of water through the soil profile. The effects will likely be most immediate and pronounced in the sandier soils with high hydraulic conductivities. In such cases rainfall may flush soil water and accompanying solutes through the soil. Flushing could also result in the mixing of rainfall with the existing soil water to dilute solute concentrations.

To better evaluate the time for rainfall to percolate through the soil profile to the depth of the sampling equipment, hydraulic conductivity values were used to estimate travel time using the same technique as that previously presented in the hydraulic conductivity results section. In this instance, however, the entire soil profile was considered, as opposed to just the 30 cm layer between the biosolids row bottom and the equipment. The deep core sample provided an estimate of hydraulic conductivity for the 30 cm directly above the pan lysimeter; the middle core sample provided an estimate for the 30 cm above the deep layer, and the shallow core provided an estimate for the remaining upper profile (i.e., to the surface). The upper profile layer varied in length dependent upon the biosolids application rate and changes in elevation.

For a given subplot, the hydraulic conductivity value for each layer was divided by the respective depth of that layer to determine the travel time through the layer. These

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three time lengths were then summed to determine the total time for rainfall to travel

from the surface to the sampling equipment (Schwab, et al., 1993). Results are shown in

Table 14 and Figure 67.

Sorted by Subplot ID							
	Rainfall travel time from surface						
Subplot	to sampling						
ID	equipment (hour)	Time (days)					
1A	12.08	0.50					
1B	1010.27	42.09					
1C	37.86	1.58					
1D	1.88	0.08					
1E	92.85	3.87					
1F	216.72	9.03					
1G	113.26	4.72					
1H	157.05	6.54					
1I	270.16	11.26					
2A	189.39	7.89					
2B	145.94	6.08					
2C	908.24	37.84					
2D	827.37	34.47					
2E	9925.39	413.56					
2F	350.76	14.62					
2G	691.63	28.82					
2H	1272.95	53.04					
2I	138.52	5.77					
3A	92499.96	3854.17					
3B	3316.25	138.18					
3C	445.80	18.58					
3D	26594.83	1108.12					
3E	20356.17	848.17					
3F	4037.95	168.25					
3G	8049.82	335.41					
3H	681.66	28.40					
3I	393.53	16.40					
4A	971.75	40.49					
4B	149.98	6.25					
4C	15188.68	632.86					

 Table 14. Rainfall travel time through soil profile to sampling equipment depth.

 Sected by Subplet ID

Sorted by Travel Time								
Subplot	Rainfall travel time from surface to sampling		Time					
ID	equipment (hour)	Time (days)	(months)					
1D	1.88	0.08						
1A	12.08	0.50						
1C	37.86	1.58						
1E	92.85	3.87						
1G	113.26	4.72						
2I	138.52	5.77						
2B	145.94	6.08						
4B	149.98	6.25						
1H	157.05	6.54						
2A	189.39	7.89						
1F	216.72	9.03						
1I	270.16	11.26						
2F	350.76	14.62	0.49					
3I	393.53	16.40	0.55					
3C	445.80	18.58	0.62					
3H	681.66	28.40	0.95					
2G	691.63	28.82	0.96					
2D	827.37	34.47	1.15					
2C	908.24	37.84	1.26					
4A	971.75	40.49	1.35					
1B	1010.27	42.09	1.40					
2H	1272.95	53.04	1.77					
3B	3316.25	138.18	4.61					
3F	4037.95	168.25	5.61					
3G	8049.82	335.41	11.18					
2E	9925.39	413.56	13.79					
4C	15188.68	632.86	21.10					
3E	20356.17	848.17	28.27					
3D	26594.83	1108.12	36.94					
3A	92499.96	3854.17	128.47					



Figure 67. Number of days for rainfall to reach sampling equipment.

Results encompass a broad range of time, reflecting the differing soil properties over the experimental site. It is more likely that those subplots with travel times less than two weeks will be susceptible to a flushing effect from the rainfall. Those soil properties that inhibit rainfall flow and produce longer travel times will dissipate the flushing potential of the rainfall. Subplot order in Figure 67 is similar to the biosolids leachate travel time previously presented in Figure 64, but in some instances subplot order has changed. Such changes reflect the more marked effect of certain hydraulic conductivity levels from the middle and shallow depth levels. In addition, some of the shallow depth level hydraulic conductivity values had greater influence on the travel time due to the greater length associated with that layer (i.e., greater than the 30 cm lengths associated with the middle and deep layers). These travel estimates will be used in subsequent sections to evaluate whether or not analytical results indicate that a flushing effect may have occurred.

Soil Water Analysis and Results: Overview

Pan lysimeter soil water sample collection began in April 2003, upon completion of biosolids application to the experimental plot. Suction lysimeters were installed in July and August 2003 and sample collection began in November 2003. Results presented encompass sample collection activities through December 2004. Table 15 documents the dates of collection and number of samples collected.

	Pan Lysime	ter Samples	5	S	Suction Lysimeter Samples					
20	03	20	04	20)03	2004				
Sampling	Number of samples collected	Sampling	Number of samples collected	Sampling	Number of samples collected	Sampling	Number of samples collected			
1/22/2002	(11)	1/7/2004	(11)	Date	(11)	1/6/2004	(11)			
5/7/2003	10	1/12/2004	10			1/0/2004	43			
5/28/2003	0	1/13/2004	0			1/12/2004	31			
6/4/2003	9	2/10/2004				2/11/2004	47			
6/17/2003	9	2/17/2004	9			2/18/2004	46			
6/23/2003	9	2/25/2004	7			2/26/2004	45			
7/8/2003	11	3/8/2004	9			3/10/2004	46			
7/16/2003	9	3/19/2004	8			3/19/2004	47			
7/23/2003	10	3/26/2004	7			3/26/2004	45			
8/6/2003	8	4/9/2004	10			4/9/2004	45			
8/13/2003	11	4/23/2004	9			4/23/2004	45			
8/20/2003	11	4/30/2004	6			4/30/2004	43			
9/3/2003	8	5/13/2004	9			5/14/2004	48			
9/10/2003	10	5/21/2004	9			5/21/2004	44			
9/22/2003	11	5/27/2004	10			5/27/2004	45			
10/14/2003	10	6/16/2004	10			6/16/2004	50			
10/20/2003	11	6/23/2004	11			6/23/2004	47			
10/29/2003	10	6/29/2004	12			6/30/2004	47			
11/9/2003	9	8/17/2004	11	11/10/2003	43	8/18/2004	50			
11/16/2003	8	8/23/2004	11	11/18/2003	44	8/25/2004	49			
11/24/2003	11	8/30/2004	11	11/23/2003	44	8/31/2004	44			
12/3/2003	10	10/15/2004	10	11/30/2003	46	10/16/2004	49			
12/10/2003	9	10/22/2004	11	12/8/2003	44	10/23/2004	47			
12/18/2003	9	10/29/2004	11	12/21/2003	47	10/30/2004	44			
		12/3/2004	11			12/4/2004	49			
		12/12/2004	5			12/13/2004	47			
		12/21/2004	11			12/22/2004	44			

Table 15. Summary of pan and suction lysimeter sampling activities.

Provided every pan produced a sample, each sampling date should have produced 11-12 samples (10 pans plus one or two equipment blanks). Similarly, for suction lysimeters, each collection date should have produced 51 samples (50 suction lysimeters plus one equipment blank). As shown in Table 15 above, however, not all sampling events produced these numbers. Recall that pan lysimeters capture saturated flow. Consequently, once gravimetric flow ceases, or other potentials and preferential flow paths in the soil override the gravimetric potential, flow to the pan lysimeters will be reduced. For suction lysimeters, which capture both saturated and unsaturated flow, the matric forces in the subsurface around the equipment will determine whether or not the suction placed on the lysimeter will be strong enough to pull soil water into the equipment. Therefore, if the soil is extremely dry, it will be difficult to capture any sample. In addition, equipment malfunctions (e.g., plugs in the sampling lines from particulates or frozen sample) also prevented collection on several occasions.

Samples varied in appearance (e.g., color, clarity, types of particulates) and properties, with pH ranging from 5.03-8.20 for pan lysimeters and 4.82 – 11.33 for suction lysimeters. Figure 68 below shows the average pH in relation to each equipment type and position for subplots with biosolids and without biosolids (i.e., controls). For subplots with biosolids, notable trends shown in this graphic include: 1) for vertical flow suction lysimeters, pH decreased with increasing distance from the biosolids row; 2) vertical flow suction lysimeters produced values greater than lateral flow suction lysimeters; 3) the pan lysimeters (all of which were positioned at the same 30 cm depth below the biosolids row) produced samples with lower pH values than any of the vertically placed suction lysimeter samples. In addition, with the exception of the lateral

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flow suction lysimeter placed 30cm from the biosolids row (i.e., SL-PL-30cm), all control values were less than those subplots with biosolids.

These trends reflect a number of influences to which the soil water is subject, including travel time and soil interaction. Equipment closest to the biosolids row will likely contain the most unaltered leachate from the biosolids because the shorter flow path means it has contact with less soil as well as less time in contact with this soil. Given the high pH level of the biosolids, those samples closest to the flow path from the biosolids will likely have higher pH values. For this reason as well, it is not surprising that the vertical flow suction lysimeters would have higher pH values than the lateral flow samples, because the lateral flow samples are not directly underneath the biosolids rows, but rather to the side of the rows. This suggests that lateral flow is not a major factor in transport from the biosolids pack.

This trend in vertical flow vs. lateral flow of suction lysimeters, as well as the difference between the suction lysimeters and the pan lysimeters, also reflects the manner in which equipment was installed. Pan lysimeter installation was performed underneath the biosolids row, and did not directly disturb the biosolids. Lateral flow suction lysimeters required drilling to either side of the biosolids row, and also did not disturb the biosolids row. For the those suction lysimeters designed to capture vertical flow, however, installation required drilling directly through and underneath the biosolids row to appropriately position the suction lysimeter. Although bentonite plugs around the suction lysimeter prevent a preferential flow path from forming along the wall of the lysimeter, the initial drilling process was often accompanied by some leachate flow into the lysimeter hole. This, and the disturbance of the biosolids itself, likely contributed to

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the higher pH values in the suction lysimeter samples. Finally, the lower pH values associated with the controls are consistent with the fact that the controls do not contain a recent application of high-pH biosolids.



Figure 68. Average pH values for suction lysimeter (SL) and pan lysimeter samples.

The 2-bar ceramic cup of the suction lysimeter had a pore size of 1*um*. As a result, suction lysimeter samples were subjected to an initial filtration during the collection process. Pan samples, however, were not initially filtered and remained that way in the pan between collection dates. As would be expected, pan samples contained more particulates than the suction lysimeters. In general, pan samples tended to become clearer after several months of collection (though most still contained particulates). Both

pan and suction lysimeter samples from many of the subplots contained small rustcolored flakes that are speculated to be an iron precipitate. Within 30 minutes of collection, the precipitate became more prevalent, and precipitate settled to the bottom of the collection container. Figures 69-72 and 74-77 show samples from multiple subplots, demonstrating the varied appearance. Figures 73 and 78 show the residue captured by the filter paper for a set of pan and suction lysimeter samples.



Figure 69. Pan lysimeter samples from block 1.



Figure 70. Pan lysimeter samples from block 2.



Figure 71. Pan lysimeter samples from block 3.



Figure 72. Pan lysimeter samples from control and equipment blank.


Figure 73. Residue from filtration of pan lysimeter samples.



Figure 74. Suction lysimeter samples from a block 1 subplot.

Figure 75. Suction lysimeter samples from a block 2 subplot.



Figure 76. Suction lysimeter samples from a block 3 subplot.

Figure 77. Suction lysimeter samples from a control subplot.



Figure 78. Residue from filtration of SL samples.

Soil Water Results: Total Nitrogen and Ammonium (NH₄⁺-N) Data

Biosolids applied to the experimental plot contained on average 41,200 mg/kg total nitrogen and 2,700 mg/kg of ammonium nitrogen. The cation ammonium represents the inorganic portion of total nitrogen. By simple calculation, inorganic nitrogen constitutes 7% of the total nitrogen. Consequently, a majority of the nitrogen applied (93%) was in the form of organic nitrogen. Unless this organic nitrogen exists in dissolved form, movement into the soil profile will be limited.

Ammonification is the first step in the decomposition of organic nitrogen, and is performed by a variety of heterotrophic organisms in both aerobic and anaerobic environments. The product, ammonium, is soluble in water and easily infiltrates the soil profile, though movement is often limited by the cation's attraction to negatively charged particles in the soil (Haynes, 1986). As stated above, ammonium was already present in the applied biosolids in notable quantities. Therefore, notwithstanding ammonium production from organic nitrogen that could have occurred over time, an ample supply existed at the start of the experiment.

Soil water samples were analyzed for both total nitrogen and ammonium nitrogen. For the pan lysimeters, 427 samples were analyzed. When these individual subplot values were averaged within each quarter, the 427 results were consolidated to 222 average values. For the suction lysimeters, 1450 samples were analyzed. When these individual lysimeter results were averaged within each quarter, the 1450 results were consolidated to 562 average values. Unless otherwise stated, the data presented and discussed below represent the quarterly averages of the individual monthly values.

Results show that a majority of the total nitrogen measured in the samples was in the form of ammonium. In theory, ammonium values should be less than or equal to total nitrogen values. Field and analytical variability, however, is a standard component of any experiment and must be considered in the interpretation of results. Therefore, ammonium values less than or equal to 120% of total nitrogen values were considered within the range of acceptable analytic variability. All ammonium values that were greater than total nitrogen values for the same sample were reanalyzed multiple times at the laboratory to provide the most representative results possible. The high total nitrogen and ammonium concentrations in some of these samples, as well as other components of the sample matrix, were likely responsible for the analytical variability. Despite these analytical challenges, only an extremely small portion of the samples (4 from the pan lysimeters) exhibited ammonium values greater than 120% of total nitrogen values.

The information in Table 16 indicates that 88% of the pan results and 78% of the suction lysimeter results contain ammonium concentrations equivalent to total nitrogen (i.e., those NH_4^+ values within 80-120% of total N). Pan lysimeters generated more results within this range and a smaller percentage of samples below this range compared to the suction lysimeters. Due to the similarity in values and patterns between total N and ammonium, it would be redundant to present both sets of results. Consequently, ammonium results are presented in this section, and total nitrogen results are provided in Appendix 2 for reference. Ammonium results are the primary focus of this discussion, with some supplemental discussion of total nitrogen where appropriate. Pan lysimeter results will be presented first, followed by suction lysimeter results.

Pan Lysimeter Samples	Suction Lysimeter Samples	
$22 \text{ of } 222 \text{ NH}_4^+ \text{ values} = 10\% \text{ were}$	$122 \text{ of } 562 \text{ NH}_4^+ \text{ values} = 22\% \text{ were}$	
< 80% of the total N values.	< 80% of the total N values.	
196 of 222 NH_4^+ values = 88% were	440 of 562 NH_4^+ values = 78% were	
> 80% and $< 120%$ of total N values	> 80% and $< 120%$ of total N values	
4 of 222 NH_4^+ values = 2% were	0 of 562 NH_4^+ values = 0% were	
> 120% of total N values.	> 120% of total N values.	

Table 16. Overall comparison of total nitrogen and ammonium values.

Pan Lysimeter Samples

Total N and ammonium results showed appreciable concentrations across application rates, with controls exhibiting the lowest values. A summary of the concentration ranges is presented in Table 17.

Table 17. Frequency of pan lysimeter results in successive concentration ranges.

	Total N	NH4 ⁺
Values < 10 mg/L	34 of 222 = 15%	33 of 222 = 15%
Values from 10-20 mg/L	15 of 222 = 7%	18 of 222 = 8%
Values from 20-50 mg/L	25 of 222 = 11%	25 of 222 = 11%
Values from 50-100 mg/L	33 of 222 = 15%	30 of 222 = 13%
Values from 100-500 mg/L	81 of 222 = 36%	80 of 222 = 36%
Values from 500-1000 mg/L	15 of 222 = 7%	17 of 222 = 8%
Values $> 1000 \text{ mg/L}$	19 of 222 = 9%	19 of 222 = 9%

Though 34% of results were less than 50 mg/L, within the general range of the control results, an equally high percentage of results had more significant values between 100-500 mg/L. Distribution of values was essentially the same between total nitrogen and ammonium. Ammonium results are presented by each application rate in Figures 79-81 below. Total nitrogen results are included in Appendix 2. Due to the wide range in concentrations, results are presented in a logarithmic scale.



Figure 79. Ammonium quarterly average concentrations for low-level application rate in pan lysimeters.



Figure 80. Ammonium quarterly average concentrations for mid-level application rate in pan lysimeters.



Figure 81. Ammonium quarterly average concentrations for high-level application rate in pan lysimeters.

Results from the controls (0 kgN/ha; 0 trees/ha) ranged between 3.2 – 43 mg/L for total N and 1.1 – 44 mg/L for ammonium; approximately 60% of these values were greater than 10 mg/L. Prior testing of untreated soil at the tree farm showed total nitrogen values of 100 mg/kg and ammonium values of 1.2 mg/kg (Pepperman, 1995). These values, which represent the amount in one kilogram of soil, can be used to estimate the amount in the aqueous portion of the soil based on several soil property assumptions.

Assuming an average bulk density for subsoil samples ranging between 1.6-1.9 g/cm³, each kg (1000g) of soil would provide: $1000g/(1.6g/cm^3) = 625 \text{ cm}^3$ to $1000g/(1.9g/cm^3) = 526 \text{ cm}^3$ of soil volume. Given this range in soil volume of 526-625 cm³, and assuming a volumetric water content between 25-50%, we can estimate the

volume of water present in this given volume of soil. For these volumetric water contents: $(0.25 \text{ cm}^3 \text{ water}/1 \text{ cm}^3 \text{ soil}) * 526 \text{ cm}^3 \text{ soil} = 131 \text{ cm}^3 \text{ water} = 131 \text{ mL}$ water. Similarly, 625 cm³ of soil would generate 156 mL water; 0.50 cm³ water/1 cm³ soil and 526 cm³ soil would generate 263 mL water; and 0.50 cm³ water/1 cm³ soil and 625 cm³ soil would generate 312 mL water.

Not all of the total nitrogen will be soluble in water. Based on information regarding the soluble nitrogen content in plant matter (Haynes, 1986), it is reasonable to assume that no more than 10% will be in solution. The 100 mg/kg of total nitrogen measured in the soil sample therefore provides a maximum of 10 mg of nitrate in 131-312 mL of soil water, or a range of 32-76 mg/L. This range in value of 32 - 76 mg/L for background levels of total nitrogen is comparable to the values seen in the samples collected during this experiment.

Assuming most of the measured ammonium in the soil sample will be in solution, the 1.2 mg/kg of ammonium measured in the soil sample is equal to 1.2 mg of ammonium in 131-312 mL of soil water. This range of 4-9 mg/L represents the lower levels of ammonium found in the control samples. The higher values found in the controls could be a result of residual ammonium from prior biosolids applications or decomposition of vegetation with subsequent percolation through the subsurface.

Using the 1-45 mg/L control results as background levels, sample values greater than 100 mg/L will be more closely examined. Figure 82 provides a breakdown of the number of samples from each application rate with values greater than 100 mg/L.



Figure 82. Number of ammonium samples > 100 mg/L by application rate and tree density.

Comparing application rates, 19,650 kg N/ha and 39,300 kg N/ha produced the same number of results with values greater than 100 mg/L (i.e., 47 results), whereas 58,900 kg N/ha produced a markedly lower number of these higher values (22 results). A breakdown of these total numbers by the three tree densities shows that 0 trees/ha contained the most numerous greater than 100 mg/L results for all three application rates. The differences between 716 and 1074 trees/ha were less consistent across application rates. Not shown in this figure but also worth noting is the fact that although the 0 trees/ha density exhibited the greater number of values above 100 mg/L, this tree density was not always associated with the highest ammonium concentrations. From Figures 79-81, it is evident that for19,650 kg N/ha application rate, the 716 trees/ha density had

much higher values than either the 0 trees/ha or 1074 trees/ha densities. For both 39,300 kg N/ha and 58,900 kg N/ha, both 0 trees/ha and 1074 trees/ha shared the highest values, all of which shows that the highest concentrations were spread across all tree densities.

An overview of variability within each quarter is presented in Figures 83-85, in which the results for the three blocks within a particular treatment were averaged and standard deviation determined. Visual inspection shows no definitive trend in standard deviation over time.



Figure 83. Ammonium average concentrations across blocks with standard deviation for low-level application rate in pan lysimeters.



Figure 84. Ammonium average concentrations across blocks with standard deviation for mid-level application rate in pan lysimeters.



Figure 85. Ammonium average concentrations across blocks with standard deviation for high-level application rate in pan lysimeters.

Statistical Analysis

Statistical analysis included evaluation of the following interactions for significance:

- Application rate
- Tree density
- Application rate by tree density
- Quarter
- Application rate by quarter
- Tree density by quarter
- Application rate by tree density by quarter

When evaluating results, the more complex statistically significant interactions were first considered because they impart more detail on what experimental condition is most influencing the differences. In addition, the more complex interaction will capture any of the simpler interactions represented by the included conditions.

