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## University of Alberta

Forest Floor as an Amendment in Oil Sand Reclamation

by

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A thesis submitted to the Faculty of Graduate Studies and Research in partial fulfillment of the requirements for the degree of Master of Science

in

Soil Science

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#### ABSTRACT

Syncrude Canada Inc. and Suncor Energy Ltd. are two oil sand operators in the Athabasca region. Following surface mining, land reclamation entails re-establishment of functioning ecosystems using salvaged mineral soil materials and organic amendments. While traditional reclamation uses peat, benefits from using the forest floor stripped from pre-mining areas were examined. This study investigated the reclamation techniques in terms of nitrogen (N) fluxes. Specifically, net N nitrogen mineralization rates and microbial biomass were measured in an undisturbed forest and several reclaimed sites at Syncrude and Suncor. Results suggested no significant difference in net N mineralization rates between sites. However, there were differences in microbial biomass possibly related to differences in moisture content. A lab experiment manipulating soil moisture content showed a positive relationship between microbial biomass N and moisture content. No relationship with microbial biomass C was found, possibly due to a shift in microbial community structure.

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#### **CHAPTER 1. GENERAL INTRODUCTION**

#### **1.1 Introduction**

The Athabasca region, in northeastern Alberta, lies within the boreal zone, hosting both deciduous and coniferous species. Oil sand mining is one of the main industries in this region. The Athabasca oil sand deposit is of the largest reserves of hydrocarbons in the world, with almost one trillion barrels of bitumen (Kimball et al. 2000). Due to the shallowness of the deposit, extraction occurs through surface mining, which results in extensive land disturbance. Approximately 40,000 hectares of land disturbed by the oil sand operators Syncrude Canada Ltd. and Suncor Energy Inc. will need to be reclaimed back to their original use or to other productive uses (Fung and Macyk 2000).

During the oil sand mining process surface soil (up to 20 cm thickness) is stripped and stockpiled to be used for reclamation. However, changes in soil properties, such as a reduction in organic matter quality, may occur during the stripping/stockpiling process (Schwenke et al. 1999). Because disturbances may have a profound effect on soil properties, this study was conducted to examine and compare soils in a disturbed environment to those in a natural, undisturbed environment.

Although reclamation success has typically been defined by vegetation abundance and diversity, soil characteristics are also important indicators of ecosystem health (Mummey et al. 2002). Bendfeldt et al. (2001) compiled, from the literature, a listing of basic indicators of soil quality, fertility, and health such as total organic C and N, microbial biomass C and N, mineralizable C and N, pH and bulk density. Typically in boreal forests nitrogen is the most limiting nutrient for ecosystem productivity (Kimmins 1996; Little et al. 2002) and is critical for maintaining a sustainable ecosystem (Raison and Stottlemyer 1991).

The main objectives of land reclamation are to control erosion, to establish a productive, sustainable forest of native vegetation, and to establish forests usable for forestry, wildlife or recreation (Syncrude Canada Ltd. 1981). To meet these objectives organic soil treatments are used. Current reclamation processes use peat, due to its availability in large portions of the mining footprint in the Fort McMurray area, as well as its ability to increase soil water holding capacity, improve plant root penetration and retain nutrients (Lucas et al., 1965). However, peat is formed in lowland environments, and it is unknown how it will function in an upland environment. Consequently, the advantages of using the forest floor (LFH horizon) stripped from upland areas are now being investigated as an alternative to peat. LFH has been shown to provide an excellent seed bank for native plant species (Granström 1981; Qi and Scarratt 1998). Additionally, it may enhance microbiological activity and provide sufficient nutrients to kick-start successful biogeochemical cycling in the reclaimed soils.

Studies have been conducted to examine reclamation using treatments such as straw, yard waste compost, and topsoil in surface metal and coal mines (Coyne et al. 1997; Benfeldt et al. 2001; Stolt et al. 2001). However, fewer studies have examined the techniques used in oil sand reclamation (Logan 1978; Lanoue, 2003, Li et al. 2003), and none have examined the critical issue of nitrogen cycling in these treatments.

The objectives of this study were:

- 1. To compare the chemical, biological and physical characteristics of reclaimed sites to those of a neighboring undisturbed boreal forest, and
- 2. To quantify net N mineralization rates and microbial biomass of peat and LFH treatments used in oil sand reclamation.