Statistical analyses showed no significant differences ($\alpha = 0.05$) between any application rates, tree densities, or time. This includes the comparison of controls to the other treatments. Though the statistics indicate that the higher values from all three biosolids application rates, which ranged from 100 mg/L – 3178 mg/L, were not significantly different from the controls, which exhibited values less than 45 mg/L, it is evident from the results that ammonium is leaching from the biosolids to the pan lysimeters. The lack of statistical significance may be related in part to the fact that the high results were not consistently reproduced amongst replicates (i.e., blocks), and does not negate the fact that these higher concentrations of total nitrogen (mostly in the form of ammonium) are present in those samples.

To determine if these high concentration levels are reasonable, the concentrations in the biosolids must be revisited. Recall from the prior biosolids results section that the average concentration of ammonium in the biosolids applied to the experimental plot was 700 mg/kg (0.07%) on a wet weight basis. Given an average percent moisture content of 72%, and assuming that water-soluble ammonium would be in the aqueous phase of the biosolids, the concentration of ammonium in solution is calculated as follows: 700 mg/kg of biosolids*1kg of biosolids/0.72 kg water = 972 mg/kg of water. Assuming a water density of 1g/mL (or 1 kg/L), the estimated concentration of ammonium in the aqueous phase is 972 mg/L. This concentration is an estimate, and does not account for the fact that ammonium, as a cation, is adsorbed to organic compounds and soil particles with negative charges. This adsorption will impact compartmentalization of ammonium in the aqueous versus the solid phase, as well as movement with the water (Sopper and Kerr, 1979; Haynes, 1986).

Regardless, this calculation does support the higher values found in the samples. Values above 1000 mg/L can be explained by either the microbial breakdown of total nitrogen into ammonium and/or the concentration of ammonium in the soil during dryer time periods when water content decreases. Stednick and Wooldridge's (1979) lysimeter studies evaluating use of liquid digested sludge in a tree stand supports the latter condition, noting that high nutrient concentrations in the soil solutions tended to be associated with low water flow and soil moisture content.

Experiments conducted by Brutsaert, et al. (2004) on nitrate leaching from biosolids stockpiles showed that leachate samples collected over an eight month time frame in pan lysimeters installed in the soil profile one and two feet under the stockpile contained 800-

1500 mg/L total Kjeldahl nitrogen (most of which was in the form of ammonium). Further down the profile, three feet below the stockpile, a marked decrease in total Kjeldahl nitrogen was noted, with values typically below 100 mg/L. Leachate collected directly from the stockpile contained 2,800 – 4000 mg/L ammonium, demonstrating that some attenuation or conversion of ammonium had occurred.

Based on the fact that ammonium is held in the soil by the reversible process of cation exchange, in which ammonium is adsorbed to negatively-charged soil sites, as well as the non-exchangeable process of fixation within clay lattices (Haynes, 1986), it may have been expected that ammonium would be more selectively absorbed by those subplots with higher silt and clay concentrations. Haynes (1986) and others have noted that, barring other factors, leaching losses of ammonium are usually only problematic in soils with a low cation exchange capacity (CEC), as is often evidenced in sandy soils. Block 3 contained the highest amount of clay in the soils, followed by block 2. Block 1 contained the sandiest of the subplots. Based on this logic, block 1 should allow the highest amount of ammonium to flow through the soil profile to the pans, followed by block 2, with block 3 hindering flow the most. It is evident from the results that no single block stands out as having predominantly higher results across the treatments.

This mix of results can be explained by a number of factors. Note from earlier descriptions of the soil that within each of the three blocks, the subsoil profile was not well structured, having been disturbed during mining activities and later during previous biosolids applications. Most subplots in block 3 contained pockets of sandier soil, just as subplots in blocks 1 and 2 contained pockets of high clay-content soil. This variation in the soil will impact cation exchange capacity and hydraulic conductivities, both of which

will affect the adsorption of ammonium to the soil and rate of soil water flow from the biosolids to the pan lysimeters.

Another important factor impacting the adsorption of ammonium to the soil is the presence of other cations. The biosolids were heavily limed and contained on average 119,000 mg/kg calcium (11.9%) on a dry weight basis and 34,200 mg/kg (3.42%) on a wet weight basis. Potassium in the biosolids, measured as potash (K_2O), was also present in appreciable quantities (30,000 mg/kg on a dry weight basis and 1,200 mg/kg on a wet weight basis). Magnesium concentrations were 3,100 mg/kg on a dry weight basis and 900 mg/kg on a wet weight basis. These cations will compete with ammonia for exchange sites both in the biosolids and in the soil profile. According to Barber (1995) ammonium is similar in size to potassium and will therefore be held on soil exchange sites with similar strength. Haynes (1986) reported the following order of replacing power on cation exchange sites in soils: $Al^{3+} > Fe^{3+} > Ca^{2+} > Mg^{2+} > K^+ > Na^+$. Though this lineup does not include ammonium (NH_4^+) , it does include potassium (K^+) . As noted above, potassium has a similar holding strength to ammonium. Consequently, ammonium will be out competed for exchange sites by elements known to be present in large quantities in the biosolids used at the experimental plot.

Loehr et al (1979) estimated that, within the pH range of most soils (4.5-7.5), the presence of calcium, magnesium, and potassium could reduce the amount of CEC available for ammonium to approximately 5%. For a soil with an average CEC value (approximately 15 meq/100g), Loehr calculated that the soil's capacity for ammonium would be limited to 112 kg/ha. The ammonium content of the biosolids used at the ERCO site was approximately 7% of the total nitrogen. The low-level total nitrogen

application rate was 19,650 kg N/ha. Seven percent of this application rate is 19,650*0.07 = 1,375 kg NH₄⁺/ha, a 10-fold increase above the cutoff presented by Loehr. Leaching of ammonium is therefore not an unexpected occurrence at the ERCO experimental plot. Suction lysimeter results, which evaluated three different distances below the biosolids row and two positions lateral to the biosolids row, may provide more insight regarding the direction and depth to which ammonium leaching occurred.

Suction Lysimeter Samples

As with the pan lysimeters, total N and ammonium results from suction lysimeters showed appreciable concentrations across application rates, with controls exhibiting the lowest values. A summary of the frequency of results in successive concentration ranges is presented in Table 18.

1 2		
	Total N	NH ₄ ⁺ *
Values < 10 mg/L	92 of 562 = 16%	108 of 562 = 19% (pan=15%)
Values from 10-20 mg/L	41 of 562 = 7%	29 of 562 = 5% (pan=8%)
Values from 20-50 mg/L	56 of 562 = 11%	58 of 562 = 11% (pan=11%)
Values from 50-100 mg/L	77 of 562 = 14%	69 of 562 = 12% (pan=13%)
Values from 100-500 mg/L	151 of 562 = 27%	150 of 562 = 27% (pan=36%)
Values from 500-1000 mg/L	41 of 562 = 7%	45 of 562 = 8% (pan=8%)
Values > 1000 mg/L	104 of 562 = 18%	103 of 562 = 18% (pan=9%)

Table 18. Frequency of suction lysimeter results in successive concentration ranges.

*For comparison, pan lysimeter percentages are included in parentheses (pan=xx%).

Though a high percentage of results were less than 50 mg/L, a higher percentage of results had more significant values above 100 mg/L. Distribution of values was essentially the same between total nitrogen and ammonium. Compared to pan lysimeters, distribution was the same for values less than 100 mg/L. A higher percentage of suction lysimeter values, however, were distributed in the highest range (i.e., values > 1000 mg/L), with corresponding lower amounts in the 100-500 mg/L range.

Suction lysimeter results for ammonium are presented by each application rate in Figures 86-94 below. Total nitrogen is presented in Appendix 2. Data plots show quarterly values for each block and depth. The initial data plot with all four quarters represented is included to provide an overall view of results over time. Because of the number of data points, however, the resolution of individual application rate by tree density combinations was compromised. Consequently, this original data plot was further separated into two charts, each of which includes two of the four quarters. Within these charts, quarter, block and position designations are noted. Due to the wide range in concentrations, results are plotted on a logarithmic scale.

Recall that five suction lysimeters were installed within each of the 30 subplots. Three capture vertical flow and were positioned 15, 30, and 60 cm underneath the bottom of the biosolids rows. Two capture vertical flow and were positioned 15 and 30cm lateral from the edge of a biosolids row at a depth equal to the bottom of the biosolids row (Figure 6). In the bar charts and tables that follow, these positions are indicated as PV15, PV30, PV60, PL15, and PL30, respectively. As a reminder, the time periods associated with each of the suction lysimeter quarterly designations include:

Q4 = November 2003 – February 2004; Q5 = March 2004 – May 2004; Q6 = June 2004 – August 2004 (recall that samples were not collected in July); Q7 = October 2004 – December 2004 (recall that samples were not collected in November).









Q7-B3-PL30

Q7-B3-PL15

Q7-B3-PV60

Q7-B3-PV30

Q7-B3-PV15

Q7-B2-PL30

Q7-B2-PL15

Q7-B2-PV60

Q7-B2-PV30

Q7-B2-PV15

Q7-B1-PL30

Q7-B1-PL15

Q7-B1-PV60 Q7-B1-PV30 Q7-B1-PV15 Q6-B3-PL30

Q6-B3-PL15 Q6-B3-PV60 Q6-B3-PV30

Q6-B3-PV15

Q6-B2-PL30

Q6-B2-PL15

Q6-B2-PV60

Q6-B2-PV30

Q6-B2-PV15

Q6-B1-PL30

Q6-B1-PL15

Q6-B1-PV60

Q6-B1-PV30

Q6-B1-PV15

0

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0







average concentrations for mid-level app. rate in SL Ammonium quarterly samples: Q4&5. Figure 90.



SL average concentrations for mid-level app. rate in Ammonium quarterly samples: Q6&7. Figure 91.







SL Ammonium quarterly average concentrations for high-level app. rate in Figure 93.



samples: Q6&7. Figure 94.

Results from the controls (0 kgN/ha; 0 trees/ha) ranged between 0.91 - 51 mg/L for total N and 0.07 - 55 mg/L for ammonium, similar to the ranges associated with the pan lysimeters. Of the 28 control values, only four for total nitrogen and three for ammonium (11-14%) were greater than 10 mg/L. The smaller proportion of higher values is different from the pan lysimeters, for which 60% of the control results were above 10 mg/L. As noted in the prior discussion on pan lysimeter results, these control values are consistent with background level estimates previously determined on untreated soil samples from the farm.

As with the pan lysimeters, the trends for total nitrogen and ammonium coincide. Total nitrogen and ammonium results demonstrate essentially the same partitioning into concentration ranges as well as distribution over suction lysimeter positions and quarters. Using the 0.07 - 55 mg/L control results as background levels, sample values greater than 100 mg/L will be more closely examined.

Figure 95 compares the percentages of ammonium results in different concentration ranges across application rates. Figure 96 further examines ammonium results greater than 100 mg/L within each application rate by tree density. Of the three application rates, 39,300 kg N/ha produced the highest percentage of results greater than 100 mg/L. Although 19,650 kg N/ha rate had the highest percentage of results greater than 1,000 mg/L, it was not notably higher than the other two application rates. Results greater than 100 mg/L were distributed evenly across tree densities for all application rates. Differences in concentrations over time were not obvious from this general overview, but will be presented in more detail when discussing statistical analysis results.



Figure 95. Suction lysimeter ammonium results: distribution across concentration ranges for each application rate.



Figure 96. Suction lysimeter ammonium results: distribution of results >100 mg/L across tree densities for each application rate.

Differences across each application rate are more clearly linked to suction lysimeter position. From the quarterly results presented in Figures 86-94 above, and as more clearly shown in summary level in Figure 97 below, a greater concentration of total nitrogen and ammonium had traveled with greater frequency directly underneath the biosolids rows to the shallowest of the three vertical suction lysimeters, with successively lower concentrations by depth. The highest results (those greater than 1,000 mg/L) are associated with the vertical flow lysimeters in closest proximity to the biosolids row (i.e., PV15 and PV30) across all quarters, with PV15 standing out amongst all other positions. Within the PV15, PV30, and PV60 positions, each application rate produced similar numbers of high results, with 19,650 kg N/ha producing slightly more values in the PV15 position and 58,900 kg N/ha producing more in the PV30 and PV60 positions.

Compared to the vertical flow positions, the lateral flow positions had a lower frequency of soil water with concentrations above 100 mg/L, especially the lysimeter positioned furthest from the edge of the biosolids row (PL30), for which the highest application rate (58,900 kg N/ha) produced no results greater than 100 mg/L. This supports the postulation that flow of leachate from the biosolids row is predominantly downward.



Figure 97. Suction lysimeter ammonium results: distribution of results > 100 mg/L across lysimeter positions.

Table 19 provides supporting information to Figure 97, and provides ranges in concentrations for each position. The high end of the concentration range for the suction lysimeters (6723 and 5103 mg/L for TN and NH_4^+ , respectively) was considerably higher than that of the pan lysimeters (2800 and 3178 mg/L for TN and NH_4^+ , respectively). The position of the pan lysimeter, however, is 30 cm below the biosolids trench. Therefore, it is most appropriate to compare the pan lysimeter results to those from the PV30 suction lysimeters. The high values of 3875 and 4020 mg/L for the suction lysimeter.

	TN&NH ₄ ⁺ Concentration	Number of Results $> 100 \text{ mg/L}$			
SL	Range for those values >	19,650	39,300	58,900	
Position	100 mg/L	kgN/ha	kgN/ha	kgN/ha	Totals
	TN: 124–6273				
PV15	NH ₄ ⁺ : 113-5103	34	31	31	96
	TN: 119–3875				
PV30	NH4 ⁺ : 120-4020	22	19	24	65
	TN: 100–2082				
PV60	NH4 ⁺ : 101-2113	18	21	22	61
	TN: 102-1247				
PL15	NH ₄ ⁺ : 104-1221	17	19	16	52
	TN: 103–357				
PL30	NH4 ⁺ : 100-350	7	17	0	24

Table 19. Comparison of suction lysimeter positions for results > 100 mg/L.

It could be reasoned that the slightly higher values for the suction lysimeter are logical, given the type of water collected from the suction lysimeter versus that from the pan lysimeter. The pan lysimeter predominantly collects saturated flow due to gravimetric forces. This water travels quickly through the larger pores in the soil with less time for soil contact. The suction lysimeter, however, predominantly collects soil water held in the soil by matric forces. This water is subject to a longer residence time that could allow for concentration or conversion of nutrients in the soil (Barbee and Brown, 1986; Marques, et. al., 1996). Conversely, one could argue that saturated flow is often associated with a flushing effect that moves nutrients and other water-soluble compounds through the soil profile, and can account for elevated concentrations of nutrients (Brady and Weil, 2002). Such flushing effects are more likely to be captured by pan lysimeters, which continuously collect sample over time and capture flow from a greater cross sectional area, as opposed to suction lysimeters, which capture soil water from a smaller area and only during the time that suction is applied (for the purposes of this experiment, approximately three days).

When evaluating these conditions, the source of the soil water flowing to the equipment must also be considered. The two main sources of aqueous solution to the soil are rainfall and leachate from the biosolids rows. The elevated ammonium concentrations are clearly from the biosolids (given the much lower values in the controls and the known amounts from the biosolids analysis). Provided the biosolids rows are continuously dewatering, with a majority of flow in the vertical direction, the pan and suction lysimeters will both be capturing soil water introduced from this source. Dilution effects from rainfall and subsequent percolation through the soil as well as concentration effects from drier periods will impact the concentration levels captured by both types of equipment. Consequently, a variety of factors influence the type of soil water that is collected from the two types of lysimeters.

Prior studies comparing pan and suction lysimeters (presented in the literature review section) as well as observations noted in this thesis study show that, with the exception of the known fact that zero-tension lysimeters capture saturated flow and suction lyismeters capture both saturated and more predominantly unsaturated flow, no one physical, chemical, or biological process can be selectively linked to either collection apparatus. Rather, the soil water collected by either of these lysimeters is a product of the specific circumstances governing the experimental set up and environmental conditions. Using both types of sampling equipment does, however, ensure that a comprehensive representation of the soil solution in the soil profile was obtained.

Rainfall Flushing Effect

Another facet of environmental conditions worth considering in the context of these results is whether or not storm events influenced the results. Of particular interest is

whether or not precipitation may have introduced a flushing effect that would have produced elevated results in samples collected during the flushing time frame. Due to the nature of the sample collection process, this evaluation can only consider results from suction lysimeters, which capture flow from the select period of time (usually 3 days) during which suction is placed on the equipment. Pan lysimeters, on the other hand, continuously collect soil water between each monthly collection event. Consequently, and despite the fact that other studies have reasoned that pan lysimeters are more efficient at collecting saturated flow, it is not possible to distinguish between what may be a combination of storm flow and non-storm flow in the pan lysimeter samples.

For this evaluation, individual unconsolidated ammonium results (i.e., those results produced from each sampling event, as opposed to the quarterly averages used for most other evaluations in this discussion) that were greater than 1000 mg/L were identified. Over 247 results encompassing 25 of the 30 subplots were included. Those subplots without results greater than 1000 mg/L included the controls (subplots 4A, 4B, and 4C) and two subplots from block 3 (3E and 3H). Based on the hydraulic conductivity values collected over the soil profile in each subplot, an estimate of rainfall travel time to the equipment was determined (as noted and shown in the previous section on rain gauge data, Table 14 and Figure 67). It is important to note, however that these calculations are based on assumptions of saturated flow. If at any point unsaturated flow conditions exist, which is likely, the percolation of precipitation would be much slower.

Storm events producing greater than 2.54 cm (1 inch) of rainfall were then identified. Based on the dates of the storm events and the previously determined soil profile travel times, the time frame during which the infiltrated rainfall would reach the suction

lysimeters in each subplot was calculated. This rainfall arrival time frame was then compared to the collection dates for samples with the high ammonium concentrations to see if the two time intervals coincided. Of the 247 results identified, 51 (approximately 20%) had sample collection dates that matched the intervals during which a precipitation event would have reached the equipment. With only 20% of the results falling in this category, it is evident that an overall flush effect was not observed. The high ammonium values are instead more closely tied to the ongoing dewatering of the biosolids.

Statistical Analysis

Unlike the pan lysimeters, statistical analysis of suction lysimeter results showed significant differences ($\alpha = 0.05$) for a number of treatment interactions for both total nitrogen and ammonium. Because these trends were similar for both nitrogen forms, ammonium will be the focus of this discussion, with references to total nitrogen as appropriate. A number of interactions were statistically analyzed and included:

- Application rate
- Tree density
- Application rate by tree density
- Position
- Application rate by position
- Tree density by position
- Application rate by tree density by position
- Quarter
- Application rate by quarter
- Tree density by quarter

- Application rate by tree density by quarter
- Position by quarter
- Application rate by position by quarter
- Tree density by position by quarter
- Application rate by tree density by quarter.

When evaluating the results, the more complex statistically significant interactions were first considered because they impart more detail on what experimental condition is influencing the differences most. In addition, the more complex interaction will capture any of the simpler interactions represented by the included conditions.

Controls vs. other application rates

Comparison of controls (0 kgN/ha and 0 trees/ha) to other application rates and tree density combinations showed significant differences ($\alpha = 0.05$) for time (i.e., quarter) and position. With regard to time, an internal comparison of control results across quarters revealed no consistent trends. Quarter 4 was not significantly different than quarters 6 and 7, though quarter 5 was less than quarters 4, 6, and 7, and quarter 6 was less than quarter 7.

Comparison of controls to other application rates showed that all control results for each of the four quarters (i.e., quarters 4, 5, 6 and 7) were significantly less than almost all other quarterly results for all other combinations of application rate and tree density. Figure 98 provides a scatter plot of quarterly results by each application rate to demonstrate these differences. Each set of application rate results is offset from the other within each quarter designation on the plot to ensure that these values are readily seen for comparison.



Figure 98. Suction lysimeter quarterly average ammonium results: controls vs. all other application rates.

Evaluation of differences between positions (i.e., PV15, PV30, PV60, PL15, and PL30) showed an internal control difference as well as differences from other application rates. Comparing control position results to one another, the PV60 position was significantly higher than PV15, PV30 and PL30 (though not PL15). Figure 99 below plots the control results across positions, definitively showing that results from the PV60 position does contain results that are higher than most other positions. While most results from all positions are near or below 10 mg/L, a cluster of results in the PV60 set is notably higher with values between 35-55 mg/L. These values are from the same suction lysimeter (and therefore the same control subplot, 4A) over all four quarters. After evaluating the installation notes and sample collection logs, no notable differences

between this suction lysimeter and others was found. The values from this lysimeter are consistent across time and represent the high end of ammonium concentrations found in background levels at the site.



Figure 99. Suction lysimeter quarterly average ammonium results by position for controls.

Comparison of controls to other application rates again shows a general trend of control results being less than results from other application rates across all positions, though some positions produced more statistically significant differences ($\alpha = 0.05$) than others. The number of non-control application rate by tree density by position combinations equals 3*3*5 = 45 different combinations against which each of the five positions from the controls can be compared. Table 20 below provides a breakout of the number of statistically significant ($\alpha = 0.05$) differences (evaluated by LSD) associated

with each control position. In all of the noted instances, the controls were significantly less than the other application rates.