Organic treatments examined in this thesis include: peat-mineral mix, LFH over peat mineral mix, LFH over secondary material, and LFH over "lean" (<8% bitumen) oil sand.

Soil formation and the re-establishment of functioning microbial communities and nutrient cycles in reclaimed soils involve processes acting over monthly time scales to centuries. Thus, I hypothesized that the natural forest would have higher net N mineralization rates and higher microbial biomass C and N than the juvenile reclaimed sites. Furthermore, I hypothesized that within the reclaimed sites, the sites amended with LFH, containing upland microbial communities and seed banks, would have higher mineralization rate and microbial biomass than sites reclaimed with the peat-mineral mix, containing lowland microbial communities and seeds banks.

This thesis is organized into five chapters. Following this introductory chapter (Chapter 1), Chapter 2 is a concise literature review on nitrogen mineralization, specifically net nitrogen mineralization occurring in the Boreal Plain Ecozone. It is divided into sections based on Jenny's (1941) soil forming factor equation. Chapter 3 presents the findings of a field-based and laboratorybased study comparing nitrogen (N) mineralization rates in a reclaimed LFH and peat-mineral mix over a mineral secondary/tailing sand substrate. Chapter 4 compares the soil properties of a natural forest, reclaimed LFH and peat-mineral mix overlying lean oil sand. Several soil properties were examined in relation to net N mineralization rates including microbial biomass C and N, total C and N, pH, bulk density and moisture content. Chapters 3 and 4 are treated separately for several reasons. First, the types of overburden material that was used (lean oil sands and secondary/tailing sands), and soil textures of the two areas are different (ranging from a sand to a silty loam). Secondly, the fertilization and planting practices are different between the two areas, including the type of vegetation, and the amount and duration of fertilizer application. Lastly, the source and composition of peat and LFH used in reclaiming the two sites are different. The experimental sites were established before this study began and it was not possible to quantitatively compare the two areas by performing experiments on every individual factor that differs between the two. Chapter 5 concludes the thesis, by summarizing the results of Chapters 3 and 4.

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# CHAPTER 2. LITERATURE REVIEW- NITROGEN MINERALIZATION IN BOREAL FORESTS

#### 2.1 Introduction

The boreal forest region, composed of both coniferous and deciduous tree species, covers 14.7 million km<sup>2</sup> of the earth (Brais et al. 1995). Low nitrogen availability, low temperatures, and moisture deficits are thought to be the main growth limiting factors in the boreal forest zone (Binkley and Hogberg 1997). In boreal forests, nitrogen is the nutrient that is most often limiting for plant growth because most of it is tied up in unavailable organic forms (Kimmins 1996; Little et al. 2002). Organic nitrogen may be further divided into two organic matter pools: a fresh (labile) pool and an older stabilized pool (Springob and Kirchmann 2003). In addition to its importance for productivity, nitrogen cycling (Figure 2-1) processes in soils are intimately linked to the carbon cycle (Raison and Stottlemyer 1991).



Figure 2-1: Schematic representation of N cycling in soils (modified from Hausenbuille 1985)

Total nitrogen is defined as the difference between the amount of nitrogen added to the soil *via* atmospheric deposition and biological fixation, and the amount lost through leaching, runoff, volatilization and denitrification. Plantavailable nitrogen further depends on abiotic controls, such as adsorption on the soil exchange complex, as well as on soil microbial activity through mineralization and immobilization processes. Nitrogen mineralization is the biological conversion of organic nitrogen to inorganic nitrogen, in the form of ammonium (NH<sub>4</sub><sup>+</sup>) and nitrate (NO<sub>3</sub><sup>-</sup>) through microbial activities, while immobilization refers to the assimilation of ammonium by the microbial biomass. Plants and microbes may compete for inorganic nitrogen (Binkley et al. 1990), although organic (i.e. amino-acid) nitrogen uptake is becoming increasingly recognized as an important nitrogen source for plant nutrition in boreal forests (Persson and Näsholm 2001).