	Number of significantly different results (out of 45 possible)		
Control Position	Total N	$\mathbf{NH_4}^+$	
PV15	43	44	
PV30	40	43	
PV60	22	18	
PL15	39	41	
PL30	38	44	

Table 20. Tabulation of control by position results that are less than other treatments.

It is evident that the control values from all but the PV60 control positions were significantly less than almost all other application rate-tree density-position combinations. The control PV60 position was significantly lower than most of the PV15 and PV30 positions from all other application rate-tree density combinations (accounting for the 22 and 18 significant differences cited in Table 20). It was not always different, however, from the PV60, PL15 and PL30 positions from the non-control treatments. This is consistent with the previously noted trend for the non-control application rates, in which vertical flow lysimeter results decreased with distance from the biosolids row, and lateral flow lysimeter results were generally lower than all vertical flow lysimeter results. For the non-control treatments, these positions produced lower results more likely to overlap with the PV60 control values.

This trend is better depicted in Figure 100 below. The control values for PV15, PV30, PL15, and PL30 all are in a lower range bracket than the corresponding noncontrol application rates. The PV60 control values, however, overlap with the values for the other application rates, not only within the PV60 position, but also across the PL15 and PL30 positions. Notwithstanding these exceptions, the statistical evaluations
definitively show that the control results were significantly lower than all other treatments.



PV=Vertical placement below biosolids row; PL=lateral placement from edge of biosolids row

Figure 100. Suction lysimeter quarterly average ammonium results: comparison of application rates for each suction lysimeter position.

Differences between and within non-control treatments

Evaluation of differences for all non-control treatments showed the following

interactions to be statistically significant:

- Application rate by tree density by quarter (for both ammonia and total nitrogen)
- Tree density by position by quarter (for both ammonia and total nitrogen)
- Application rate by position by quarter (for total nitrogen only)

The "application rate by tree density by quarter" interaction produced significant differences ($\alpha = 0.05$) between quarters within a given application rate-tree density combination. No differences were generated across application rate-tree density combinations. In most cases, quarter 4 (i.e., the first of the four quarters associated with suction lysimeter collection) was significantly less than successive quarters 5, 6 and 7. Though not as prevalent as with quarter 4, in some instances quarters 5 and 6 were also significantly less than successive quarters. This may indicate an overall trend of an increase in concentration with time, but it is important to note that the trend is not consistent and is generalized, because it does not differentiate between suction lysimeter positions (i.e., the "application rate by tree density by position by quarter" interaction was not significantly different). In addition, not all application rate-tree density combinations exhibited such differences, though a majority did. With these noted reservations, this statistical analysis may indicate an increase in the leaching of total nitrogen and ammonium over time. This would be consistent with the ongoing dewatering of the biosolids over time, assuming dewatering had not yet started to decline. Table 21 provides a listing of which treatments produced different quarterly results (i.e., those that exhibited the general trends in quarterly increases noted above) and which ones did not. Figures 101-103 present the results for the three application rate-tree density combinations that most clearly show a consistent statistically significant trend over time. Given that these differences may not be visually obvious (due to the logarithmic scale and variability of the data), the statistically significant differences are noted in the figures. Note that each quarter captures results from the three blocks, each of which contain five suction lysimeter positions, for a maximum of 15 results.

Treatments with quarterly differences		Treatments without quarterly differences	
(kg N/ha – trees/ha)		(kg N/ha – trees/ha)	
Total Nitrogen	Ammonium	Total Nitrogen	Ammonium
19,650 - 0	19,650 - 0	19,650 - 1074	19,650 - 1074
19,650 - 716	19,650 - 716	39,300 - 0	
	39,300 - 0	58,900 - 716	58,900 - 716
39,300 - 716	39,300 - 716		
39,300 - 1074	39,300 - 1074		
58,900 - 0	58,900 - 0		
58,900 - 1074	58,900 - 1074		

Table 21. Application rate-tree density combinations with quarterly differences.



Figure 101. Suction lysimeter quarterly average ammonium results for 19,650 kg N/ha - 716 trees/ha (application rate * tree density * quarter interaction).



Figure 102. Suction lysimeter quarterly average ammonium results for 39,300 kg N/ha - 716 trees/ha (application rate * tree density * quarter interaction).



Figure 103. Suction lysimeter quarterly average ammonium results for 58,900 kg N/ha - 0 trees/ha (application rate * tree density * quarter interaction).

Both the "tree density by position by quarter" and "application rate by position by quarter" interactions exhibited some differences over time (i.e., quarters) in specific instances, but were dominated more by differences between the suction lysimeter positions, with a general pattern of PV15 > PV30 > PV60. In addition PV15 was almost always greater than PL15 and PL30 and PV30 was often greater than PL15 and PL30. Differences between PV60, PL15 and PL30 were not as pronounced.

For the "tree density by position by quarter" interaction, the PV15 position for each tree density across all quarters was significantly greater than some of the PV30 and PV60 positions, and almost all of the PL15, and PL30 positions for all quarters across all tree densities. PV30 exhibited differences from PL15 and PL30 across many quarters.

Differences between PV30 and PV60 were not pronounced, nor were differences between PV60 and PL15 and PL30. Comparison of each position to itself across the three tree densities showed no differences between tree densities (e.g., PV15 quarterly results from 0 trees/ha, 716 trees/ha and 1074 trees/ha were not different from one another). Consequently, no major differences exist between tree densities; it is more a product of position.

Further evaluating results of the "tree density by position by quarter" interaction, an investigation of each position within a tree density across quarters does show some instances in which concentrations increase over time. The following tree density-position combinations show an increase in concentrations from quarters 4 and/or 5 to quarters 6 and/or 7, though in many of these instances, the trend is not completely sequential over all quarters: 1) for the 0 trees/ha tree density, the PV15, PV30, PV60 and PL30 positions show increases over time (quarters); 2) for the 716 trees/ha tree density, the PV15 and PL15 positions show increases over time (quarters); 3) for the 1074 trees/ha tree density, the PV30 and PL15 positions show increases over time (quarters); 3) for the 1074 trees/ha tree density, the PV30 and PL15 positions show increases over time (quarters); 3) for the 1074 trees/ha tree density, the PV30 and PL15 positions show increases over time (quarters); 3) for the 1074 trees/ha tree density, the PV30 and PL15 positions show increases over time (quarters); 3) for the 1074 trees/ha tree density, the PV30 and PL15 positions show increases over time (quarters); 3) for the 1074 trees/ha tree density, the PV30 and PL15 positions show increases over time (quarters).

Figures 104-107 present a subset of the data, specifically from the 0 trees/ha tree density, to provide examples of the specific statistically significant differences ($\alpha = 0.05$) determined. Because differences may not be obvious visually (due to the logarithmic scale and variability of results), they are noted in each figure. Note that each quarter captures results from the three blocks, each of which contains four application rates, for a maximum of twelve results.



Figure 104. Suction lysimeter quarterly average ammonium results for 0 trees/ha, PV-15cm (tree density*position*quarter interaction).



Figure 105. Suction lysimeter quarterly average ammonium results for 0 trees/ha, PV-30cm (tree density*position*quarter interaction).



Figure 106. Suction lysimeter quarterly average ammonium results for 0 trees/ha, PV-60cm (tree density*position*quarter interaction).



Figure 107. Suction lysimeter quarterly average ammonium results for 0 trees/ha, PL-30 cm (tree density*position*quarter interaction).

The "application rate by position by quarter" interaction was significant for total nitrogen only, one of the few instances in which the statistical results were not consistent between total nitrogen and ammonium. Because of the similarity in results between the two parameters, it may simply be a matter of total nitrogen exhibiting differences slightly more pronounced than ammonium such that total nitrogen crossed the threshold of being statistically significant.

Statistical significance was determined between positions. Similar to findings from other interactions, all PV15 values across all quarters for 19,650 kg N/ha, 39,300 kg N/ha, and 58,900 kg N/ha were significantly greater than many PV30 and PV60 values and almost all PL15 and PL30 values across all quarters and application rates. PV15 values from one application rate were not different the other two application rates (i.e., differences were not found between application rates).

PV30 values did not exhibit as pronounced differences from the PV60 position, but were often greater than the laterally positioned lysimeters (PL15 and PL30) across application rates and quarters. Figure 108 below shows total nitrogen results for each application rate by position. Figure 100 above, previously presented when evaluating control results, shows the same results for ammonium. Although this interaction for ammonium was not statistically significant, it is evident that both sets of results follow a similar pattern.



Figure 108. Suction lysimeter total nitrogen results by position for each application rate.

As with the "tree by position by quarter" interaction, the "application rate by position by quarter" interaction showed some quarterly trends within a particular application rateposition combination. The following application rate-position combinations show an increase in concentrations from quarters 4 and/or 5 to quarters 6 and/or 7, though in many of these instances, the trend is not completely sequential over all quarters:

- For the 19,650 kg N/ha application rate, the PV60 and PL30 positions show increases over time (quarters).
- For the 39,300 kg N/ha application rate, the PV15, PV30, PV60, and PL30 positions show increases over time (quarters).
- For the 58,900 kg N/ha application rate, the PV15, PV30, PV60, and PL15 positions show increases over time (quarters).

Figures 109-112 below present a subset of the data, specifically from the 58,900 kg N/ha application rate, to provide examples of the specific statistically significant differences ($\alpha = 0.05$) determined. Because differences may not be obvious visually (due to the logarithmic scale and variability of results), they are noted in each figure. Note that each quarter captures results from the three blocks, each of which contains three tree densities, for a maximum of nine results.



Figure 109. Suction lysimeter quarterly average total nitrogen results for 58,900 kg N/ha, PV-15cm (application rate*position*quarter interaction).



Figure 110. Suction lysimeter quarterly average total nitrogen results for 58,900 kg N/ha, PV-30cm (application rate*position*quarter interaction).







Figure 112. Suction lysimeter quarterly average total nitrogen results for 58,900 kg N/ha, PL-15cm (application rate*position*quarter interaction).

Conclusions

More than half of both the pan and suction lysimeter samples contained ammonium concentrations in excess of 100 mg/L, with some values greater than 1000 mg/L. For the pan lysimeters, the higher concentrations were distributed fairly evenly across application rates, but were more prevalent in the 0 trees/ha tree density. Statistical analysis of the pan samples, however, showed no significant differences (α = 0.05) for application rate (which included the controls), tree density, or time.

Suction lysimeters provided more distinct differences. Unlike the pan lysimeters, higher concentrations were not associated with 0 trees/ha, but rather were distributed evenly across tree densities. Comparison of application rates did show the controls to be significantly less than other application rates, but no overriding trend was determined between the non-control application rates.

The most notable trend from both observational and statistical analysis is that an inverse relationship exists between depth below the biosolids row and ammonium concentration. As depth increases, concentration decreases, suggesting that ammonium is leaching out of the biosolids row and, over the course of this experiment, the highest concentrations have thus far reached the first suction lysimeter. An alternative explanation for the lower concentrations in the deeper vertical lysimeters could be that ammonia is nitrifying to produce nitrate, with subsequent denitrification of nitrate. This explanation is not as plausible, however, given the negligible concentration of nitrite and nitrate found in all samples (see subsequent sections for results) and the improbability of enough oxygen reaching areas below the biosolids within the first couple of years. Even if nitrate was denitrifying once it presumably was produced and leached to lower, more

oxygen-deprived locations, at some point a sample would be collected that captured nitrate before it leached further in the profile and/or was denitrified.

An overall increase in concentration with time has also been indicated within each lysimeter position, though further data collection over time will be necessary to provide more insight to the extent of this trend.

Soil Water Results: Nitrite (NO₂⁻) Data

Nitrite is produced from the breakdown of ammonia in the first part of the two-step process of nitrification. Aerobic conditions, the appropriate microbes, and a carbon source for the microbes are required. Upon application, the biosolids were encased in the overburden/soil from the site and were saturated with water, effectively impeding the flow of oxygen to the rows. During design of the experiment it was hypothesized that conversions to nitrite would be significantly diminished until water drained from the biosolids and oxygen was introduced to deeper layers of the soil via tree roots and drying processes. In addition, physical and chemical conditions in the soil would need to be favorable for the growth of the *Nitrosonomas* and other bacteria responsible for this conversion.

Pan Lysimeter Samples

Pan lysimeter results show consistently low levels of nitrite across all treatments, with few exceptions. Of the 430 results generated, 422 (98%) were less than the EPA drinking water MCL of 1.0 mg/L. The eight remaining results above 1.0 mg/L ranged from 1.2-30 mg/L. When individual subplot values were averaged within each quarter, the 430 results were consolidated to 223 results. Depictions of quarterly results for each block are provided in Figures 113-115. Results are plotted on a logarithmic scale.



Figure 113. Nitrite quarterly average concentrations for low-level application rate in pan lysimeters.



Figure 114. Nitrite quarterly average concentrations for mid-level application rate in pan lysimeters.



Figure 115. Nitrite quarterly average concentrations for high-level application rate in pan lysimeters.

Three groups of higher values (> 1.0 mg/L) stand out: (a) the quarter 1 value for the control (0 kgN/ha; 0 trees/ha) in Block 2, which is identified as subplot 4B (b) the quarter 1 value for 19,650 kgN/ha; 0 trees/ha in Block 2, which is identified as subplot 2I and (c) quarter 3, 4, 6,7 and 8 values for 58,900 kgN/ha; 0 trees/ha, all in Block 2, which is identified as subplot 2C.

In the case of the control, the high concentration is associated with subplot ID 4B (the control in Block 2) and is succeeded in quarter 2 by a lower, though still slightly elevated value of 0.44 mg/L (elevated, comparatively speaking, from the rest of the data) and a non-detect value in quarter 3. This indicates that a nitrogen source was present in the control at the beginning of collection activities that was also exposed to enough oxygen and microbes to facilitate some level of nitrification. In addition, the nitrification

products were transported approximately 90-100cm through the soil profile (or less if the nitrification occurred below the surface) to the pan lysimeter.

A plausible explanation for this initial spike (albeit a low level spike) in concentration and ensuing subsidence would be the disturbance during installation of the pan lysimeter of the thick vegetative cover that was established at the control subplot. According to a University of Wisconsin-Extension Fact Sheet (Korb, et al., 1999), grass clippings are organic fertilizers with 3-4% nitrogen on a dry weight basis, and grass clippings from a 1,000 ft² (92.9m²) lawn would supply 0.25 lbs (0.11kg) of nitrogen. The surface area disturbed when the installation trench was dug for the pan lysimeter installation was approximately $3m \ge 1.5 m = 4.5m^2$. This represents 5% of the $92.9m^2$ surface area that supplied 0.11kg of nitrogen. Therefore, the disturbed area could supply approximately 0.005kg (500mg) of nitrogen. Dependent upon the rate of decomposition, rainfall, flow paths in the subsurface profile, and other environmental factors, it is feasible that some portion of this nitrogen could have been converted to water-soluble nitrite and been transported to the pan to produce a final concentration level of 1mg/L. Because no biosolids were applied to the control site, this initial grass cover would likely be the only appreciable source of nitrogen, though other forms of organic matter (organisms, etc.) in the vegetative cover or in the disturbed soil profile could also contribute nitrogen. Consequently, the decrease and eventual absence of nitrite in the pan lysimeter samples over time is logical.

Provided conditions were favorable for the *Nitrobacter* (and other) organisms that convert nitrite to nitrate, it is more likely that nitrate would be present in higher concentrations than nitrite, since the conversion from nitrite to nitrate is usually rapid.

Therefore, the subsequent section presenting results on nitrate will also examine the values for these same samples.

It is worthwhile to point out that the nitrite results from the other two controls were non-detects or near non-detects for quarters 1 and 2, with a slightly higher result for both blocks in quarter 3 (0.02 mg/L) and oscillations between non-detects and 0.05 mg/L over quarters 4-8. Such low levels are representative of background levels. It might be expected that the same vegetative cover disturbance in these other two controls should produce the same spike in concentration. The hydraulic conductivities in the uppermost layer (30-40cm) of these subplot surfaces, however, were $1.0x10^{-6}$ cm/sec for control block 1 and $1.8x10^{-5}$ cm/sec for control block 3, which is much lower than the shallow depth conductivity of $2.3x10^{-4}$ cm/sec for control block 2. Consequently, flow through these two subplots proceeded at slower rates than in control block 2. This could allow more time for conversion of nitrite to nitrate followed by immobilization or denitrification. It also could be that the disturbed vegetative cover at these control plots decomposed at the surface and were washed to another location during a storm event, or that any decomposition products percolated outside the pan lysimeter area.

It is also important to note that although cover vegetation was a potential source of nitrogen at the control subplots, a vegetative cover was not present over the plots in which the biosolids were applied, because the site had been leveled and any cover destroyed months before the biosolids application commenced.

The second incidence of nitrite values >1.0 mg/L occurred in quarter 1 at one of the three 19,650 kgN/ha; 0 trees/ha subplots (the block 2 subplot, subplot ID = 2I). After this initial higher average value of 1.69 mg/L, concentrations oscillated between non-detects

and 0.02 mg/L. Concentrations in the other two subplots associated with blocks 1 and 3 oscillated between non-detects and 0.07 mg/L. This level of nitrogen could be associated with nitrite present in the biosolids at the time of application or nitrite that was formed from the ammonium present in the biosolids. Evaluation of pan installation documentation indicates nothing out of the ordinary (i.e., no disturbance or difficulty during the installation process that could have created an out of the ordinary flow path or source of nitrification). Sample collection was also routine, with approximately 3000mL of volume collected for the sample associated with quarter 1. As with other incidences of notable nitrite values, the nitrate concentrations associated with this sample will be evaluated.

The third group of nitrite values > 1.0 mg/L occurred in greater quantity than the prior two incidences, and are associated with the 58,900 kgN/ha; 0 trees/ha treatment. The highest values (those >1.0 mg/L) are all associated with the block 2 subplot (ID = 2C). These high values were as follows: q3 = 3.8mg/L; q4 = 1.3mg/L; q6 = 4.0mg/L; q7 = 13.2 mg/L; q8 = 30.2mg/L. Sample was not available for q5 because no water had flowed into the pan during this quarter (i.e., from March-May, 2004). Pan lysimeter collections from this subplot were characterized by low volumes (< 125mL), which could result in greater concentration of solutes per unit volume. Sample pH ranged from 7.5-8.12, which indicates that some high pH leachate from the limed biosolids reached the pan.

Overall, Figure 115 shows an increase in nitrite concentration over time for the interval considered (i.e., Q1-Q8 = April 2003-December 2004). It is evident that a source of nitrite was available to subplot 2C. The other two subplots associated with this

application rate by tree density combination (1I and 3F) produced lower values that ranged between non-detects and 0.5 mg/L. These subplots also captured larger volumes of leachate than subplot 2C (750-10,000mL for 1I; 75-225mL for 3F) and pH levels were in a more neutral range (6.91-7.15 for 1I; 6.25-7.0 for 3F). Nitrate concentrations will be evaluated in conjunction to determine if conversion to nitrate is occurring as well.

Statistical Analysis

As with ammonium, statistical analysis encompassed evaluation of the following interactions for significance:

- Application rate
- Tree density
- Application rate by tree density
- Quarter
- Application rate by quarter
- Tree density by quarter
- Application rate by tree density by quarter

When evaluating the results, the more complex statistically significant interactions were first considered because they impart more detail on what experimental condition is influencing the differences most. In addition, the more complex interaction will capture any of the simpler interactions represented by the included conditions.

Statistical analysis of pan lysimeter results showed significant effects for 1) application rate by tree density interactions and 2) quarterly time intervals. Further evaluation through LSD showed the 58,900 kgN/ha by 0 trees/ha combination to be significantly different from all other application rate by tree density combinations, which is not surprising, given the results shown in Figures 113-115. No other application rate by tree density combinations were different from one another (including controls). Quarterly differences were general, and showed quarters 3, 4, 5, and 6 to be lower than quarters 7 and 8. The scatter plot of nitrite results shown in Figure 116 below highlights these findings.



Figure 116. Scatter plot of nitrite quarterly average results for pan lysimeters.

The plot clearly demonstrates a trend of 58,900 kgN/ha; 0 Trees/ac results that are markedly higher than all other values for most quarters. It is also apparent that the results from all treatments have lower overall values in quarters 3, 4, 5, and 6 compared to quarters 1, 2, 7 and 8, with quarters 7 and 8 being different enough the elicit a statistically significant response.

Suction Lysimeter Samples

Of the 1453 results generated from the suction lysimeters, 1448 (99.6%) were less than the EPA NO₂-N MCL of 1.0 mg/L. The six remaining results ranged from 1.0-32 mg/L, similar to the pans. All quarterly results are shown in Figures 117-125 below. Data plots show quarterly values for each block and depth. The initial data plot with all four quarters represented is included to provide an overall view of results over time. Because of the number of data points, however, the resolution of individual application rate by tree density combinations was compromised. Consequently, the bar charts are further separated into two charts, each of which includes two quarters. Within these charts, quarter, block and position designations are noted. Because of the preponderance of low-level results, all figures use a logarithmic scale to present the data.