Nitrogen mineralization can be divided into gross and net mineralization. Gross mineralization is equivalent to the sum of gross nitrification and gross ammonification i.e., the amount of nitrate and ammonium released during mineralization processes, while net mineralization takes into account microbial immobilization. Net mineralization is typically measured as the change of inorganic nitrogen in the soil during an incubation period either *in situ* using techniques such as the buried-bag, closed top cylinders, resin cores and resin bags (Eno 1960; Adams and Attiwill 1986; Distefano and Gholz 1986; Raison et al. 1987; Binkley and Hart 1989) or in the laboratory, under aerobic conditions (Fierer and Schimel 2002; Knoepp and Swank 2002; Lindo and Visser 2003). Because net mineralization may be a better indicator of plant-available nitrogen than gross, this literature review concentrates on net nitrogen mineralization rates found in the boreal forest. In general, boreal forests are associated with low rates of net N mineralization (Bonan 1990; Brais et al. 1995).

A useful soil model on which to base a discussion of soil properties is that proposed by Jenny (1941). Any soil property (S) is dependent on four environmental soil-forming factors: climate (cl), organisms (o), relief (r) and parent material (p), that act over time (t) according to the following equation:

Equation 2-1 
$$S = f(cl, o, r, p,t)$$

These soil-forming factors influence net N mineralization rates, thus changes that occur within these factors may increase or decrease net N mineralization rates. Each factor will be discussed in this chapter independently, however it is important to acknowledge that net nitrogen mineralization rates also depend on the interactions between these factors.

#### 2.2 Climate

Boreal forests are characterized by short, moderately warm, moist summers and long, extremely cold, dry winters (Larsen 1980). Climatic effects may be further divided into the effects of temperature and moisture, as well as the interaction between the two.

#### 2.2.1 Temperature

Temperature influences total microbial biomass and microbial activity within the soil (Campbell et al. 1973). The overall microbial community responsible for nitrogen mineralization is most active between 0°C and 35°C (Stanford et al. 1973). The two groups of microbes involved are broadly categorized as ammonifiers (mainly heterotrophic) and nitrifiers (mainly autotrophic) (Kowalenko and Cameron 1976). Ammonium is converted to nitrate with sufficient aeration and sufficient moisture over a broad range of temperatures (Stanford and Epstien 1974). Kowalenko and Cameron (1976) found that nitrifiers were at an optimum temperature between 15°C and 35°C, while ammonifier activity increased up to a temperatures of 35°C. Nitrification is typically inhibited by low pH, low temperatures and insufficient aeration (Myrold 2005). Optimal temperature for microbial activity may vary with soil horizons. Offord (1999) demonstrated that the maximum mineralization rates in a boreal mixedwood forest organic horizon occurred at 12°C, while in the Ae horizon a temperature of 22°C produced the highest net N mineralization rate.

Seasonal variation has been investigated to monitor changes in mineralization under different temperature and moisture regimes. Van Cleve et al. (1993) reported seasonal trends in rates of net N mineralization during a threeyear study conducted in Alaska. Minimal mineralization rates were found during the winter season, and increase during the growing season. Stottlemyer and Toczydlowski (1999) found net mineralization rates changed seasonally, and were highest in May and June for most tree species in Isle Royale National Park, USA.

Increasing soil temperature increases the decomposition rate of soil organic matter, which influences the mineralization rate (Bonan and Van Cleve 1991; Nicolardot et al. 1994). Pastor and Post (1986) suggest that an increase in air temperature may increase nitrogen mineralization by encouraging foliage with high concentrations of nitrogen and low concentrations of lignin. The importance of litter quality in nitrogen mineralization will be further discussed in section 2.3. Bonan and Van Cleve (1991) modeled plant production and decomposition fluxes in boreal forests. They reported that although warmer soils resulted in a loss of carbon in soil *via* an increase in microbial respiration, tree growth was enhanced due to an increase in nitrogen mineralization. Microclimate variations may also influence decomposition rates. Köchy and Wilson (1997) compared decomposition rates in aspen and grass litter placed in a forest and prairie environment. Shade seemed to hinder decomposition rates. Nitrogen mineralization was not examined in this study.