Recall that five suction lysimeters were installed within each of the 30 subplots. Three capture vertical flow and were positioned 15, 30, and 60 cm underneath the bottom of the biosolids rows. Two others were positioned 15 and 30cm lateral from the edge of a biosolids row at a depth equal to the bottom of the biosolids row (Figure 6). In the bar charts and tables that follow, these positions are indicated as PV15, PV30, PV60, PL15, and PL30, respectively. As a reminder, the time periods associated with each of the suction lysimeter quarterly designations include:

Q4 = November 2003 - February 2004

Q5 = March 2004 - May 2004

Q6 = June 2004 – August 2004 (recall that samples were not collected in July) Q7 = October 2004 – December 2004 (recall that samples were not collected in November).



Figure 117. Nitrite quarterly average concentrations for low-level application rate in suction lysimeter (SL) samples – all quarters.





SL Figure 119. Nitrite quarterly average concentrations for low-level application rate in samples: Q6&Q7.















SL Figure 125. Nitrite quarterly average concentrations for high-level app. rate for samples: Q6&Q7.

Quarter-Block-Position ID

Q7-B3-PL30

Q7-B3-PL15

Q7-B3-PV60

Q7-B3-PV30

Q7-B3-PV15

Q7-B2-PL30

Q7-B2-PL15

Q7-B2-PV60

Q7-B2-PV30

Q7-B2-PV15

Q7-B1-PL30

Q7-B1-PL15

Q7-B1-PV60 Q7-B1-PV30

Q7-B1-PV15

Q6-B3-PL30

Q6-B3-PL15

Q6-B3-PV60 Q6-B3-PV30

Q6-B3-PV15

Q6-B2-PL30 Q6-B2-PL15

Q6-B2-PV60

Q6-B2-PV30

Q6-B2-PV15

Q6-B1-PL30

Q6-B1-PL15

Q6-B1-PV60

Q6-B1-PV30

Q6-B1-PV15

0.00

0.01

When individual position values for each subplot were averaged within each quarter, the 1453 results were consolidated to 562 results. This also resulted in consolidation of the initial six values to three values that were greater than 1.0 mg/L. These three values (with accompanying monthly values) included:

- 1.23 mg/L for 19,650 kgN/ha; 0 trees/ha for Q7-B1-PL15 (quarter 7, block 1, lateral SL 15cm from the side of the biosolids row, subplot ID=1F). Monthly values for quarter 7 were 2.37 mg/L for October 2004 and 0.09 mg/L for December 2004.
- 1.39 mg/L for 19,650 kgN/ha; 716 trees/ha for Q7-B1-PV30 (quarter 7, block1, vertical SL 30cm below the biosolids row, subplot ID=1D). Monthly values for quarter 7 were 1.57 mg/L for October 2004 and 1.21 for December 2004.
- 27.8 mg/L for 58,900 kgN/ha; 716 trees/ha for Q7-B1-PL15 (quarter 7, block 1, lateral SL 15cm from the side of the biosolids row, subplot ID=1G). Monthly values were 32.1 mg/L for October 2004 and 23.5 mg/L for December 2004.

All values are associated with quarter 7. Individual October and December values from quarter 7 were both greater than 1 mg/L with the exception of the December 2004 value for Q7-B1-PL15. Values for the prior three quarters were all less than 1 mg/L. Values are associated with both laterally and vertically positioned suction lysimeters. The only commonality between these results and those from the pan lysimeters is the 19,650 kgN/ha; 0 trees/ha application, though the quarter and block are different from the single pan lysimeter high value from that treatment. Data from future analyses will need to be evaluated to determine whether or not these higher values signal the start of a trend, or are isolated incidents. Due to the fact that these represent an extremely small number of values from the experiment, the overriding conclusion is that insignificant amounts of

nitrite are present in the soil profile. Nitrate values for these samples will also be evaluated to determine if complete nitrification occurred.

Rainfall Flushing Effect

As with the high concentration ammonium samples, the five monthly nitrite values greater than 1 m/L were also evaluated to determine if they reflect a flushing effect from storm events. The protocol used was described in the ammonium results section. Of the five results identified, all of which were from block 1, subplots 1D, 1F and 1G, only 1 (i.e., 20%) was linked to a storm event. It therefore does not appear that a flushing effect was responsible for the higher nitrite results.

Statistical Analysis

As with the ammonium results, the same interactions were statistically analyzed and included:

- Application rate
- Tree density
- Application rate by tree density
- Position
- Application rate by position
- Tree density by position
- Application rate by tree density by position
- Quarter
- Application rate by quarter
- Tree density by quarter
- Application rate by tree density by quarter

- Position by quarter
- Application rate by position by quarter
- Tree density by position by quarter
- Application rate by tree density by quarter.

When evaluating the results, the more complex statistically significant interactions were first considered because they impart more detail on what experimental condition is influencing the differences most. In addition, the more complex interaction will also capture any of the simpler interactions represented by the included conditions.

Statistical analysis showed no significant differences ($\alpha = 0.05$) between any of the application rate or tree density combinations (including controls) and no significant differences for any of these treatments over time. Statistically significant differences did occur, however, for position by quarter interactions both within and between positions. A comparison within each position across quarters does show an increase in concentrations from quarters 4 and/or 5 to quarters 6 and/or 7, though in many of these instances, the trend is not completely sequential over all quarters. Within position differences are visually depicted in Figures 126-130. It is important to note that these trends are general across all experimental conditions and are not associated with a specific application rate or tree density.



Figure 126. Nitrite quarterly average results: Suction lysimeter 15 cm vertical positionwithin position differences.



Figure 127. Nitrite quarterly average results: Suction lysimeter 30 cm vertical positionwithin position differences.



Figure 128. Nitrite quarterly average results: Suction lysimeter 60 cm vertical positionwithin position differences.



Figure 129. Nitrite quarterly average results: Suction lysimeter 15 cm lateral positionwithin position differences.



Figure 130. Nitrite quarterly average results: Suction lysimeter 30 cm lateral positionwithin position differences.

Although statistically significant differences ($\alpha = 0.05$) were noted for each position over time, and in general those trends show higher levels in later quarters compared to earlier quarters, these charts depict an overriding facet of the data: comparisons mostly involve such low levels that the differences noted are inconsequential. As previously stated, controls were no different than any other application rate and tree density combinations. With the exception of a couple of higher values, all of these results are equivalent to background levels.

Statistically significant differences ($\alpha = 0.05$) also were established between positions over different quarters. In general, the 15cm vertical position (PV15) values across all quarters were greater than many of the values from the other positions over all quarters. The 30 cm vertical position (PV30) values also exhibited some significant
differences (αfrom the 15cm and 30 cm lateral position (PL15 and PL30) values across quarters. Differences between PV60, PL15 and PL30 were not notable. What these results show is not a particular increase or decrease over time, but more a trend of relatively higher values in the suction lysimeters positioned closest to the biosolids rows. As was noted above, however, *all* values were extremely low, such that the differences noted do not bear any applied significance. A comparison of the values for the vertical suction lysimeters across quarters is provided in Figure 131 to show the relatively higher values of PV15 compared to PV30 and PV60. Figure 132 provides a comparison of the PV15 position to the lateral positions (PL15 and PL30).



Figure 131. Nitrite suction lysimeter quarterly average results: comparison of vertical positions over time.



Figure 132. Nitrite suction lysimeter quarterly average results: comparison of vertical 15cm position to lateral positions over time.

The figures above depict trends in which the PV15 values, though not always higher than all values from other positions, do show a greater frequency of values in higher concentration ranges than the other positions.

Conclusions

Overall, nitrite concentrations were low, with suction lysimeter samples exhibiting slightly lower values that those from the pan lysimeters. For pan lysimeters, the 58,900 kg N/ha-0 trees/ha combination produced significantly higher values than all other application rate-tree density combinations, though thus far the high values are associated with only one of the three replicates for this treatment. All other application rate-tree

density combinations, including controls, were not statistically different from one another. A very general trend of quarterly results potentially increasing over time was determined statistically, but applied to all experimental conditions collectively (i.e., no application rate and/or tree density by quarter interactions were significant).

Suction lysimeter results showed no significant differences ($\alpha = 0.05$) between or within application rate and/or tree density (including controls), nor were differences found for application rate and/or tree density over time. Some differences were noted within and between suction lysimeter positions. Within a position, concentrations from earlier quarters (Q4 and Q5) were sometimes different from the latter quarters (Q6 and Q7), though not consistently so. Across positions, differences were more a product of position rather than exhibiting a trend over quarters. In general, PV15 results contained the greatest frequency of higher values than other positions. A spike in values during quarter 7 (October-December 2004) for the 15 cm lateral suction lysimeter in one replicate of the 58,900 kgN/ha-716 trees/ha treatment may indicate the presence of mineralization, though future collection events will need to be evaluated to determine if a trend forms.

Though the above-noted statistical differences were ascertained, the overriding conclusion is that nitrite values were so low across all experimental conditions that they are not considered to have any applied significance.

Soil Water Results: Nitrate (NO₃⁻) Data

Nitrate is the final product of the two-step nitrification process that oxidizes ammonia to nitrite and then nitrate. The transition from nitrite to nitrate is usually rapid, provided conditions are suitable for the *Nitrobacter* (and other) bacteria that perform the conversions. Similar to *Nitrosonomas*, these conditions include: the presence of nitrite, oxygen, a carbon source in the form of carbon dioxide or bicarbonates, appropriate temperature, pH, and absence of toxic compounds. Once nitrate is produced, it can be: immobilized by microbes; consumed by the poplar trees; converted to gaseous forms via denitrification processes; and/or leach through the soil profile with the flow of water. Collection of samples over time from both pan and suction lysimeters in close proximity to the biosolids rows provides an evaluation of the occurrence and transport of nitrate in the soil at this site.

Pan Lysimeter Samples

Pan lysimeter results show consistently low levels of nitrate across all treatments, with few exceptions. Of the 426 results generated, 423 (99%) were less than the EPA drinking water MCL of 10 mg/L. The three remaining results above 10 mg/L ranged between 13.7 - 37.6 mg/L. When individual subplot values were averaged within each quarter, the 426 results were consolidated to 220 results. Depictions of quarterly results for each block are provided in Figures 133-135. To provide further insight into the lower level values, results are plotted on a logarithmic scale.



Figure 133. Nitrate quarterly average concentrations for low-level application rate in pan lysimeters.



Figure 134. Nitrate quarterly average concentrations for mid-level application rate in pan lysimeters.



Figure 135. Nitrate quarterly average concentration for high-level application rate in pan lysimeters.

Focusing on those values close to and greater than 1mg/L, the following results shown in Table 22 below, sorted by application rate and tree density, warrant a closer look.

Subplot ID	Block	App. Rate (kgN/ha)	Tree Density (trees/ha)	quarter	Time Period	# monthly results averaged	NO ₃ -N Mean
4B	2	0	0	1	April-May 2003	1	37.59
4B	2	0	0	2	June-Aug. 2003	3	6.38
1E	1	19,650	1074	7	October 2004	1	1.20
3I	3	39,300	0	1	April-May 2003	1	0.72
1B	1	39,300	1074	4	Dec. 2003-Feb. 2004	3	0.61
1B	1	39,300	1074	5	March-May 2004	3	0.87
1B	1	39,300	1074	6	June-Aug. 2004	2	0.63
3G	3	39,300	1074	8	December 2004	1	0.65
2C	2	58,900	0	3	SeptNov. 2003	2	0.94
2C	2	58,900	0	4	Dec. 2003-Feb. 2004	2	0.74
2C	2	58,900	0	6	June-Aug. 2004	1	2.54
2C	2	58,900	0	7	October 2004	1	2.16
2C	2	58,900	0	8	December 2004	1	3.50
2A	2	58,900	716	1	April-May 2003	1	3.65
2B	2	58,900	1074	1	April-May 2003	1	13.66

Table 22. Nitrate pan lysimeter results close to and greater than 1 mg/L.

The subplot with the highest consecutive results is the control from block 2 (subplot ID=4B). These higher results from quarters 1 and 2 are consistent with those presented for nitrite values in the previous section. As previously explained, because biosolids were not applied to the control sites, another source of nitrogen had to account for the surge in concentration. The most likely candidate is the vegetation that covered the subplot area prior to installation of the pan lysimeter. Concentrations continued to decline in quarter 3, with a value of 0.05 mg/L, and then oscillated between non-detects and 0.03 mg/L during the rest of the time covered, as is shown in either of Figures 133-135. Results from controls in the other two blocks ranged between non-detects and a maximum of 0.12 mg/L, reflecting low level background concentrations. Figure 136 below presents these control values by each block over the eight quarters.



Figure 136. Nitrate: Pan lysimeter average quarterly values for each control subplot.

Isolated higher-level values were evidenced for subplots 1E, 3I, 2A, and 2B. Most of these values were well below any level of concern, but subplot 2B exhibited a concentration of 13.7 mg/L, which exceeds the EPA NO₃-N drinking water MCL of 10 mg/L. Although it is not a goal of this operation to produce leachate in close proximity to the biosolids that meets drinking water standards, such limits are a good benchmark for discussing potential environmental concerns. All other results from both this subplot and the other two at the 58,900 kgN/ha-1074 trees/ha treatment produced low-level results ranging from non-detects to 0.09 mg/L. Nitrite values were low for this subplot as well. Suction lysimeter results will be evaluated to determine if they reflect this apparent isolated event in subplot 2B.

Two application rate/tree density combinations showed slightly elevated results over multiple quarters. The first set includes data from the 39,300 kgN/ha-1074 trees/ha treatment (specifically subplots 1B and 3G), which collectively produced nitrate results ranging from 0.61-0.87 mg/L during quarters 4, 5, 6, and 8. All other results for all three subplots ranged from non-detects to 0.14 mg/L. Though not discussed in the nitrite section because results were so low, nitrite results did show a similar, though less prevalent pattern for this treatment. In quarters 5 and 6, subplot 1B produced nitrite concentrations of 0.14 and 0.20 mg/L, respectively. Although these levels are not of concern, they are slightly higher than the other results for this treatment. Consequently, some nitrification may have occurred.

The second set includes results from a single subplot associated with the 58,900 kgN/ha-0 trees/ha treatment (subplot 2C). As previously shown in Table 22, results increased with time from quarters 3 through 8. This pattern is also similar to those for the nitrite results, although in this instance the nitrite values were higher than nitrate. Given the usual rapid conversion of nitrite to nitrate as it is being produced, nitrite is typically present at lower levels than nitrate. This transposition indicates that either 1) the sample was collected at the specific time when the nitrite was produced and had not yet been converted to nitrate, 2) as soon as nitrate was being produced, it encountered an anaerobic microsite and underwent denitrification and/or 3) the conversion of nitrite to nitrate was inhibited.

Inhibition could occur if conditions were more hostile to the *Nitrobacter* population (i.e., the microbes that convert nitrite to nitrate) than the *Nitrosonomas* population (i.e., the microbes that convert ammonium to nitrite). Elevated salt concentrations, as are

present in the leachate from heavily limed biosolids, are known to inhibit microbial growth (Haynes, 1986). Haynes (1986) also reported that high ammonium concentrations have been shown to selectively inhibit *Nitrobacter* species. The maximum tolerable ammonium concentrations under which nitrification would still occur (specifically the conversion of nitrite to nitrate) was 400-800 mg/kg. As is evident from the prior discussion of ammonium results, concentrations in the biosolids as well as the leachate traveling to the pan lysimeters exceed these levels. Consequently, until ammonium concentrations decrease either through conversion to nitrite, immobilization by microbes, or uptake by trees, production of nitrate will be limited in this region of the soil profile.

The time periods (quarters) associated with these two sets of data cover both active and dormant periods in the ecosystem, and therefore do not indicate a cyclical trend. Overall, these levels may indicate an extremely low level of leaching nitrate that was either 1) present in the biosolids upon application, 2) produced when an extremely lowlevel of nitrification occurred or 3) produced when a higher level of nitrification occurred, but the resulting nitrate was immobilized in microbial biomass or taken up by the poplar trees. Regardless, these levels are not of concern from an environmental or health perspective.

Statistical Analysis

Statistical analysis evaluated the same interactions previously presented in the ammonium and nitrite sections. Significant differences (α = 0.05)were determined for the application rate by tree density by quarter interaction, both within and between

application rate/tree density combinations for specific quarters. In most cases, however, consistent trends were not demonstrated.

Controls exhibited statistical differences from other treatments for specific quarters. Overall, the quarter 2 control results were significantly greater than multiple (but not all) quarters across each of the treatments. Subsequent (but not all) quarters for the controls were significantly less than one or more quarters in all other treatments. Note that multiple quarters for the controls were not significantly different than other treatments. Control results from quarter 1, in fact, were not significantly different from any other treatment by quarter combinations. In summary, only isolated instances, and not an overall difference between the controls and other treatments, was demonstrated. This supports the overall observation that nitrate concentrations in the treatments were not different from the controls.

Within treatment differences were examined and are summarized in Table 23 below. In most instances, differences do not exhibit a repeated, sequential trend over time. More often, the differences oscillate or are attributed to one or two relatively higher concentrations that peaked and then diminished in subsequent quarters. The 58,900 kgN/ha; 0 trees/ha treatment shows a potential trend towards increasing values with time, but additional data from later sampling quarters (beyond the scope of this thesis) would need to be evaluated to determine what type of trend was established. Also note that neither of the other tree densities for 58,900 kgN/ha application rate show this pattern.

Application Rate: Tree Density	Significant Quarterly Differences			
0 lbN/ha : 0 trees/ha	Q2 is greater than Q3-Q8			
19,650 kgN/ha : 0 trees/ha	Q2 and Q3 are greater than Q8			
19,650 kgN/ha : 716 trees/ha	Q2 and Q4 are greater than Q5			
19,650 kgN/ha : 1074 trees/ha	Q2 is greater than Q4 and Q5			
	Q3 is greater than Q5			
	Q5 is less than Q7			
39,300 kgN/ha : 0 trees/ha	Q3 is less than Q2, Q4, and Q8			
	Q4 is greater than Q5, Q6			
	Q5 is less than Q8			
	Q6 is less than Q7 and Q8			
39,300 kgN/ha : 716 trees/ha	Q2 and Q3 are greater than Q5 and Q8			
39,300 kgN/ha : 1074 trees/ha	Q5 and Q7 are less than Q8			
58,900 kgN/ha : 0 trees/ha	Q1, Q2, Q3, and Q4 are less than Q6 and Q8			
	Q7 is less than Q8			
58,900 kgN/ha : 716 trees/ha	Q1 is greater than Q3-8			
	Q2 is greater than Q4, Q6, Q7, and Q8			
58,900 kgN/ha : 1074 trees/ha	Q2 is greater than Q5			

Table 23. Nitrate pan lysimeter: application rate by tree density quarterly differences.

Within a given application rate, statistically significant differences in tree densities for specific quarters were determined. An itemized list of the differences is provided in Appendix 2 for reference. For the 19,650 kg N/ha application rate, differences between tree densities were spotty, and did not show a particular trend. For the 39,300 kgN/ha application rate, the 716 trees/ha density had the preponderance of values less than the other tree densities, and 1074 trees/ha were always more than the other densities. Aside from this, no explicit trends were ascertained. Finally, for the 58,900 kgN/ha application rate, the 0 trees/ha density had significantly greater values for multiple quarters compared to other densities. This is consistent with the trend of higher values associated with this application rate/tree density combination, particularly in quarters 6-8.

Between treatment differences were even more convoluted. In general, the previously discussed application rate/tree density/quarter results with relatively higher

values (i.e., > 0.6 mg/L) than general background levels produced significant differences greater than the other application/rate/tree density/quarter values. Beyond this, actual nitrate concentrations were so low, and not consistently different from the controls that these differences do not warrant further evaluation.

Suction Lysimeter Samples

Compared to pan lysimeter results, those from suction lysimeters show even more consistently low levels of nitrate across all treatments. Of the 1454 monthly results generated, 1453 (99.9%) were less than the EPA drinking water MCL of 10 mg/L. The one result above 10 mg/L was 12.5 mg/L. When individual subplot values were averaged within each quarter, the 1454 results were consolidated to 563 results. Results are shown in Figures 137-145 below. Data plots show quarterly values for each block and depth. The initial data plot with all four quarters is included to provide an overall view of results over time. Because of the number of data points, however, the resolution of individual application rate by tree density combinations was compromised. Consequently, the bar charts are further separated into two charts, each of which includes two quarters. Within these charts, quarter, block and position designations are noted. Because of the preponderance of low-level results, all figures use a logarithmic scale to present the data.

Recall that five suction lysimeters were installed within each of the 30 subplots. Three capture vertical flow and were positioned 15, 30, and 60 cm underneath the bottom of the biosolids rows. Two capture vertical flow and were positioned 15 and 30cm lateral from the edge of a biosolids row at a depth equal to the bottom of the biosolids row (Figure 6). In the bar charts and tables that follow, these positions are indicated as PV15, PV30, PV60, PL15, and PL30, respectively.

As a reminder, the time periods associated with each of the suction lysimeter quarterly designations include:

Q4 = November 2003 – February 2004

Q5 = March 2004 – May 2004

Q6 = June 2004 – August 2004 (recall that samples were not collected in July)

Q7 = October 2004 – December 2004 (recall that samples were not collected in November).







Figure 139. Nitrate quarterly average concentrations for low-level application rate in SL samples: Q6&7.







Figure 142. Nitrate quarterly average concentrations for mid-level application rate in SL samples: Q6&7.



Figure 143. Nitrate quarterly average concentrations for high-level application rate in suction lysimeter (SL) samples: all quarters.



Figure 145. Nitrate quarterly average concentrations for high-level application rate in SL samples: Q6&7.

As with nitrite results, nitrate values close to and greater than 1mg/L will be more closely examined. The small subset of quarterly results in this category are delineated in Table 24 and sorted by application rate, tree density and position. A discussion of the results follows.

Subplot ID	Block	Application Rate (kgN/ha)	Tree Density (trees/ha)	Position	quarter	Time period	# monthly results averaged	NO3-N Mean
SL-4B-2	2	0	0	lateral-15cm	5	March-May 2004	3	1.36
SL-4C-3	1	0	0	lateral-15cm	4	Nov. 2003-Feb. 2004	1	0.94
SL-4C-2	1	0	0	vertical-15cm	6	June-Aug. 2004	2	1.05
SL-1F-1	1	19,650	0	lateral-15cm	6	June-Aug. 2004	2	6.24
SL-1F-1	1	19,650	0	lateral-15cm	7	OctDec. 2004	2	5.29
SL-1F-2	1	19,650	0	lateral-30cm	5	March-May 2004	2	3.63
SL-1F-2	1	19,650	0	lateral-30cm	6	June-Aug. 2004	2	1.71
SL-1F-2	1	19,650	0	lateral-30cm	7	OctDec. 2004	2	2.39
SL-1F-4	1	19,650	0	vertical-60cm	7	OctDec. 2004	2	1.74

Table 24. Nitrate suction lysimeter results close to and greater than 1 mg/L.

Two control subplots produced quarterly results greater than 1 mg/L (4B and 4C). Control subplot 4B exhibited an elevated level in quarter 5. Elevated levels for subplot 4B were also seen in the pan lysimeter, though these occurred in quarters 1 and 2. Because suction lysimeters were not yet installed when quarters 1-3 samples were collected for the pans, the trends seen in the pan from subplot 4B could not be directly compared to the SL samples. Another control, subplot 4C, showed elevated levels in quarters 4 and 6. In both subplots, concentrations reverted to lower levels (non-detects – 0.10 mg/L) in other quarters. Also note that, although the position designations associated with the controls do correlate with specific depths below the surface, the lateral and vertical designations are not tied to a particular biosolids row, given that the controls do not contain any biosolids rows from this experiment. The remaining higher nitrate values were associated with a single subplot from the 19,650 kgN/ha-0 trees/ha treatment. Several of the suction lysimeters in this subplot established higher values over quarters 6 and 7. Although pan results did produce higher values for this subplot in quarters 6 and 7 compared to other quarters, values were 0.12 and 0.35 mg/L, respectively, well under 1 mg/L. Recall that the suction lysimeter samples represent soil water subject to both gravimetric and matric forces, and not just the gravimetric forces that primarily govern flow to the pan lysimeters. Consequently, the soil water collected from the suction lysimeters is in contact with soil surfaces for longer time periods, potentially facilitating more chemical and biological reactions.

The laterally positioned lysimeters produced most of these values. It is logical that any mineralization would initially take place from the outermost reaches of the biosolids rows. It is here that tree root systems will first establish themselves and where microbes are likely to find a more hospitable environment compared to conditions within the highly limed, salt-laden, high-pH biosolids. The higher values for SL-1F-1 is consistent with the nitrite value of 1.23 mg/L previously noted for quarter 7, further indicating that some nitrification (albeit likely a small amount) has occurred.

Rainfall Flushing Effect

As with the high concentration ammonium and nitrite samples, the 10 monthly nitrate values greater than 1 m/L were also evaluated to determine if they reflect a flushing effect from storm events. The protocol used was described in the ammonium results section. Of the ten results identified, which were from subplots 1F, 4B, and 4C, only two (i.e., 20%) were linked to storm events. It therefore does not appear that a flushing effect was responsible for the higher nitrate results.

Statistical Analysis

Statistical analyses evaluated the same interactions as those previously described for ammonium and nitrite and showed no significant differences (α = within and between any of the application rate/tree density combinations, including the controls. An overall difference between quarters was determined. Quarter 4 was significantly higher than quarters 5-7, and quarters 5 and 7 were significantly higher than quarter 6. The overall trend for quarter 4 being greater than other quarters is counter to results for subplot 1F shown in Table 24. Such a general effect, however, collectively considers values from all application rates, tree densities and positions across each quarter. Consequently, higher values from a select subplot (such as those from IF) would be overshadowed by hundreds of other values from the other subplots. A scatter plot of all results by quarters 4-7 is presented in Figure 146 below to graphically depict these statistical results. Results are separated by application rate over each quarter designation to provide for a better comparison across controls and the other three application rates.

Those previously mentioned results greater than 1 mg/L stand out against the other clusters of results, all of which are similar in value. Recall that these results were from either two replicates of the control (subplots 4B and 4C) or one replicate from the 19,650 kgN/ha-0 trees/ha treatment (subplot 1F). As stated above, statistical results do not show these treatments to be significantly different from the others. Data collected in the future, however, may provide further insight regarding whether or not these higher levels continue.



Figure 146. Nitrate quarterly average suction lysimeter results: general differences over time (quarter).

As with the nitrite data, although data do show statistically significant differences (α for time, the overall implications are minimal, given the low nitrate concentrations in most samples. The controls were designed to show typical background levels in soil that was not subjected to a recent round of biosolids application. Control results included some of the relatively higher values, demonstrating that the treatment subplots were similar to or lower than the controls.

To put these values in perspective, we can evaluate background nitrogen concentration levels previously determined at the ERCO tree farm site. For nitrate, a value of 7 mg/kg was reported (Pepperman, 1995). Assuming an average bulk density

for subsoil samples ranging between 1.6-1.9 g/cm³, each kg (1000g) of soil would provide: $1000g/(1.6g/cm^3) = 625 \text{ cm}^3$ to $1000g/(1.9g/cm^3) = 526 \text{ cm}^3$ of soil volume.

Given this range in soil volume of 526-625 cm³, and assuming a volumetric water content between 25-50%, we can estimate the volume of water present in this given volume of soil. For these volumetric water contents:

$$(0.25 \text{ cm}^3 \text{ water} / 1 \text{ cm}^3 \text{ soil}) * 526 \text{ cm}^3 \text{ soil} = 131 \text{ cm}^3 \text{ water}$$

 $(0.25 \text{ cm}^3 \text{ water} / 1 \text{ cm}^3 \text{ soil}) * 625 \text{ cm}^3 \text{ soil} = 156 \text{ cm}^3 \text{ water}$
 $(0.50 \text{ cm}^3 \text{ water} / 1 \text{ cm}^3 \text{ soil}) * 526 \text{ cm}^3 \text{ soil} = 263 \text{ cm}^3 \text{ water}$
 $(0.50 \text{ cm}^3 \text{ water} / 1 \text{ cm}^3 \text{ soil}) * 625 \text{ cm}^3 \text{ soil} = 312 \text{ cm}^3 \text{ water}$

Because nitrate is a highly water-soluble anion that is not attracted to soil particles, all nitrate measured in the soil sample will likely be in solution. Consequently, the 7mg/kg of nitrate in the soil is equal to 7 mg of nitrate in 131-312 mL of soil water.

7 mg/131mL = 0.05 mg/mL * 1000mL/L = 53 mg/L

7 mg/312 mL = 0.02 mg/mL * 1000 mL/L = 22 mg/L

This range in values of 22 - 53 mg/L for background levels of nitrate is well above the values seen in the samples collected during this experiment. For the time period covered in this experiment, it is evident that nitrate concentrations from the leachate in close proximity to the biosolids rows are not rising above background levels. This trend is independent of soil type, tree density, and biosolids application rate.

Conclusions

Pan lysimeter samples contained low-levels of nitrate, with limited exceptions in both controls and other treatments. One replicate from the 58,900 kgN/ha-0 trees/ha treatment and two replicates from the 39,300 kgN/ha-1076 trees/ha treatment produced results near or slightly above 1 mg/L over multiple quarters. This may indicate that a limited amount of nitrification was occurring. Statistical analyses showed differences in time (quarters) for certain application rate-tree density combinations. In general, no consistent trends were demonstrated from these differences, with the exception of the 58,900 kgN/ha-0 trees/ha treatment. Within this treatment, the latter quarters (quarters 6-8) had higher values than earlier quarters; across treatments, quarters 5-8 were higher than a number of other treatments.

Suction lysimeter samples had a higher percentage of low-level results than pans. A limited subset contained results close to or greater than 1 mg/L. These included results from two control replicates and one 19,650 kgN/ha-0 trees/ha treatment, none of which provides enough consistency to indicate a specific trend. The only statistical difference observed was a set of quarterly differences when examining all treatments collectively. These differences were inconsistent, with quarter 4 being greater than quarters 5-7, but with quarter 6 being less than quarters 5 and 7. From this a trend cannot be identified.

In summary, nitrate results were consistently low across application rates, tree densities, positions, and time. These results indicate that nitrification is not occurring. Conversely, it could be argued that nitrification is occurring, but is immediately followed by denitrification, immobilization, plant uptake, or a combination thereof. Neither of these scenarios, however, could account for such consistently low values of nitrate. If

nitrification were occurring, one of the many samples collected each month would show higher levels of nitrate.

As with nitrite, the overriding conclusion is that nitrate was not detected in quantities that present an environmental or health concern. In fact, with few exceptions, nitrate is present at background levels only across application rates, tree densities, and positions around the biosolids rows.

Conclusions

This study has provided valuable insight to the subsoil nitrogen dynamics surrounding the biosolids recycling operation at the ERCO Tree Farm. By closely monitoring the breakdown products of organic nitrogen, including ammonium, nitrite, and nitrate in the soil water, a better understanding of the fate and transport of these nitrogen forms has been obtained in a wide variety of soil types.

Biosolids

With the exception of one anomalous result for ammonium and magnesium, all other results were consistent across all samples. Average dry weight concentrations for total nitrogen were 4.12% (41,200 mg/kg) and for ammonium (after removal of the one outlier) were 0.23% (2,300 mg/kg).

Hydraulic conductivity

Saturated hydraulic conductivity (K_{sat}) values ranging from $1.40 \times 10^{-7} - 1.84 \times 10^{-2}$ cm/sec reflected soil composition ranging from those with high clay content to others dominated by sand and gravel. K_{sat} in Block 1 was significantly greater than K_{sat} in Block 2 and K_{sat} in Block 2 was significantly greater than K_{sat} in Block 3. Statistically significant differences were not determined, however, with depth in the soil profile, reflecting the varied soil conditions present after mining operations.

Hydraulic conductivity values were used to estimate soil water travel times in saturated conditions for percolation of leachate from the biosolids rows to the sample collection equipment (i.e., pan and suction lysimeters). In 80% of the subplots, biosolids leachate was calculated to reach the collection equipment within 8 days. The other 20%,

however, would take a minimum of one month and maximum of 13 months to travel 30 cm.

Rain gauge data

Rain gauge data were collected to provide insight to the precipitation cycles and potential impact on water flow into the soil profile from a source other than the biosolids. 2003 had more precipitation than 2004, with particularly high rainfall in May and June. In 2003 this high rainfall delayed the planting of trees at the experimental site from early May until mid-June 2003. In both years, May through September were marked by greater precipitation than other months.

Ammonium

As previously noted in the biosolids results, ammonium was already present in the biosolids in appreciable amounts (2,300 mg/kg on a dry weight basis). Because ammonium is readily soluble in water, it is not surprising that the biosolids leachate would contain comparable amounts that could be transported in the soil profile. In fact, more than half of the pan and suction lysimeter samples contained ammonium concentrations in excess of 1000 mg/L.

For the pan lysimeters, ammonium concentrations were distributed fairly evenly across application rates, with more prevalence in the 0 trees/ha tree density compared to 716 and 1074 trees/ha. Statistical analysis showed no significant differences ($\alpha = 0.05$) for application rate (including controls), tree density, or time.

For suction lysimeters, ammonium concentrations from controls were significantly less than the other application rates. The other notable trend in the non-control treatments was a decrease in concentration with distance from the biosolids row. This

supports the observation that more ammonium is reaching the first of the vertical suction lysimeters, with attenuation as it travels deeper through the soil profile. The decrease with depth could be due to cation exchange reactions in the soil that hold the ammonium and delay movement with soil water, microbial interactions (i.e., immobilization) or, though less likely, conversion of ammonium to nitrate with subsequent immediate denitrification. Finally, an overall increase in concentration with time was indicated, though more data from later time periods will need to be evaluated in the future to better define this trend.

<u>Nitrite</u>

Nitrite, the next step of organic nitrogen breakdown, was not detected in a majority of the samples, though a couple of exceptions did occur. Pan lysimeters produced significantly higher values for the 58,900 kg N/ha – 0 trees/ha treatment, though this set of higher values was primarily associated with only one of the three replicates. All other treatments, including controls, were not significantly different from one another. Suction lysimeters produced no significant differences ($\alpha = 0.05$) between application rate, tree density (including controls) or time. Some statistical differences were determined between and within positions, with the vertical position closest to the biosolids being greater than some of the other positions, though the differences were isolated. In summary, nitrite values were generally so low across experimental conditions that they are not considered to have any adverse impact on the recycling process.

<u>Nitrate</u>

Nitrate, the final product of nitrification, and the parameter of most concern from an environmental and health perspective, was consistently not detected or found in low

concentrations. Across both pan and suction lysimeters, isolated incidences of values between 1-10 mg/L were found in a control subplot and three different non-control treatments. The only notable statistical difference in pan samples pertained to the 58,900 kg N/ha- 0 trees/ha treatment, for which the later quarters had higher values than the earlier quarters within the treatment. In addition, across treatments quarters 5-8 were higher than some other treatments.

Suction lysimeter results had an even higher percentage of lower level results (< 1 mg/L) than pans, and showed no statistical differences between application rate, tree density, or position (including controls). Some quarterly differences were detected when all results were collectively combined, though these differences did not indicate any trend. As with nitrite, the overriding conclusion is that very little nitrification is occurring, and only in very isolated instances if at all. Values do not differ between controls and other application rate-tree densities, clearly demonstrating that nitrate is not present in quantities that would adversely impact human health or the environment.

Future Work

Though the data gathering thus far on this project has been extensive, additional data and evaluation would provide more insight to the processes occurring in the soil profile. Suggestions for further data gathering and study includes:

- Evaluation of pan lysimeter and suction lysimeter sample volume records over time, to determine trends that may provide insight to saturated vs. unsaturated conditions in the soil profile.
- Additional statistical analyses of the data to elicit whether or not certain isolated data are impacting the differences noted.
- Evaluation of data from standpipe wells to determine water level and oxygen content trends in the experimental plot.
- Evaluation of the phosphorus, chloride, and sulfate data being generated on a subset of samples from this project.
- Evaluation of sample results generated subsequent to the December 2004 cutoff for this thesis.
- Obtaining and evaluating monthly biosolids analysis records from the Blue Plains
 Wastewater Treatment Plant for the time period during which the biosolids rows in this experiment were installed. These data would include parameters not determined at the University of Maryland lab.
- Evaluating groundwater data from wells that encompass the perimeter of the ERCO tree farm.
- Analysis of soil cores for cation exchange capacity.
- Analysis of soil cores for sand, clay and silt content to better define soil texture.

- Collection and analysis of biosolids in the experimental plot on a yearly or twice yearly basis to determine decomposition rates at different depths in the row.
- Analysis of biosolids and soil core samples from the experimental plots for microbial activity.
- Evaluation of nitrogen content in foliar samples from the poplar trees in the experimental plot to better understand how much of the biosolids nitrogen is being consumed by the trees.
- On a yearly basis, excavation and examination of root penetration from poplar trees into the biosolids at the experimental plot to provide insight into the 1) development of channels for oxygen transport to the biosolids and 2) the extent to which the trees have enveloped the biosolids and can take up soil water and nitrogen.
- Collecting pan lysimeter samples designed to isolate storm flow. The focus of these collection efforts would be those subplots with higher hydraulic conductivity.
- Collection of lysimeter samples earlier in the process of the experimental set up, in concert with biosolids application, to capture initial leachate from the biosolids.
- Installation of tensiometers at depths consistent with sample collection equipment to more definitively study soil moisture conditions in the soil profile.
- Installation of temperature and oxygen probes in the biosolids rows and at the lysimeter depths to better monitor temperature and oxygen conditions over time.

Appendix 1 – Pan and Suction Lysimeter Installation Details **Pan Installation Diagrams**



Figure 147. Subplot 1A: Block 1; 39,300 kg N/ha; 716 trees/ha.



Figure 148. Subplot 1B: Block 1; 39,300 kg N/ha; 1074 trees/ha



Figure 149. Subplot 1C: Block 1; 39,300 kg N/ha; 0 trees/ha.


Figure 150. Subplot 1D: Block 1, 19,650 kg N/ha; 716 trees/ha.



Figure 151. Subplot 1E: Block 1; 19,650 kg N/ha; 1074 trees/ha.

78.7cm

Deep Core

109cm



Figure 152. Subplot 1F: Block 1; 19,650 kg N/ha; 0 trees/ha.



Figure 153. Subplot 1G: Block 1; 58,900 kg N/ha; 716 trees/ha.



Figure 154. Subplot 1H: Block 1; 58,900 kg N/ha; 1074 trees/ha.



Figure 155. Subplot 1I: Block 1; 58,900 kg N/ha; 0 trees/ha.



Figure 156. Subplot 4C (Control): Block 1; 0 kg N/ha; 0 trees/ha.



Figure 157. Subplot 2A: Block 2, 58,900 kg N/ha; 716 trees/ha.



Figure 158. Subplot 2B: Block 2; 58,900 kg N/ha; 1074 trees/ha.



Figure 159. Subplot 2C: Block 2; 58,900 kg N/ha; 0 trees/ha.



Figure 160. Subplot 2D: Block 2; 39,300 kg N/ha; 716 trees/ha.



Figure 161. Subplot 2E: Block 2; 39,300 kg N/ha; 1074 trees/ha.



Figure 162. Subplot 2F: Block 2; 39,300 kg N/ha; 0 trees/ha.



Figure 163. Subplot 2G: Block 2; 19,650 kg N/ha; 716 trees/ha.



Figure 164. Subplot 2H: Block 2; 19,650 kg N/ha; 1074 trees/ha.



Figure 165. Subplot 2I: Block 2; 19,650 kg N/ha; 0 trees/ha.



Figure 166. Subplot 4B (Control): Block 2; 0 kg N/ha; 0 trees/ha.



Figure 167. Subplot 3A: Block 3; 19,650 kg N/ha; 1074 trees/ha.



Figure 168. Subplot 3B: Block 3; 19,650 kg N/ha; 716 trees/ha.



Figure 169. Subplot 3C: Block 3; 19,650 kg N/ha; 0 trees/ha.



Figure 170. Subplot 3D: Block 3; 58,900 kg N/ha; 1074 trees/ha.



Figure 171. Subplot 3E: Block 3; 58,900 kg N/ha; 716 trees/ha.



Figure 172. Subplot 3F: Block 3; 58,900 kg N/ha; 0 trees/ha.



Figure 173. Subplot 3G: Block 3; 39,300 kg N/ha; 1074 trees/ha.





Figure 175. Subplot 3I: Block 3; 39,300 kg N/ha; 0 trees/ha.



Figure 176. Subplot 4A (Control): Block 3; 0 kg N/ha;0 trees/ha.