Nitrogen mineralization was found to decline with decreasing forest floor temperature (Stanford et al. 1973; Stottlemyer et al. 1995; Stottlemyer and Toczydlowski 1999). Laboratory incubation studies manipulating temperature were conducted to examine the changes in net nitrogen mineralization as temperature was altered. Although increasing the temperature increased net N mineralization rates, the rate of increase was not constant. For example, Stottlemyer and Toczydlowski (1999) examined the effects of temperature and moisture on net N mineralization in Michigan. There was no net N mineralization

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change between 5°C and 10°C; however, at temperatures higher than 10°C net N mineralization increased. A laboratory incubation experiment conducted by Stottlemyer et al. (1995) showed similar results. The laboratory incubation in that experiment found that net mineralization, regardless of tree species, increased with rise in temperature, especially for temperatures between 10°C and 15°C. Nadelhoffer et al. (1991) examined mineralization rates in six arctic soils in Alaska. The results indicated that N mineralization in these arctic soils did not change between 3° and 9°C but increased by a factor of two or more between 9° and 15°C. Soil temperature may be a more important regulator of mineralization rates in areas where monthly mean temperatures are higher and fluctuate throughout the year (Ellert and Bettany 1992).

The relationship between temperature and microbial activities can be expressed using the Arrhénius equation, which was formulated for chemical reactions (Bray and White 1966). This equation has also been used to describe the relationship between temperature and N mineralization in Gray Luvisolic soils (Campbell et al. 1984).

Equation 2-2  $\ln k = 17.75-6146/T$ 

where k=rate constant and T = absolute temperature (in  $^{\circ}$ K).

The  $Q_{10}$  factor, the increase in reaction rate for each 10° increase in temperature, can be calculated using the Arrhénius equation (Winkler et al. 1996).

Equation 2-3  $Q_{10} = K_{(t+10)}/K_t$ 

where K is the constant for the particular reaction and t is the temperature (K). A  $Q_{10}$  equal to 2.0 is typically used to describe the field relationship between temperature and N mineralization (Campbell et al. 1984). Assuming first order kinetics,  $Q_{10}$  values may be calculated for short durations by substituting N<sub>o</sub> (cumulative net N mineralization) with k in the Arrhénius equation (Kladivko and Keeney 1987).

Nicolardot et al. (1994) suggested that applying the Arrhénius equation to complex reactions is of limited value because the initial reactants or end products are not well known, and different microbial species and enzyme reactions are present during soil processes. Novak (1974) further noted that during decomposition the temperature coefficient for microbial growth is substrate dependent, hence would not follow first order kinetics.

#### 2.2.2 Moisture

Sufficient moisture contents are needed to transport nutrients to microorganisms (Filonov et al. 1999). In addition, an optimal moisture content allows movement of mobile microbes to substrates and provides water for metabolic processes of microbes and other organisms, while still allowing good soil aeration (Zaman and Chang 2004).

In nature, soil moisture content may vary over a wide range depending on the ecosystem and the environmental conditions at that ecosystem (Filonov et al. 1999). As previously mentioned, soil organic matter decomposition is important in net N mineralization. Kladivko and Keeney (1987) determined that a water tension between 10 kPa and 33 kPa is optimal for decomposition of soil organic matter. Offord (1999) found N mineralization to be optimal at 20 kPA. Optimum soil moisture content is close to field capacity (Stanford and Epstein 1974; Zaman and Chang 2004). A moisture content that exceeds this optimal water content will slow decomposition rates by decreasing the aerobic microbial community (Filonov et al. 1999).

Net N mineralization has been found to be positively correlated to moisture content (Kowalenko and Cameron 1976; Campbell et al. 1984; Stottlemyer and Toczydlowski 1999; Carmosini et al. 2003; Zaman and Chang 2004), although Offord (1999) and Pastor et al. (1984) found no correlation. The effect of moisture content on net N mineralization may depend on the experimental procedure. Cassman and Munns (1980) found that when water was added to air-dried soil, N mineralization decreased; however when the same soil was equilibrated by pressure membranes, nitrogen mineralization increased. Similarly to temperature, seasonal trends can occur with moisture content. Stottlemyer et al. (1995) observed that the highest soil moisture content in a southern boreal forest occurred in mid-May possibly due to snowmelt inputs, increased precipitation and low evapotranspiration. After mid-May the higher temperature and declining soil moisture reduced the net N mineralization. Nitrogen mineralization is also affected by rapid re-wetting of dry soil. This reaction has been shown to increase nitrogen mineralization rates for a few days following the increased water content (Franzluebbers et al. 2000). The quick increase in water may cause microbial cell lysis, releasing a flush of labile carbon and nitrogen into the soil (Van Gestel et al. 1992).