Pan Lysimeter Installation Tables

Table 25.	Pan	lysimeter	[·] installatio	n inform	ation – a	all blocks.
		2				

Subplot	Application	Tree	Biosolids	Pan Position	Depth to	Date	Weather
ID	Rate	Density	Row Under	(Distance	Bottom of Bio.	Installed	
	(kg N/ha)	(trees/ha)	Which Pan	from S. End	Row (m)		
	_		Installed	of Plot) (m)			
1A	39,300	716	6	145.4	0.86	8/1/2002	Sunny, 26.7-29.4°C
1B	39,300	1074	6	173.1	0.94	8/6/2002	Sunny, breezy, 26.7-29.4°C
1C	39,300	0	6	186.5	0.94	8/6/2002	Sunny, breezy, 26.7-29.4°C
1D	19,650	716	19	142.3	0.69	11/8/2002	Sunny, 15.5°C
1E	19,650	1074	20	168.8	0.76	12/2/2002	Sunny, 4.4°C
1F	19,650	0	22	186.2	0.61	1/9/2003	Sunny, 1.1 – 1.7°C
1G	58,900	716	30	*144.2	0.96	1/21/2003	Partly sunny, ⁻ 1.1 – 1.7°C
1H	58,900	1074	31	*166.1	1.04	2/5/2003	Sunny, breezy, 4.4-7.2°C
1I	58,900	0	30	*184.4	1.19	1/27/2003	Sunny, breezy, ⁻ 6.7- ⁻ 3.9°C
4C	0	0	NA	*178.6	0.61	3/25/2003	Sunny, 21.1-23.9°C
2A	58,900	716	6	84.1	1.24	7/29/2002	Sunny, humid, 32.2-35°C
2B	58,900	1074	6	106.7	1.02	7/30/2002	Sunny, humid, 32.2-35°C
2C	58,900	0	6	125.6	1.07	8/1/2002	Sunny, breezy, 26.7-29.4°C
2D	39,300	716	17	79.2	0.99	10/23/2002	Sunny, breezy, 16.7°C
2E	39,300	1074	16	106.7	0.94	10/7/2002	Partly sunny, 21.1°C
2F	39,300	0	17	122.5	0.89	10/28/2002	Cloudy, showers in AM, 11.1°C
2G	19,650	716	29	*78.0	0.64	1/10/2003	Sunny, p. cloudy, breezy, 4.4°C
2H	19,650	1074	29	*106.7	0.69	1/13/2003	Sunny, 4.4°C
2I	19,650	0	30	*122.5	0.64	1/20/2003	Part cloudy, breezy, ^{-1.1-} 1.7°C
4B	0	0	NA	*122.5	0.61	3/19/2003	Overcast, breezy, 10-12.8°C
3A	19,650	1074	5	10.7	0.61	7/19/2002	Sunny, humid, 32.2-35°C
3B	19,650	716	4	32.3	0.61	7/16/2002	Sunny, humid, 32.2°C
3C	19,650	0	5	58.7	0.71	7/25/2002	Partly cloudy, breezy, 29.4°C
3D	58,900	1074	14	11.9	1.37	10/2/2002	Sunny, 31.1°C
3E	58,900	716	16	35.7	1.29	10/14/2002	Sunny, breezy, 15.5-18.3°C
3F	58,900	0	17	55.5	1.22	10/21/2002	Partly cloudy, 10-12.8°C
3G	39,300	1074	27	*6.10	0.95	12/23/2002	Sunny, 7.22°C

Subplot ID	Application Rate (kg N/ha)	Tree Density (trees/ha)	Biosolids Row Under Which Pan Installed	Pan Position (Distance from S. End of Plot) (m)	Depth to Bottom of Bio. Row (m)	Date Installed	Weather
3H	39,300	716	29	*36.6	0.79	1/7/2003	Partly sunny, breezy, ^{-1.1-1.7°C}
3I	39,300	0	29	*58.5	0.76	1/8/2003	Overcast, 1.7°C
4A	0	0	NA	*58.5	0.61	2/14/2003	Overcast, 1.7-4.4°C

*Distance from south end is with respect to the western-most north-south strip of experimental subplots, the entire strip of which is positioned 12.2m south of the eastern and middle north-south strips of experimental subplots (See Figure 3).

Subplot	Notes/Comments
1A	Sandy soil; drilling easier than in eastern subplots of Blocks 2 and 3.
1B	Soil profile had clay on top, but underneath was a sandy lens, so surface clay is not representative of a clay lens. Water slowly streamed in from old biosolids rows.
1C	Soil profile had clay on top, but underneath was a sandy lens, so surface clay is not representative of a clay lens. Installation was problematic. Unstable soil resulted in a large chunk breaking off from the ceiling of the pan installation cavity. This created an arch of dead space 7.6-10.2cm above the inserted pan. To fill the gap, two sets of pan screens were placed on top of the pan (to provide extra support and filtering capability) and sand was distributed on top of the screens to fill in the void.
1D	Very gravelly, sandy soil. Water streamed out of old biosolids rows. Drilling relatively easy except when rocks encountered.
1E	Sandier soil. Relatively easy drilling. Drill poked into old biosolids row on the south side of the pan wall. No influx of water or dark biosolids was noted. Readjusted pan installation to the south to avoid having the pan resting against the breach.
1F	Due to the instability of mine spoil/soil and trench wall, left 68.6cm distance between the installation trench and biosolids row under which pan was installed. Final distance between front end of installed pan and installation trench = 63.5cm, leaving 2-3cm of pan outside biosolids row. Very wet soil profile. Water also seeped through soil into pan installation cavity. After positioning pan in the installation cavity, some soil from the ceiling of the cavity fell onto the screen covering the pan. Sand was used to fill the resulting gap in the ceiling, Water gushed out of old biosolids rows. Pump required to prevent installation trench from filling too much and impeding installation efforts.
1G	Sandy soil. Frozen ground at top 8-15cm of soil/mine spoil. Easy drilling. Small amount of drainage from old biosolids rows.
1H	Installation trench walls unstable; a layer of overburden from wall opposite installation broke off when digging installation trench. Wall supports prevented further breakoff. Drilling of average difficulty; negligible drainage from old biosolids rows.
11	Soil consisted of gravelly backfill with < 5cm diameter rocks. Frozen ground at top 8-15cm of soil/mine spoil. Drilling proceeded quickly. Due to instability of installation trench, kept distance between installation trench and biosolids row at 76cm. With a maximum possible drilling distance of 117cm (due to length of auger), only was able to fit 41cm of the 53.3cm pan length under the biosolids row. Water gushed in from old biosolids rows, requiring pump out.
4C (control)	Soil was dark brown and contained more clay and less sand than other installations in Block 1. Water streamed in from old biosolids rows, which were wider than most. After pan was installed and soil packed back into the pan installation hole, the track loader experienced mechanical problems. While waiting for the track loader to be repaired, a layer of the installation trench wall fell away, but did not interfere with the pan installation hole. The entire installation trench was then filled. Note: A small fissure/crack was created at the surface over the biosolids row, but was filled when the track loader smoothed over the entire installation area.

Table 26. Pan installation notes – block 1.

Subplot	Notes/Comments
ID	
2A	Drilling of average difficulty. Soil was dry due to drought.
2B	Drilling of average difficulty. Soil was dry due to drought.
2C	Soil contains sand and gravel. Water seeped into the pan installation cavity from the left (north) side. Odorous water trickled
	out of the old biosolids row on the left (north). Collected a soil sample directly underneath the old biosolids row to the north
	of the pan. Laboratory analysis showed negligible amounts of nitrate (< 1 mg/L).
2D	Soil contains pebbles and is crumbly. Although clay was notably present in different locations of the soil profile, sandier
	patches of soil/overburden existed. Soil had a variable profile that was difficult to characterize. Water trickled from old
	biosolids rows.
2E	Average drilling. Negligible water from old biosolids rows.
2F	Trench walls unstable. Pan installed 5cm short of originally intended placement due to time constraints imposed by transient
	trench wall conditions. Odorous water gushed from old biosolids rows.
2G	Soil/overburden consists of grayish/white packed sand and clay mix. Similar to concrete. Drilling difficult and slow.
2H	Drilling of average difficulty. Trickle of water coming from old biosolids rows.
2I	Sandy/clay mix with pebbles. Frozen ground at top 8-15cm of soil/mine spoil. Trickle of water coming from old biosolids
	rows. Drilling of average difficulty.
4B	Sandy clay with pebbles that was difficult to drill. Soil packed like concrete. Water trickled in from old biosolids rows.

Table 27. Pan installation notes – block 2.

Subplot ID	Notes/Comments
3A	High clay content. Soil dry due to drought. Drilling slow.
3B	High clay content. Soil dry due to drought. Drilling slow.
3C	High clay content. Soil dry due to drought. Drilling slow.
3D	Drilling of average difficulty. Negligible water from old biosolids trenches.
3E	Drilling slow. Shaping of pan cavity took a long time. Water gushed from old biosolids rows.
3F	Soil/overburden fairly soft and easier to drill through than other pans in Block 3. Water seeping through soil into ceiling of
	pan installation cavity. Water also streaming out of old biosolids row on north side of pan installation.
3G	High clay content, wet and packed. Drilling very slow. Water gushed out of old biosolids rows. Pump used to remove water
	from installation trench.
3H	Sandy clay soil. Smooth drilling. Water seeped through soil into ceiling of pan installation cavity. Water trickled from old
	biosolids rows.
3I	Drilling of average difficulty. Water gushed out of old biosolids rows, and filled installation trench to a 0.3m depth within 10
	minutes. Used pump to remove water.
4A	Drilling of average difficulty. Water streamed into installation trench from old biosolids.

Table 28. Pan installation notes – block 3.

Suction Lysimeter Installation Tables

Subplot	Application	Tree	Biosolids	SL Position	Biosolids	Overburden	Water Seepage	Date
ID	Rate	Density	Row Under	in Relation	Depth	Depth (m)	Into Installation	Installed
	(kg N/ha)	(trees/ha)	Which SL	to Biosolids	(m)		Cavity?	
	_		Installed	Row (cm)*				
SL-1A-1	39,300	716	5	15 – v	0.79	0.53	No	8/18/2003
SL-1A-2	39,300	716	4	60 – v	0.81	0.48	No	8/18/2003
SL-1A-3	39,300	716	3	30 - v	0.91	0.51	No	8/18/2003
SL-1A-4	39,300	716	2	30 – west	1.0	0.53	No	8/18/2003
SL-1A-5	39,300	716	2	15 – east	1.0	0.53	No	8/18/2003
SL-1B-1	39,300	1074	7	60 – v	0.94	0.30	No	8/15/2003
SL-1B-2	39,300	1074	6	30 - v	0.76	0.56	Yes – damp	8/15/2003
SL-1B-3	39,300	1074	5	15 – west	0.71	0.53	No	8/15/2003
SL-1B-4	39,300	1074	5	30 – east	0.71	0.53	No	8/15/2003
SL-1B-5	39,300	1074	4	15 – v	0.68	0.58	Yes – damp	8/15/2003
SL-1C-1	39,300	0	9	60 –v	0.96	0.41	Yes – pooled high	8/14/2003
SL-1C-2	39,300	0	8	15 – west	0.96	0.53	Yes – pooled high	8/14/2003
SL-1C-3	39,300	0	8	30 - east	0.96	0.53	No	8/14/2003
SL-1C-4	39,300	0	4	15 –v	0.94	0.41	No	8/14/2003
SL-1C-5	39,300	0	3	30 - v	0.94	0.35	No	8/14/2003

Table 29. Suction lysimeter (SL) installation information – block 1: 39,300 kg N/ha.

* v = vertically positioned lysimeters; depth = distance below bottom of biosolids row.

East = laterally positioned lysimeter placed at the specified distance from the east edge of the biosolids row at depth equal to the depth of the biosolids row.

West = laterally positioned lysimeter placed at the specified distance from the west edge of the biosolids row at depth equal to the depth of the biosolids row.

Subplot	Application	Tree	Biosolids	SL Position	Biosolids	Overburden	Water Seepage	Date
ID	Rate	Density	Row Under	in Relation	Depth	Depth (m)	Into	Installed
	(kg N/ha)	(trees/ha)	Which SL	to Biosolids	(m)		Installation	
			Installed	Row (cm)*			Cavity?	
SL-1D-1	19,650	716	16	30 – v	0.68	0.56	No	7/30/2003
SL-1D-2	19,650	716	15	15 – v	0.48	0.15	No	7/30/2003
SL-1D-3	19,650	716	14	60 – v	0.56	0.64	No	7/30/2003
SL-1D-4	19,650	716	13	15 – west	0.56	0.53	No	7/30/2003
SL-1D-5	19,650	716	13	30 – east	0.56	0.53	No	7/30/2003
SL-1E-1	19,650	1074	16	60 – v	0.76	0.41	No	7/30/2003
SL-1E-2	19,650	1074	15	15 – v	0.46	0.46	No	7/30/2003
SL-1E-3	19,650	1074	14	30 - v	0.51	0.56	No	7/30/2003
SL-1E-4	19,650	1074	13	30 – west	0.63	0.61	No	7/30/2003
SL-1E-5	19,650	1074	13	15 – east	0.63	0.61	No	7/30/2003
SL-1F-1	19,650	0	17	15 – west	0.63	0.35	No	7/31/2003
SL-1F-2	19,650	0	17	30 – east	0.63	0.35	No	7/31/2003
SL-1F-3	19,650	0	16	15 – v	0.68	0.20	No	7/31/2003
SL-1F-4	19,650	0	15	60 - v	0.61	0.45	No	7/31/2003
SL-1F-5	19,650	0	14	30 -v	0.71	0.43	No	7/31/2003

Table 30. Suction lysimeter (SL) installation information – block 1: 19,650 kg N/ha.

* v = vertically positioned lysimeters; depth = distance below bottom of biosolids row.

East = laterally positioned lysimeter placed at the specified distance from the east edge of the biosolids row at depth equal to the depth of the biosolids row.

West = laterally positioned lysimeter placed at the specified distance from the west edge of the biosolids row at depth equal to the depth of the biosolids row.

Subplot	Application	Tree	Biosolids	SL Position	Biosolids	Overburden	Water Seepage	Date
ID	Rate	Density	Row Under	in Relation	Depth	Depth (m)	Into	Installed
	(kg N/ha)	(trees/ha)	Which SL	to Biosolids	(m)		Installation	
			Installed	Row (cm)*			Cavity?	
SL-1G-1	58,900	716	29	60 – v	1.04	0.48	No	8/26/2003
SL-1G-2	58,900	716	28	15 – v	1.17	0.48	No	8/26/2003
SL-1G-3	58,900	716	26	30 - v	1.24	0.51	No	8/26/2003
SL-1G-4	58,900	716	25	30 – west	1.19	0.43	No	8/26/2003
SL-1G-5	58,900	716	25	15 – east	1.19	0.43	No	8/26/2003
SL-1H-1	58,900	1074	27	15 – v	0.94	0.30	No	8/22/2003
SL-1H-2	58,900	1074	26	30 -v	0.91	0.33	No	8/22/2003
SL-1H-3	58,900	1074	25	60 – v	0.99	0.43	No	8/22/2003
SL-1H-4	58,900	1074	24	30 – west	0.96	0.43	No	8/22/2003
SL-1H-5	58,900	1074	24	15 – east	0.96	0.43	No	8/22/2003
SL-1I-1	58,900	0	27	15 – v	0.89	0.38	No	8/22/2003
SL-1I-2	58,900	0	27	30 -v	0.89	0.38	Yes – pooled	8/22/2003
SL-1I-3	58,900	0	26	15 – west	1.0	0.38	No	8/22/2003
SL-1I-4	58,900	0	26	60 - v	1.0	0.38	Yes – damp	8/22/2003
SL-1I-5	58,900	0	26	30 – east	1.0	0.38	No	8/22/2003

Table 31. Suction lysimeter (SL) installation information – block 1: 58,900 kg N/ha.

Subplot	Application	Tree	Biosolids	SL Position	Biosolids	Overburden	Water Seepage	Date
ID	Rate	Density	Row Under	in Relation	Depth	Depth (m)	Into	Installed
	(kg N/ha)	(trees/ha)	Which SL	to Biosolids	(m)		Installation	
			Installed	Row (cm)*			Cavity?	
SL-4C-1	0	0	NA	30 - v	0.32	0.30	No	8/25/2003
SL-4C-2	0	0	NA	15 – v	0.32	0.30	No	8/25/2003
SL-4C-3	0	0	NA	15 – west	0.32	0.30	No	8/25/2003
SL-4C-4	0	0	NA	30 - east	0.32	0.30	No	8/25/2003
SL-4C-5	0	0	NA	60 - v	0.32	0.30	No	8/25/2003

Table 32. Suction lysimeter (SL) installation information – block 1:0 kg N/ha.

* v = vertically positioned lysimeters; depth = distance below bottom of biosolids row. East = laterally positioned lysimeter placed at the specified distance from the east edge of the biosolids row at depth equal to the depth of the biosolids row. West = laterally positioned lysimeter placed at the specified distance from the west edge of the biosolids row at depth equal to the depth of the biosolids row.
| Subplot | Application | Tree | Biosolids | SL Position | Biosolids | Overburden | Water Seepage | Date |
|---------|-------------|------------|-----------|--------------|--------------|------------|---------------|-----------|
| ID | Rate | Density | Row Under | in Relation | Depth | Depth (m) | Into | Installed |
| | (kg N/ha) | (trees/ha) | Which SL | to Biosolids | (m) | | Installation | |
| | | | Installed | Row (cm)* | | | Cavity? | |
| SL-2A-1 | 58,900 | 716 | 8 | 15 – v | 1.07 | 0.58 | No | 8/24/2003 |
| SL-2A-2 | 58,900 | 716 | 5 | 60 – v | 1.17 | 0.56 | No | 8/24/2003 |
| SL-2A-3 | 58,900 | 716 | 4 | 30 – west | 0.91 | 0.56 | No | 8/24/2003 |
| SL-2A-4 | 58,900 | 716 | 4 | 15 – east | 0.91 | 0.56 | No | 8/24/2003 |
| SL-2A-5 | 58,900 | 716 | 3 | 30 - v | 0.96 | 0.58 | No | 8/24/2003 |
| SL-2B-1 | 58,900 | 1074 | 5 | 15 – v | 0.86 | 0.51 | No | 8/19/2003 |
| SL-2B-2 | 58,900 | 1074 | 4 | 30 - v | 0.91 | 0.51 | No | 8/19/2003 |
| SL-2B-3 | 58,900 | 1074 | 3 | 60 – v | 0.91 | 0.41 | No | 8/19/2003 |
| SL-2B-4 | 58,900 | 1074 | 2 | 15 – west | 0.89 | 0.43 | No | 8/19/2003 |
| SL-2B-5 | 58,900 | 1074 | 2 | 30 – east | 0.89 | 0.43 | No | 8/19/2003 |
| SL-2C-1 | 58,900 | 0 | 5 | 30 -v | 0.99 | 0.41 | No | 8/19/2003 |
| SL-2C-2 | 58,900 | 0 | 4 | 15 – v | 0.91 | 0.46 | No | 8/19/2003 |
| SL-2C-3 | 58,900 | 0 | 3 | 15 – west | 0.86 | 0.51 | No | 8/19/2003 |
| SL-2C-4 | 58,900 | 0 | 3 | 30 – east | 0.86 | 0.51 | No | 8/19/2003 |
| SL-2C-5 | 58,900 | 0 | 2 | 60 - v | 0.91 | 0.51 | No | 8/19/2003 |

Table 33. Suction lysimeter (SL) installation information – block 2: 58,900 kg N/ha.

* v = vertically positioned lysimeters; depth = distance below bottom of biosolids row.

East = laterally positioned lysimeter placed at the specified distance from the east edge of the biosolids row at depth equal to the depth of the biosolids row.

West = laterally positioned lysimeter placed at the specified distance from the west edge of the biosolids row at depth equal to the depth of the biosolids row.

Subplot	Application	Tree	Biosolids	SL Position	Biosolids	Overburden	Water Seepage	Date
ID	Rate	Density	Row Under	in Relation	Depth	Depth (m)	Into Installation	Installed
	(kg N/ha)	(trees/ha)	Which SL	to Biosolids	(m)		Cavity?	
			Installed	Row (cm)*				
SL-2D-1	39,300	716	16	15 – v	0.76	0.64	No	8/1/2003
SL-2D-2	39,300	716	15	60 – v	0.86	0.61	No	8/1/2003
SL-2D-3	39,300	716	14	15 – west	0.86	0.56	No	8/1/2003
SL-2D-4	39,300	716	14	30 – east	0.86	0.56	No	8/1/2003
SL-2D-5	39,300	716	13	30 - v	0.86	0.53	No	8/1/2003
SL-2E-1	39,300	1074	20	60 – v	0.74	0.61	Yes – pooled high	8/1/2003
SL-2E-2	39,300	1074	19	15 – v	0.71	0.61	No	8/1/2003
SL-2E-3	39,300	1074	18	30 – west	0.76	0.64	Yes – pooled	8/1/2003
SL-2E-4	39,300	1074	18	15 – east	0.76	0.64	Yes – pooled	8/1/2003
SL-2E-5	39,300	1074	17	30 - v	0.76	0.48	No	8/1/2003
SL-2F-1	39,300	0	19	30 - v	0.96	0.43	No	7/31/2003
SL-2F-2	39,300	0	19	60 – v	0.96	0.43	No	7/31/2003
SL-2F-3	39,300	0	15	15 – v	0.86	0.35	Yes – pooled	7/31/2003
SL-2F-4	39,300	0	14	15 – west	0.89	0.45	Yes – pooled	7/31/2003
SL-2F-5	39,300	0	14	30 – east	0.89	0.45	No	7/31/2003

Table 34. Suction lysimeter (SL) installation information – block 2: 39,300 kg N/ha.

* v = vertically positioned lysimeters; depth = distance below bottom of biosolids row.

East = laterally positioned lysimeter placed at the specified distance from the east edge of the biosolids row at depth equal to the depth of the biosolids row.

West = laterally positioned lysimeter placed at the specified distance from the west edge of the biosolids row at depth equal to the depth of the biosolids row.

Subplot	Application	Tree	Biosolids	SL Position	Biosolids	Overburden	Water Seepage	Date
ID	Rate	Density	Row Under	in Relation	Depth	Depth (m)	Into Installation	Installed
	(kg N/ha)	(trees/ha)	Which SL	to Biosolids	(m)		Cavity?	
			Installed	Row (cm)*				
SL-2G-1	19,650	716	27	15 – v	0.51	0.53	No	7/28/2003
SL-2G-2	19,650	716	26	60 – v	0.56	0.63	No	7/28/2003
SL-2G-3	19,650	716	25	15 – west	0.51	0.51	No	7/28/2003
SL-2G-4	19,650	716	25	30 – east	0.63	0.51	No	7/28/2003
SL-2G-5	19,650	716	24	30 - v	0.63	0.51	No	7/28/2003
SL-2H-1	19,650	1074	28	60 – v	0.51	0.43	No	7/28/2003
SL-2H-2	19,650	1074	27	30 - v	0.51	0.41	No	7/28/2003
SL-2H-3	19,650	1074	26	15 – v	0.51	0.51	No	7/28/2003
SL-2H-4	19,650	1074	25	30 – west	0.51	0.51	Yes – pooled	7/28/2003
SL-2H-5	19,650	1074	25	15 – east	0.51	0.51	Yes - pooled	7/28/2003
SL-2I-1	19,650	0	28	15 – v	0.43	0.51	No	7/29/2003
SL-2I-2	19,650	0	27	30 - v	0.51	0.51	No	7/29/2003
SL-2I-3	19,650	0	26	30 – west	0.58	0.51	No	7/29/2003
SL-2I-4	19,650	0	26	15 – east	0.58	0.51	No	7/29/2003
SL-2I-5	19,650	0	25	60 - v	0.56	0.51	No	7/29/2003

Table 35. Suction lysimeter (SL) installation information – block 2: 19,650 kg N/ha.

Subplot	Application	Tree	Biosolids	SL Position	Biosolids	Overburden	Water Seepage	Date
ID	Rate	Density	Row Under	in Relation	Depth	Depth (m)	Into Installation	Installed
	(kg N/ha)	(trees/ha)	Which SL	to Biosolids	(m)		Cavity?	
			Installed	Row (cm)*				
SL-4B-1	0	0	NA	30 – west	0.32	0.30	No	8/21/2003
SL-4B-2	0	0	NA	15 – east	0.32	0.30	No	8/21/2003
SL-4B-3	0	0	NA	30 – v	0.32	0.30	No	8/21/2003
SL-4B-4	0	0	NA	60 - v	0.32	0.30	No	8/21/2003
SL-4B-5	0	0	NA	15 – v	0.32	0.30	No	8/21/2003

Table 36. Suction lysimeter (SL) installation information – block 2: 0 kg N/ha.

* v = vertically positioned lysimeters; depth = distance below bottom of biosolids row. East = laterally positioned lysimeter placed at the specified distance from the east edge of the biosolids row at depth equal to the depth of the biosolids row. West = laterally positioned lysimeter placed at the specified distance from the west edge of the biosolids row at depth equal to the depth of the biosolids row.

Subplot	Application	Tree	Biosolids	SL Position	Biosolids	Overburden	Water Seepage	Date
ID	Rate	Density	Row Under	in Relation	Depth	Depth (m)	Into Installation	Installed
	(kg N/ha)	(trees/ha)	Which SL	to Biosolids	(m)		Cavity?	
			Installed	Row (cm)*				
SL-3A-1	19,650	1074	9	15 – west	0.63	0.51	No	7/24/2003
SL-3A-2	19,650	1074	9	15 – v	0.51	0.51	Yes – pooled	7/24/2003
SL-3A-3	19,650	1074	9	30 – east	0.56	0.76	No	7/24/2003
SL-3A-4	19,650	1074	7	30 – v	0.61	0.66	No	7/24/2003
SL-3A-5	19,650	1074	5	60 – v	0.61	0.91	No	7/24/2003
SL-3B-1	19,650	716	7	15 – west	0.53	0.61	No	7/24/2003
SL-3B-2	19,650	716	7	30 – east	0.53	0.61	No	7/24/2003
SL-3B-3	19,650	716	6	15 – v	0.41	0.43	No	7/24/2003
SL-3B-4	19,650	716	4	30 -v	0.53	0.48	No	7/24/2003
SL-3B-5	19,650	716	3	60 – v	0.61	0.61	No	7/24/2003
SL-3C-1	19,650	0	8	15 – v	0.58	0.71	No	7/25/2003
SL-3C-2	19,650	0	7	30 -v	0.43	0.53	Yes – pooled	7/25/2003
SL-3C-3	19,650	0	4	15 – west	0.53	0.63	No	7/25/2003
SL-3C-4	19,650	0	4	30 - east	0.53	0.63	No	7/25/2003
SL-3C-5	19,650	0	3	60 – v	0.46	0.76	No	7/25/2003

Table 37. Suction lysimeter (SL) installation information – block 3: 19,650 kg N/ha.

* v = vertically positioned lysimeters; depth = distance below bottom of biosolids row.

East = laterally positioned lysimeter placed at the specified distance from the east edge of the biosolids row at depth equal to the depth of the biosolids row.

West = laterally positioned lysimeter placed at the specified distance from the west edge of the biosolids row at depth equal to the depth of the biosolids row.

Subplot	Application	Tree	Biosolids	SL Position	Biosolids	Overburden	Water Seepage	Date
ID	Rate	Density	Row Under	in Relation	Depth	Depth (m)	Into Installation	Installed
	(kg N/ha)	(trees/ha)	Which SL	to Biosolids	(m)		Cavity?	
			Installed	Row (cm)*				
SL-3D-1	58,900	1074	19	30 – west	0.91	0.58	Yes - pooled	8/27/2003
SL-3D-2	58,900	1074	19	15 – east	0.91	0.58	Yes - pooled	8/27/2003
SL-3D-3	58,900	1074	18	15 – v	0.99	0.66	No	8/27/2003
SL-3D-4	58,900	1074	17	60 – v	1.17	0.68	No	8/27/2003
SL-3D-5	58,900	1074	16	30 - v	1.19	0.68	No	8/27/2003
SL-3E-1	58,900	716	18	60 – v	1.19	0.61	No	8/27/2003
SL-3E-2	58,900	716	17	15 – v	1.19	0.66	No	8/27/2003
SL-3E-3	58,900	716	15	30 – west	1.24	0.68	Yes - pooled	8/27/2003
SL-3E-4	58,900	716	15	15 – east	1.24	0.68	No	8/27/2003
SL-3E-5	58,900	716	14	30 - v	1.27	0.68	Yes - pooled	8/27/2003
SL-3F-1	58,900	0	20	15 – v	0.94	0.48	No	8/26/3003
SL-3F-2	58,900	0	19	60 – v	0.94	0.51	No	8/26/3003
SL-3F-3	58,900	0	16	30 - v	1.04	0.63	No	8/26/3003
SL-3F-4	58,900	0	15	30 – west	0.89	0.68	No	8/26/3003
SL-3F-5	58,900	0	15	15 - east	0.89	0.68	No	8/26/3003

Table 38. Suction lysimeter (SL) installation information – block 3: 58,900 kg N/ha.

* v = vertically positioned lysimeters; depth = distance below bottom of biosolids row.

East = laterally positioned lysimeter placed at the specified distance from the east edge of the biosolids row at depth equal to the depth of the biosolids row.

West = laterally positioned lysimeter placed at the specified distance from the west edge of the biosolids row at depth equal to the depth of the biosolids row.

Subplot	Application	Tree	Biosolids	SL Position	Biosolids	Overburden	Water Seepage	Date
ID	Rate	Density	Row Under	in Relation	Depth	Depth (m)	Into Installation	Installed
	(kg N/ha)	(trees/ha)	Which SL	to Biosolids	(m)		Cavity?	
			Installed	Row (cm)*				
SL-3G-1	39,300	1074	31	30 – v	0.94	0.45	No	8/4/2003
SL-3G-2	39,300	1074	31	15 – v	0.94	0.45	No	8/4/2003
SL-3G-3	39,300	1074	30	30 – west	0.84	0.61	No	8/4/2003
SL-3G-4	39,300	1074	30	15 – east	0.84	0.61	No	8/4/2003
SL-3G-5	39,300	1074	29	60 – v	0.89	0.45	No	8/4/2003
SL-3H-1	39,300	716	28	15 – v	0.76	0.68	No	8/5/2003
SL-3H-2	39,300	716	27	30 - v	0.76	0.61	No	8/5/2003
SL-3H-3	39,300	716	26	60 – v	0.76	0.61	No	8/5/2003
SL-3H-4	39,300	716	25	30 – west	0.81	0.61	No	8/5/2003
SL-3H-5	39,300	716	25	15 – east	0.81	0.61	No	8/5/2003
SL-3I-1	39,300	0	31	15 – v	0.76	0.38	No	8/5/2003
SL-3I-2	39,300	0	30	60 – v	0.68	0.48	No	8/5/2003
SL-3I-3	39,300	0	27	30 – west	0.71	0.56	No	8/5/2003
SL-3I-4	39,300	0	27	15 – east	0.71	0.56	No	8/5/2003
SL-3I-5	39,300	0	26	30 – v	0.71	0.56	No	8/5/2003

Table 39. Suction lysimeter (SL) installation information – block 3: 39,300 kg N/ha.

Subplot	Application	Tree	Biosolids	SL Position	Biosolids	Overburden	Water Seepage	Date
ID	Rate	Density	Row Under	in Relation	Depth	Depth (m)	Into Installation	Installed
	(kg N/ha)	(trees/ha)	Which SL	to Biosolids	(m)		Cavity?	
			Installed	Row (cm)*				
SL-4A-1	0	0	NA	30 – v	0.32	0.30	No	8/18/2003
SL-4A-2	0	0	NA	60 – v	0.32	0.30	No	8/18/2003
SL-4A-3	0	0	NA	30 – west	0.32	0.30	No	8/18/2003
SL-4A-4	0	0	NA	15 – east	0.32	0.30	No	8/18/2003
SL-4A-5	0	0	NA	15 – v	0.32	0.30	No	8/18/2003

Table 40. Suction lysimeter (SL) installation information - block 3: 0 kg N/ha

* v = vertically positioned lysimeters; depth = distance below bottom of biosolids row. East = laterally positioned lysimeter placed at the specified distance from the east edge of the biosolids row at depth equal to the depth of the biosolids row. West = laterally positioned lysimeter placed at the specified distance from the west edge of the biosolids row at depth equal to the depth of the biosolids row.

Appendix 2 – Supplemental Results

Biosolids Supplemental Results

Sample ID =											
Date of		NH ₄ -N	P_2O_5	K_2O				Mn	Zn	Cu	Moisture
Collection	N (%)	(%)	(%)	(%)	Ca (%)	Mg (%)	S (%)	(ppm)	(ppm)	(ppm)	(%)
3/7/2002	1.09	0.06	0.80	0.09	3.12	0.06	0.05	29.30	89.00	52.60	74.70
3/18/2002	1.17	0.12	0.69	0.09	2.81	0.05	0.18	30.40	78.70	49.00	74.90
3/15/2002	1.15	0.06	0.80	0.07	2.70	0.05	0.17	29.60	77.00	55.80	77.20
3/13/2002	0.96	0.03	0.75	0.07	3.19	0.07	0.18	44.20	91.10	69.30	65.50
6/25/2002	1.23	0.08	0.82	0.08	3.35	0.06	0.20	64.10	97.50	58.60	69.60
6/26/2002	1.07	0.05	0.75	0.07	2.59	0.05	0.16	56.40	78.70	50.10	74.70
6/28/2002	1.54	0.09	1.02	0.09	2.80	0.06	0.24	71.60	143.30	77.50	67.40
7/26/2002	1.11	0.05	0.77	0.08	2.24	0.05	0.21	38.50	92.90	58.30	74.00
7/29/2002	1.16	0.06	0.78	0.07	2.45	0.05	0.21	28.80	107.60	62.10	73.60
7/30/2002	1.19	0.06	0.76	0.06	2.31	0.05	0.20	39.80	134.90	55.70	73.50
8/23/2002	1.18	0.04	0.82	0.09	2.19	0.06	0.31	48.90	131.40	79.00	71.10
8/27/2002	1.21	0.05	0.74	0.12	3.70	0.07	0.34	42.20	138.50	80.00	68.20
8/28/2002	1.44	0.09	1.07	0.10	3.30	0.06	0.30	74.50	192.00	76.40	68.70
9/27/2002	1.10	0.06	0.85	0.09	2.79	0.07	0.23	79.40	96.40	56.00	73.10
9/30/2002	1.05	0.08	0.83	0.11	2.84	0.08	0.21	72.90	89.40	63.60	74.20
9/30/2002	1.17	0.08	0.91	0.14	4.63	0.10	0.31	81.20	171.50	86.30	63.30
10/25/2002	1.10	0.05	0.91	0.13	2.66	0.07	0.18	70.10	170.50	59.40	75.80
10/28/2002	1.32	0.08	1.04	0.13	4.90	0.08	0.22	71.20	129.90	71.20	68.80
10/28/2002	1.36	0.11	1.08	0.14	6.49	0.08	0.24	77.20	187.30	72.80	66.30
11/27/2002	1.19	0.07	0.89	0.11	2.98	0.07	0.17	60.90	100.80	57.60	72.30
11/27/2002	1.26	0.07	0.87	0.13	4.87	0.91	0.18	64.10	153.50	51.70	68.10
11/27/2002	1.17	0.05	0.78	0.12	2.34	0.06	0.15	46.00	80.60	49.30	74.60
12/23/2002	0.92	0.04	0.66	0.11	2.24	0.05	0.12	39.25	70.69	40.36	77.70
12/23/2002	0.99	0.04	0.70	0.11	2.59	0.06	0.13	43.29	79.33	48.67	76.60
12/23/2002	1.14	0.06	0.87	0.13	6.27	0.08	0.18	48.76	87.46	47.82	68.54
1/20/2003	1.22	0.06	0.97	0.15	6.09	0.09	0.20	46.62	91.60	49.57	67.17
1/21/2003	1.11	0.07	0.83	0.18	3.57	0.06	0.16	42.44	85.67	46.66	73.64
1/24/2003	1.02	0.05	0.90	0.23	3.75	0.08	0.18	45.00	93.05	52.74	72.39
2/25/2003	1.23	0.05	0.87	0.20	3.68	0.08	0.16	40.44	99.51	61.92	68.41
3/10/2003	1.11	0.06	0.82	0.15	3.74	0.07	0.15	35.13	94.76	54.32	70.48
3/4/2003	1.08	0.05	0.77	0.13	3.61	0.07	0.13	30.88	99.44	54.11	71.67
3/25/2003	1.10	0.07	0.81	0.13	5.01	0.08	0.16	41.93	95.47	54.96	71.67
3/25/2003	1.18	0.06	0.92	0.21	5.78	0.11	0.18	55.56	107.53	63.92	70.13
3/26/2003	1.07	0.35	0.93	0.11	2.14	0.06	0.13	41.58	229.51	56.22	76.77
4/25/2003	1.19	0.07	0.83	0.10	2.12	0.06	0.13	32.98	88.39	51.90	72.97
4/30/2003	1.09	0.09	0.64	0.07	2.26	0.05	0.13	25.32	75.18	44.58	73.62
4/30/2003	1.06	0.08	0.74	0.07	2.41	0.05	0.13	28.61	78.49	45.41	73.75
Mean	1.15	0.07	0.84	0.12	3.42	0.09	0.19	49.16	111.04	58.53	71.76
Standard											
Deviation	0.12	0.05	0.11	0.04	1.25	0.14	0.06	16.70	38.40	11.19	3.55
Coefficient of											
Variation	10.57	69.66	12.78	35.49	36.67	155.85	31.73	33.96	34.58	19.11	4.94

Table 41. Biosolids analysis results (wet weight basis).

Sample ID =												
Date of	Moisture	Solids (%)		NH ₄ -N	P_2O_5	K ₂ O				Mn	Zn	Cu
Collection	(%)	= 100-%M	N (%)	(%)	(%)	(%)	Ca (%)	Mg (%)	S (%)	(ppm)	(ppm)	(ppm)
3/7/2002	74.70	25.30	4.31	0.24	3.16	0.36	12.33	0.24	0.20	115.81	351.78	207.91
3/18/2002	74.90	25.10	4.66	0.48	2.75	0.36	11.20	0.20	0.72	121.12	313.55	195.22
3/15/2002	77.20	22.80	5.04	0.26	3.51	0.31	11.84	0.22	0.75	129.82	337.72	244.74
3/13/2002	65.50	34.50	2.78	0.09	2.17	0.20	9.25	0.20	0.52	128.12	264.06	200.87
6/25/2002	69.60	30.40	4.05	0.26	2.70	0.26	11.02	0.20	0.66	210.86	320.72	192.76
6/26/2002	74.70	25.30	4.23	0.20	2.96	0.28	10.24	0.20	0.63	222.92	311.07	198.02
6/28/2002	67.40	32.60	4.72	0.28	3.13	0.28	8.59	0.18	0.74	219.63	439.57	237.73
7/26/2002	74.00	26.00	4.27	0.19	2.96	0.31	8.62	0.19	0.81	148.08	357.31	224.23
7/29/2002	73.60	26.40	4.39	0.23	2.95	0.27	9.28	0.19	0.80	109.09	407.58	235.23
7/30/2002	73.50	26.50	4.49	0.23	2.87	0.23	8.72	0.19	0.75	150.19	509.06	210.19
8/23/2002	71.10	28.90	4.08	0.14	2.84	0.31	7.58	0.21	1.07	169.20	454.67	273.36
8/27/2002	68.20	31.80	3.81	0.16	2.33	0.38	11.64	0.22	1.07	132.70	435.53	251.57
8/28/2002	68.70	31.30	4.60	0.29	3.42	0.32	10.54	0.19	0.96	238.02	613.42	244.09
9/27/2002	73.10	26.90	4.09	0.22	3.16	0.33	10.37	0.26	0.86	295.17	358.36	208.18
9/30/2002	74.20	25.80	4.07	0.31	3.22	0.43	11.01	0.31	0.81	282.56	346.51	246.51
9/30/2002	63.30	36.70	3.19	0.22	2.48	0.38	12.62	0.27	0.84	221.25	467.30	235.15
10/25/2002	75.80	24.20	4.55	0.21	3.76	0.54	10.99	0.29	0.74	289.67	704.55	245.45
10/28/2002	68.80	31.20	4.23	0.26	3.33	0.42	15.71	0.26	0.71	228.21	416.35	228.21
10/28/2002	66.30	33.70	4.04	0.33	3.20	0.42	19.26	0.24	0.71	229.08	555.79	216.02
11/27/2002	72.30	27.70	4.30	0.25	3.21	0.40	10.76	0.25	0.61	219.86	363.90	207.94
11/27/2002	68.10	31.90	3.95	0.22	2.73	0.41	15.27	2.85	0.56	200.94	481.19	162.07
11/27/2002	74.60	25.40	4.61	0.20	3.07	0.47	9.21	0.24	0.59	181.10	317.32	194.09
12/23/2002	77.70	22.30	4.13	0.18	2.96	0.49	10.04	0.22	0.54	176.01	317.00	180.99
12/23/2002	76.60	23.40	4.23	0.17	2.99	0.47	11.07	0.26	0.56	185.00	339.02	207.99
12/23/2002	68.54	31.46	3.62	0.19	2.77	0.41	19.93	0.25	0.57	154.99	278.00	152.00
1/20/2003	67.17	32.83	3.72	0.18	2.95	0.46	18.55	0.27	0.61	142.00	279.01	150.99
1/21/2003	73.64	26.36	4.21	0.27	3.15	0.68	13.54	0.23	0.61	161.00	325.00	177.01
1/24/2003	72.39	27.61	3.69	0.18	3.26	0.83	13.58	0.29	0.65	162.98	337.02	191.02
2/25/2003	68.41	31.59	3.89	0.16	2.75	0.63	11.65	0.25	0.51	128.02	315.00	196.01
3/10/2003	70.48	29.52	3.76	0.20	2.78	0.51	12.67	0.24	0.51	119.00	321.00	184.01
3/4/2003	71.67	28.33	3.81	0.18	2.72	0.46	12.74	0.25	0.46	109.00	351.01	191.00
3/25/2003	71.67	28.33	3.88	0.25	2.86	0.46	17.68	0.28	0.56	148.01	336.99	194.00
3/25/2003	70.13	29.87	3.95	0.20	3.08	0.70	19.35	0.37	0.60	186.01	359.99	213.99
3/26/2003	76.77	23.23	4.61	1.51	4.00	0.47	9.21	0.26	0.56	178.99	987.99	242.01
4/25/2003	72.97	27.03	4.40	0.26	3.07	0.37	7.84	0.22	0.48	122.01	327.01	192.01
4/30/2003	73.62	26.38	4.13	0.34	2.43	0.27	8.57	0.19	0.49	95.98	284.99	168.99
4/30/2003	73.75	26.25	4.04	0.30	2.82	0.27	9.18	0.19	0.50	108.99	299.01	172.99
Mean	71.76	28.24	4.12	0.27	2.99	0.41	11.94	0.31	0.66	173.55	394.20	207.42
Standard												
Deviation	3.55	3.55	0.43	0.22	0.36	0.14	3.39	0.43	0.17	53.57	139.15	29.54
Coefficient of												
Variation	4.94	12.56	10.37	83.19	12.16	33.89	28.42	140.58	26.42	30.87	35.30	14.24

Table 42. Biosolids analysis results (dry weight basis).