#### 2.2.3 Water-Temperature Interaction

Although there is a general agreement in the literature that temperature and moisture both influence mineralization rates, there are conflicting results regarding the temperature-moisture interaction and its potential influence on mineralization rates. Experiments have shown that nitrogen mineralization rates at optimal temperature decrease with decreasing moisture content. Kladivko and Keeney (1987) examined the relationship between the water content-soil temperature interaction and nitrogen mineralization using the Arrhénius equation. It was concluded that, although there may be a relationship between moisture content and N mineralization rate, there was no water-temperature interaction influencing nitrogen mineralization. In contrast, Wildung et al. (1975), Kowalenko and Cameron (1976), and Cassman and Munns (1980) all concluded that an interaction between soil temperature-water content existed. Knoepp and Swank (2002) found a moisture-temperature interaction in the A horizon of a hardwood forest when temperature was not limiting; however when temperature was limiting, increasing moisture content had little affect on nitrogen mineralization. There was no temperature-moisture interaction in the AB horizon.

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#### 2.3 Organisms

Nitrogen inputs to soils are primarily derived from atmospheric deposition and nitrogen fixation (Bhatti et al. 2002). Atmospheric deposition can range up to 75 kg ha<sup>-1</sup> y<sup>-1</sup> found in industrial areas (Dise and Wright 1995). High deposition in industrial areas makes up a large fraction of annual tree N requirements in many northern hemisphere temperate forests (Raison and Stottlemyer 1991).

Most of the nitrogen in the nitrogen cycle is in the form of atmospheric nitrogen (N<sub>2</sub>) and is fixed before entering the soil (Hausenbuiller 1985) (Figure 2-1). Biological fixation is mediated by symbiotic and non-symbiotic prokaryotes, while abiotic fixation occurs through lightning discharge or chemical fertilizer production (Hausenbuiller 1985). Diazotrophs, organisms that have the ability to fix nitrogen, are divided into archeabacteria, bacteria, and cyanobacteria. Of these organisms group only a very small proportion of species can perform the fixation process (Dixon and Wheeler 1986). Non-symbiotic microorganisms fix less then 1 kg ha<sup>-1</sup> y<sup>-1</sup> (Raison and Stottlemyer 1991). Nitrogen fixing plants, such as Alder (*Alnus* spp.), are found in various ecosystems (Keeney 1980) and form a symbiotic relationship with microorganisms (Dixon and Wheeler 1986) fixing up to 200 kg ha<sup>-1</sup> y<sup>-1</sup> (Raison and Stottlemyer 1991). Legumes, which form symbiotic relationships with *Rhizobium* spp., fix 50-150 kg ha<sup>-1</sup> y<sup>-1</sup> (Raison and Stottlemyer 1991).

The transformation of organic nitrogen to ammonium is performed by chemo-heterotrophic microbes forming ammonium as a by-product of carbon usage (Hausenbuiller 1985). Nitrification is mainly mediated by autotrophic *Nitrosomonas* spp. and *Nitrobacter* spp. (Kowalenko and Cameron 1976; Hausenbuiller 1985), although it is now recognized that heterotrophic nitrification occurs and can be of significance, especially in acidic forest soils (Schimel et al. 1984; Kuenen et al. 1988).

The surface organic layer in a forest is divided into L, F, and H horizons based on the stage of decomposition (Keeney 1980). This organic layer is an important source of nutrients, contributes to soil moisture retention and structure (Prescott 2000b), and contains most of the seed bank (Granström 1981). The L,

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or litter layer, consists of material that is undecomposed, mainly from overstory vegetation such as cones, twigs and leaves. The F, fermented or fibric layer, is partially decomposed and the H, humic layer, is completely decomposed with no identifiable plant material present (Alban 1982). The L layer generally has a C:N ratio of 35 to 67:1, whereas the F and H horizons have C:N ratios of 25 to 49:1 (Keeney 1980). The thickness of the humic layer is important in controlling decomposition rates. Fyles and McGill (1986) compared the decomposition rates of foliage litter from jack pine, white spruce, balsam fir, green alder, two feathermosses, and lichen. The thin forest floor associated with pine/lichen stands had a rapid rate of decomposition, while the thicker humic layer associated with spruce, fir, and aspen showed a slower decomposition rate.