Sample ID	NH4-N (%)	Sample ID	Mg (%)
3/7/2002	0.24	3/7/2002	0.24
3/18/2002	0.48	3/18/2002	0.20
3/15/2002	0.26	3/15/2002	0.22
3/13/2002	0.09	3/13/2002	0.20
6/25/2002	0.26	6/25/2002	0.20
6/26/2002	0.20	6/26/2002	0.20
6/28/2002	0.28	6/28/2002	0.18
7/26/2002	0.19	7/26/2002	0.19
7/29/2002	0.23	7/29/2002	0.19
7/30/2002	0.23	7/30/2002	0.19
8/23/2002	0.14	8/23/2002	0.21
8/27/2002	0.16	8/27/2002	0.22
8/28/2002	0.29	8/28/2002	0.19
9/27/2002	0.22	9/27/2002	0.26
9/30/2002	0.31	9/30/2002	0.31
9/30/2002	0.22	9/30/2002	0.27
10/25/2002	0.21	10/25/2002	0.29
10/28/2002	0.26	10/28/2002	0.26
10/28/2002	0.33	10/28/2002	0.24
11/27/2002	0.25	11/27/2002	0.25
11/27/2002	0.22	11/27/2002	0.24
11/27/2002	0.20	12/23/2002	0.22
12/23/2002	0.18	12/23/2002	0.26
12/23/2002	0.17	12/23/2002	0.25
12/23/2002	0.19	1/20/2003	0.27
1/20/2003	0.18	1/21/2003	0.23
1/21/2003	0.27	1/24/2003	0.29
1/24/2003	0.18	2/25/2003	0.25
2/25/2003	0.16	3/10/2003	0.24
3/10/2003	0.20	3/4/2003	0.25
3/4/2003	0.18	3/25/2003	0.28
3/25/2003	0.25	3/25/2003	0.37
3/25/2003	0.20	3/26/2003	0.26
4/25/2003	0.26	4/25/2003	0.22
4/30/2003	0.34	4/30/2003	0.19
4/30/2003	0.30	4/30/2003	0.19
Mean	0.23		0.24
Standard Deviation	0.07		0.04
Coefficient of			
Variation	29.94		17.35

Table 43. Biosolids results: revised after removal of outliers (dry weight basis).

Hydraulic Conductivity Supplemental Results

 Table 44. Hydraulic conductivity by block

Block 1			Block 2			Block 3			
	Depth from			Depth from			Depth from		
	Top of	Hydraulic		Top of	Hydraulic		Top of	Hydraulic	
	Installation	Conductivity		Installation	Conductivity		Installation	Conductivity	
Subplot ID	Trench (cm)	(cm/sec)	Subplot ID	Trench (cm)	(cm/sec)	Subplot ID	Trench (cm)	(cm/sec)	
1A-shallow	64	5.27E-03	2A-shallow	94	4.66E-04	3A-shallow	30	4.05E-07	
1A-middle	94	1.64E-03	2A-middle	124	7.73E-04	3A-middle	61	1.40E-07	
1A-deep	124	3.05E-03	2A-deep	155	7.47E-05	3A-deep	91	1.30E-05	
1B-shallow	64	1.71E-03							
1B-middle	94	7.78E-05	2B-middle	102	4.73E-04				
1B-deep	124	9.53E-06	2B-deep	132	9.17E-05	3B-deep	94	1.02E-05	
1C-shallow	64	8.33E-04	2C-shallow	76	2.88E-05	3C-shallow	41	2.68E-04	
1C-middle	94	1.67E-03	2C-middle	107	3.60E-04	3C-middle	71	2.84E-05	
1C-deep	124	2.92E-03	2C-deep	137	2.23E-03	3C-deep	102	9.44E-05	
1D-shallow	46	1.85E-02	2D-shallow	74	1.01E-04	3D-shallow	107	1.48E-06	
1D-middle	76	1.71E-02	2D-middle	104	2.18E-05	3D-middle	137	8.91E-06	
1D-deep	107	1.81E-02	2D-deep	135	4.36E-05	3D-deep	168	3.13E-06	
1E-shallow	48	4.71E-04	2E-shallow	64	1.27E-04	3E-shallow	107	3.81E-06	
1E-middle	79	6.03E-03	2E-middle	94	2.12E-05	3E-middle	137	7.44E-07	
1E-deep	109	1.57E-04	2E-deep	124	9.05E-07	3E-deep	168	9.99E-05	
1F-shallow	38	2.11E-03	2F-shallow	69	1.32E-04	3F-shallow	102	1.41E-04	
1F-middle	69	8.56E-05	2F-middle	99	5.77E-04	3F-middle	132	3.74E-06	
1F-deep	99	7.64E-05	2F-deep	130	5.30E-05	3F-deep	163	5.48E-06	
1G-shallow	74	2.77E-04	2G-shallow	41	4.77E-05	3G-shallow	72	3.24E-06	
1G-middle	104	4.10E-04	2G-middle	71	4.67E-05	3G-middle	103	1.77E-05	
1G-deep	135	2.52E-03	2G-deep	102	4.59E-05	3G-deep	133	1.67E-04	
1H-shallow	81	2.40E-04	2H-shallow	46	9.93E-05	3H-shallow	56	3.80E-04	
1H-middle	112	4.75E-04	2H-middle	76	9.16E-06	3H-middle	86	1.38E-05	
1H-deep	142	3.10E-04	2H-deep	107	4.74E-05	3H-deep	117	5.76E-04	
1I-shallow	97	1.26E-04	2I-shallow	41	1.40E-04	3I-shallow	53	4.04E-04	
1I-middle	127	4.00E-04	2I-middle	71	8.74E-04	3I-middle	84	2.91E-05	
1I-deep	157	3.99E-03	2I-deep	102	4.67E-04	3I-deep	114	1.52E-04	
4C-shallow	41	1.02E-06	4B-shallow	41	2.28E-04	4A-shallow	41	1.79E-05	
4C-middle	71	2.03E-03	4B-middle	71	4.89E-04	4A-middle	71	2.07E-04	
4C-deep	102	1.92E-03	4B-deep	102	1.31E-04	4A-deep	102	1.32E-04	





Jun-Aug-03

Apr-May-03



Jan/Feb-04

04

Quarter-Block ID

Sept-Nov-03



Figure 178. Total nitrogen concentrations for mid-level app. rate in pan lysimeters.



Figure 179. Total nitrogen concentrations for high-level app. rate in pan lysimeters.

Application			
Rate	Total N Trends		
19,650 kgN/ha	0 trees/ha had the most numerous values greater than 100 mg/L (19). Value		
	ranged from 158-496 mg/L and were distributed amongst all blocks and		
	guarters.		
	716 trees/ha had 18 values $> 100 \text{ mg/L}$, with the consistently highest values		
	of the tree densities, as follows:		
	• Block 2 had the highest values ranging from 750-2800 mg/L across all		
	quarters.		
	• Blocks 1 and 3 had lower values between 104-500 mg/L in quarters 3-8		
	1074 trees/ha had the lowest number of values greater than $100 mg/L$ (8).		
	These were associated with blocks 1 and 3 and ranged from $105-184 \text{ mg/L}$		
	across most quarters		
39 300 kgN/ha	0 trees/ha had the most values greater than 100 mg/L (18) and higher values		
57,500 Kgi (/ild	with a range between $204 - 2275 \text{ mg/L}$ Most values were associated with		
	blocks 1 and 3 across all quarters, though several values were from block 2		
	716 trees/ha had the next highest number of values (16), ranging between 147		
	-580 mg/I All of these values were associated with blocks 2 and 3 Block		
	1 had many values less than 10 mg/I		
	1074 trees/ha had 14 values ranging from $104-953 \text{ mg/L}$ All were associated		
	<u>1074 freesha</u> had 14 values fairging from 104-555 fig/L. All were associated		
59.000 kaN/ba	with blocks 1 and 2 except one value from block 5. $0 transfit has had the most values greater than 100 mg/L (11). Seven values were$		
56,900 Kgin/lia	0.00000000000000000000000000000000000		
	from block 2 across an quarters and were an $> 1000 \text{ mg/L}$. The remaining four values were from block 1, guarters 5.8, and renged from $102 - 150$		
	Tour values were from block 1, quarters 5-8, and ranged from $105 - 150$		
	IIIg/L.		
	$\frac{716 \text{ trees/na}}{100 \text{ mg/L}}$ had only three values greater than 100 mg/L, ranging from 150-		
	190 mg/L. All were associated with block 1, quarters 6-8. All block 2 and 3		
	values were less than 15 mg/L. 1074 (see the less than 15 mg/L) is 147		
	10/4 trees/na nad the second highest number of values (8) ranging from 14/ -		
	2202 mg/L. Seven values were from block 3 and ranged between 781 - 2202		
	mg/L; one value was from block 1 and was 147 mg/L.		

Table 45. Trends for pan lysimeter total N and NH_4^+ results with values > 100 mg/L.

Application	
Rate	NH ₄ ⁺ Trends
19, 650 kgN/ha	<u>0 trees/ha</u> had the most numerous values greater than 100 mg/L (20). Values
	ranged from 102-415 mg/L and were distributed amongst all blocks and
	quarters.
	<u>716 trees/ha</u> had 18 values $> 100 \text{ mg/L}$, with the consistently highest values of the tree densities, as follows:
	• Block 2 had the highest values ranging from 867-3178 mg/L across all
	quarters.
	• Blocks 1 and 3 had lower values between 112-508 mg/L in quarters 3-8.
	1074 trees/ha had the lowest number of values greater than 100 mg/L (9).
	These were associated with blocks 1 and 3 and ranged from 107-188 mg/L
	across most quarters.
39,300 kgN/ha	0 trees/ha had the most values greater than 100 mg/L (18) and higher values,
	with a range between $214 - 1272 \text{ mg/L}$. Most values were associated with
	blocks 1 and 3 across all quarters, though two of the higher values were from
	block 2, quarters 5 and 6.
	<u>716 trees/ha</u> had the next highest number of values (15), ranging from 164-
	659 mg/L. All of these values were associated with blocks 2 and 3. Block 1
	had one value at 20 mg/L and the rest were less than 10 mg/L. 1074 there are the left 14 ms been substantian them 100 ms /L that are substantian.
	$\frac{10/4 \text{ trees/na}}{2 track the size high set or large still a range of CO1 1072. Place 1 had$
	Block 2 had the six highest values, with a range of 681-10/3. Block 1 had
50.000 L N/4	seven values from 108-241 mg/L. Block 3 had one value at 138 mg/L.
58,900 kgN/na	0 trees/na had the most values greater than 100 mg/L (11). Seven values were
	from block 2 across all quarters and were between 982-2521 mg/L. The
	remaining four values were from block 1, quarters 5-8, and ranged from 108 –
	138 mg/L.
	$\frac{710 \text{ trees/na}}{208 \text{ mg/L}}$ had only three values greater than 100 mg/L, ranging from 125-
	208 mg/L. All were associated with block 1, quarters 6-8. All block 2 and 5 values were less than 15 mg/I
	1074 trees/ha had the second highest number of values (8) seven of which
	were from block 3 and ranged between 654 - 2456 mg/L with a steady
	increase over time. The other value was from block 1, quarter 8 at a much
	lower value of 144 mg/L.



Quarter-Block-Position ID

Figure 180. Total nitrogen concentrations for low-level application rate in suction lysimeter (SL) samples – all quarters.







Figure 183. Total nitrogen concentrations for mid-level application rate in suction lysimeter (SL) samples – all quarters.

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Quarter-Block-Position ID

Figure 186. Total nitrogen concentrations for high-level application rate in suction lysimeter (SL) samples – all quarters.



Total nitrogen concentrations for high-level app. rate in SL samples: Q6&7. Figure 188.

	Tree		
Application	Density		
Rate (kgN/ha)	(trees/ha)	Total Nitrogen Trends*	Ammonium Trends*
19,650	0	<u>16 results > 1000 mg/L:</u>	<u>16 results > 1000 mg/L:</u>
		PV15, all blocks-all Q	PV15, all blocks-all Q
		PV30, B3-all Q	PV30, B3-all Q
		<u>17 results from 100-1000 mg/L:</u>	<u>18 results from 100-1000 mg/L:</u>
		PV30, B1&2-all Q	PV30, B1&2-all Q
		PV60, B1-Q4,6,7, B3-Q6&7	PV60, B1-all Q, B3-Q6&7
		several PL15 and PL30	several PL15 and PL30
		20 results < 100 mg/L	<u>19 results < 100 mg/L</u>
	716	<u>11 results > 1000 mg/L:</u>	<u>11 results > 1000 mg/L:</u>
		PV15, B1&3-all Q	PV15, B1&3-all Q
		PV30, B1-most Q	PV30, B1-most Q
		24 results from 100-1000 mg/L:	25 results from 100-1000 mg/L:
		PV15, B2-all Q	PV15, B2-all Q
		PV30, B3 – all Q	PV30, B1-Q4, B3 – all Q
		PV60, all blocks – most Q	PV60, all blocks – all Q
		PL15, B1&2-Q6&7	PL15, B1&2- Q6&7
		25 results < 100 mg/L	<u>24 results < 100 mg/L</u>
	1074	<u>10 results > 1000 mg/L:</u>	<u>10 results > 1000 mg/L:</u>
		PV15, B1-Q6&7, B2&3-all Q	PV15, B1-Q6&7, B2&3-all Q
		<u>18 results from 100-1000 mg/L:</u>	<u>18 results from 100-1000 mg/L:</u>
		PV30, B1-Q4&5	PV30, B1-Q4&5
		PL15, all blocks - most Q	PL15, all blocks - most Q
		PL 30, B2-allQ, B3-Q6	PL 30, B2-allQ, B3-Q6
		26 results < 100 mg/L	26 results < 100 mg/L

Table 46. Trends for suction lysimeter total N and NH_4^+ results with focus on values > 100 mg/L.

	Tree		
Application	Density		
Rate (kgN/ha)	(trees/ha)	Total Nitrogen Trends*	Ammonium Trends*
39,300	0	<u>13 results > 1000 mg/L</u>	13 results > 1000 mg/L
		PV15, all blocks – all Q	PV15, all blocks – all Q
		PV30, B1-Q7	PV30, B1-Q7
		28 results from 100-1000 mg/L	<u>28 results from 100-1000 mg/L</u>
		PV30, B1&2 - most Q	PV30, B1&2 - most Q
		PV60, B1&2 – all Q	PV60, B1&2 – all Q
		PL15, B1&2 – all Q	PL15, B1&2 – all Q
		PL30, B1-Q6&7, B2 – all Q	PL30, B1-Q6&7, B2 – all Q
		17 results < 100 mg/L	<u>17 results < 100 mg/L</u>
	716	10 results > 1000 mg/L	10 results > 1000 mg/L
		PV15, B1-all Q, B2-Q6&7	PV15, B1-all Q, B2-Q6&7
		PV60, B1&2 – several Q	PV60, B1&2 – several Q
		<u>25 results from 100-1000 mg/L</u>	<u>25 results from 100-1000 mg/L</u>
		PV15, B2-Q5	PV15, B2-Q5
		PV30, several results from B1&2	PV30, several results from B1&2
		PV60, several results from all blocks	PV60, several results from all blocks
		PL15, B1&2-all Q	PL15, B1&2-all Q
		PL30, B1&2-all Q	PL30, B1&2-all Q
		<u>19 results < 100 mg/L</u>	<u>19 results < 100 mg/L</u>
	1074	$\underline{7 \text{ results} > 1000 \text{ mg/L}}$	$\underline{7 \text{ results} > 1000 \text{ mg/L}}$
		PV15, B2-Q7, B3-all Q	PV15, B2-Q7, B3-all Q
		PV30, B1-Q6&7	PV30, B1-Q6&7
		<u>25 results from 100-1000 mg/L</u>	<u>25 results from 100-1000 mg/L</u>
		PV15, B1&2- most Q	PV15, B1&2- all Q
		PV30, B1&3 – most Q	PV30, B1-Q4&5, B3 – Q4, 5, 7
		PV60, B3-all Q	PV60, B3-all Q
		PL15, B1&2-Q6&7	PL15, B1&2-Q6&7
		PL30-B2&3-some Q	PL30-B2&3-Q6&7
		25 results < 100 mg/L	25 results < 100 mg/L

	Tree			
Application	Density			
Rate (kgN/ha)	(trees/ha)	Total Nitrogen Trends*	Ammonium Trends*	
58,900	0	11 results > 100 mg/L	<u>11 results > 100 mg/L</u>	
		PV15, B2-all Q, B3-Q6&7	PV15, B2-all Q, B3-Q6&7	
		PV30, B2 - Q5, 6, 7	PV30, B2 - Q5, 6, 7	
		PV60, B1-Q6&7	PV60, B1-Q6&7	
		<u>18 results from 100-1000 mg/L</u>	<u>19 results from 100-1000 mg/L</u>	
		PV15, B1-Q6, B3-Q4&5	PV15, B1-Q6, B3-Q4&5	
		PV30, B1-Q7, B2-Q4	PV30, B1-Q7, B2-Q4	
		PV60, B1-Q5, B2-all Q, B3-Q6&7	PV60, B1-Q5, B2-all Q, B3-Q5, 6, 7	
		PL15, B1-Q6&7, B2-all Q	PL15, B1-Q6&7, B2-all Q	
		30 results < 100 mg/L	<u>29 results < 100 mg/L</u>	
	716	<u>13 results > 1000 mg/L</u>	13 results > 1000 mg/L	
		PV15, B1-all Q	PV15, B1-all Q	
		PV30, B1&2-allQ	PV30, B1&2-allQ	
		PL15, B2-Q7	PL15, B2-Q7	
		<u>16 results from 100-1000 mg/L</u>	<u>17 results from 100-1000 mg/L</u>	
		PV15, B2&3- most Q	PV15, B2&3- most Q	
		PV60, B1-Q4, B2-all Q	PV60, B1-Q4, B2-all Q	
		PL15, B1-all Q	PL15, B1-all Q	
		24 results < 100 mg/L	PL30, B3-Q6	
			<u>23 results < 100 mg/L</u>	
	1074	13 results > 1000 mg/L	$\underline{12 \text{ results} > 1000 \text{ mg/L}}$	
		PV15, B1-Q7, B3-all Q	PV15, B1-Q7, B3-Q4, 6, 7	
		PV30, B2&3 – most Q	PV30, B2&3 – most Q	
		PV60, B1-Q4	PV60, B1-Q4	
		<u>22 results from 100-1000 mg/L</u>	<u>21 results from 100-1000 mg/L</u>	
		PV15, B1-Q4, 5, 6, B2-Q5, 6, 7	PV15, B1-Q4, 5, 6, B2-Q7, B3-Q5	
		PV30, B1-all Q	PV30, B1-all Q	
		PV60, B1&B3-most Q	PV60, B1&B3-most Q	
		PL15, B2-Q6&7, B3-Q4, 6, and 7	PL15, B2-Q6&7, B3-Q4, 6, and 7	
		23 results < 100 mg/L	<u>25 results < 100 mg/L</u>	
*Key to Abbreviation	*Key to Abbreviations: PV# = vertical lysimeter placed # distance (cm) below biosolids row; B = block; Q = quarter			

Nitrate pan lysimeter statistical results:

For each non-control application rate, a list of statistically significant differences in tree densities for specific quarters was determined. These are itemized below.

Application Rate: 19,650 kgN/ha

0 trees/acre, Q2 is greater than 716 trees/ha, Q5 and Q8

0 trees/ha, Q2 is greater than 1074 trees/ha, Q5

0 trees/ha, Q5 and Q8 is less than 716 trees/ha, Q2

716 trees/ha, Q5, Q6, and Q8 are less than 716 trees/ha, Q2

For this low-level application rate, differences between tree densities are spotty, and do not show a particular trend.

Application Rate: 39,300 kgN/ha

0 trees/ha, Q2 is greater than 716 trees/ha, Q8

0 trees/ha, Q4 is greater than 716 trees/ha, Q5 and Q8

0 trees/ha, Q8 is greater than 716 trees/ha, Q5, Q6 and Q8

0 trees/ha, Q3, Q5, Q6 is less than 1074trees/ha, Q8

716 trees/ha, Q3-Q8 are less than 1074 trees/ha, Q8

716 trees/ha, Q5 is less than 1074 trees/ha, Q2 and Q6

716 trees/ha, Q8 is less than 1074 trees/ha, Q2-Q6

Of the differences noted, the 716 trees/ha density has the preponderance of values less than the other densities, and 1074 trees/ha are always more than the other densities. Aside from this, no explicit trends are noted.

Application Rate: 58,900 kgN/ha

0 trees/ha, Q1, Q3 and Q4 are less than 716 trees/ha, Q1

0 trees/ha, Q5 is greater than 716 trees/ha, Q6-Q8

0 trees/ha, Q6 is greater than 716 trees/ha, Q3-Q8

0 trees/ha, Q8 is greater than 716 trees/ha, Q2-Q8

0 trees/ha, Q2 is greater than 1074 trees/ha, Q5

0 trees/ha, Q5 is greater than 1074 trees/ha, Q5-Q8

0 trees/ha, Q6 is greater than 1074 trees/ha, Q2-Q8

0 trees/ha, Q7 is greater than 1074 trees/ha, Q5, Q6, and Q8

0 trees/ha, Q8 is greater than 1074 trees/ha, Q2-Q8

716 trees/ha, Q1 is greater than 1074 trees/ha, Q2-Q8

716 trees/ha, Q2 is greater than 1074 trees/ha, Q4-Q8

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