The physical and chemical properties of litter affect soil organic matter decomposition rates which, in turn, influence forest floor characteristics (Swift et al. 1979; Fyles and McGill 1986). Differences in nutrient availability among different forest types may be due to differences in litter properties (Prescott 2002). Low nutrient availably in soils may be explained by a poor litter quality, that is litter with a high lignin content or a high lignin: N ratio, and a slow decomposition rate. Scott and Binkley (1997) combined data from studies with a variety of vegetation, climate, and soil types across North America to determine the relationship between above-ground litter chemistry and annual net N mineralization. The analysis indicated that in forested ecosystems, net N mineralization decreased as the litter lignin:N ratio increased. Köchy and Wilson (1997) compared the decomposition rates of aspen litter to that of grass litter. When the two litter types were incubated in the same area decomposition rates were significantly lower for the aspen than for the grass litter. After being incubated for 149 days 81% of the original aspen remained, whereas only 59% of the grass litter remained. An explanation for this may be that aspen has a lower N concentration then the grass litter and a higher lignin content. The negative correlation between annual net mineralization and litter lignin:N ratio is also supported by studies performed by Stump and Binkley (1993) and Van Cleve et al. (1993). In contrast, Prescott et al. (2000b) found no relationship between litter

decomposition rates and nitrogen mineralization and Fyles and McGill (1986) found no correlation between decomposition rates and C:N ratios, lignin, or lignin:N ratios. Fox et al. (1990) reported that with legumes, lignin:N ratios were not significantly correlated with N mineralization although the lignin + polyphenol:N ratio was negatively correlated to net N mineralization.

Organic N is returned to the soil through litter fall. Therefore, vegetation directly influences net N mineralization rates through the amount and quality of its litter. Prescott (2000a) suggests that litter mass and nutrient return *via* litterfall may predict nutrient availability better than litter decay rate. Foliage characteristics determine the amount of nutrients recycled as well as nutrient availability. Pastor et al. (1984) determined that soil N mineralization increased as litter production and N return in litter increases. However, Scott and Binkley (1997) found no correlation between litterfall mass and net N mineralization.

Overall, numerous studies have been conducted to assess if there is a significant difference in mineralization rates with regard to different plants, especially comparing deciduous and coniferous species. The forest soil organic N pool size is typically larger in coniferous sites compared to deciduous sites, however this pool is also more recalcitrant in coniferous soils (Côté et al. 2000). The difference in mineralization rates between these two forest types may also be due to differences in litter quality (Parè and Bergeron 1996). Côté et al. (2000) established that based on an organic carbon basis, N mineralization rates were higher in deciduous stands compared to coniferous stands in the forest floor of a boreal mixedwood forest. In this experiment birch trees were associated with the highest N mineralization rate (8.8 mg  $g^{-1}$  OC), followed by aspen (7.18 mg  $g^{-1}$ OC), while the conifer species had the lowest N mineralization rate (5.7 mg  $g^{-1}$ OC). The N mineralized was negatively related to lignin:N and C:N ratios. Lindo and Visser (2003) examined differences between deciduous and coniferous forests in the upper boreal region and found higher mineralization rates in the soils under deciduous than in the coniferous stands. Vance and Chapin (2001) showed similar results in a taiga forest floor. Field incubations showed that north-facing sloped birch had the highest mineralization (4.1  $\mu$ g N g<sup>-1</sup>soil d<sup>-1</sup>) followed by

aspen (3.6  $\mu$ g N g<sup>-1</sup>soil d<sup>-1</sup>) and then spruce (-0.06  $\mu$ g N g<sup>-1</sup>soil d<sup>-1</sup>). Although aspen and spruce had low organic matter quality (high lignin and lignin:N) the aspen site had attributes favorable to rapid decomposition (e.g. high pH and high soil temperature). Similar results were reported by Paré and Bergeron (1996). Smolander and Kitunen (2002) found that net N mineralization was higher in soils from birch and spruce stands than in pine stands. This result is not universal. Thomas and Prescott (2000) observed that fir stands had a greater net N mineralization rate than both birch and lodgepole pine stands in British Columbia. Flyes and McGill (1986) examined the difference in nitrogen mineralization characteristics in central Alberta, within jack pine and white spruce stands. The total N mineralized over the 37-week incubation period showed that the jack pinelichen stand had a lower mineralization rate than the other stands of spruce and the pine/alder stands. Stottlemyer and Toczydlowski (1999) did not find a correlation between species composition (white birch, quaking aspen, white spruce, and balsam fir) and net N mineralization rates. Average net N mineralization rates were also similar between lodgepole pine and aspen stands from northern Colorado (Giardina et al. 2001).

#### 2.4 Relief

Topography may indirectly affect soil mineralization rates through its influence on the hydrological cycle, in particular through water movement down slope and accumulation in the lower elevation areas. The extra soil moisture in depressions can promote plant production and nutrient biocycling; on the other hand, slow decomposition rates result when soils become saturated (Zhu 1991). In northern latitudes soil radiation is greater on south facing than on north facing aspects, resulting in lower relative humidity and available soil moisture but higher daytime temperatures (Hutchins et al. 1976). This in turn influences the type of vegetation found on a given slope. Venterea et al. (2003) reported that in a hardwood conifer forest there was a positive correlation between net mineralization rates and southern aspects.

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Nitrogen mineralization rates at different slope positions have also been investigated. Little et al. (2002) examined mineralization rates along a post-fire white spruce toposequence in Gray Luvisolic soils from west-central Alberta. Although the C:N ratio varied along the toposequences there was no significant difference in the net N mineralization rate. No changes occurred between different slope positions incubated at the same temperature in an experiment on an Alberta slope conducted by Offord (1999). Walley et al. (1996) studied the influence of topography on mineralization rates after site disturbance in Prince Albert, Saskatchewan. Again, results indicated no significant difference in mineralization rates at different topographic positions. The conclusion that there is no correlation between net N mineralization rates and topographic positions is not restricted to the boreal forest zone. Zak et al. (1991) reported that net N mineralization was not significantly affected by slope position in a pine oak ecosystem in central Minnesota and Hirobe et al. (1999) did not find a clear net N mineralization gradient along a slope in a Japan forested plantation.

#### 2.5 Parent Material

The lack of nitrogen in mineral parent materials is one of the reasons that nitrogen is limiting in terrestrial ecosystems (Van Cleve 1993). Jenny (1941) described soil development as a step-wise sequence from rock to weathered rock to immature soil to mature soil. This section will examine the effects of soil texture and depth on net mineralization rates.

Soil texture is dependent on parent material mineralogy and type of deposition, and may also vary in response to the extent of the weathering process (Silver et al. 2000). Finer textured soils typically contain more organic matter and support greater microbial biomass (Zak et al. 1994), and may provide protection to microbes from grazing and drought (Côté et al. 2000). On the other hand, the fine pore size of clay soils may not allow easy microbial access, thereby restricting microbial habitats and activity (Côté et al. 2000). Additionally, texture directly influences soil moisture content (Pastor et al. 1984). Sandy-textured soils typically have low water content, which may impede nitrogen mineralization.

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In a natural Brazilian forest, higher concentrations of organic matter were found in finer textured soils (clay), and were associated with higher net N mineralization compared to coarser textured (sandy loam) soils (Neill et al. 1997). An experiment conducted by Côté et al. (2000) showed lower N mineralization in clay than in till (which also had a clay texture but a lower percentage of clay). Silver et al. (2000) found that along a 500 m textural gradient in the Amazon net N mineralization increased significantly from sand to clay in most sites. Pastor et al. (1984) determined that nitrogen mineralization was positively correlated with the subsoil silt + clay content in Wisconsin. Reich et al. (1997) found greater net N mineralization on Luvisols (~70% silt + clay) than on Regosols (~16% silt + clay).

Conversely, studies have also shown that clay content has no effect on net N mineralization rates. Giardina et al. (2001) examined mineralization rates in an aspen and pine dominated forest in northern Colorado and found no relationship between clay content and net N mineralization. Similar results were reported by Motavalli et al. (1995). Fyles and McGill (1986) found no differences in net N mineralization between the forest floor and the A horizon, but both horizons exceeded the mineralization from the B horizon. The amount mineralized was negatively correlated to the clay content, which was higher in the B horizon.

Microbial activity in boreal forest soils is concentrated in the surface forest floor layer (Fyles and McGill 1986; Van Cleve et al. 1993). Differences in mineralization rates occur among the different organic LFH horizons. The litter (L) layer is more active in nitrification while the fermented (F) and humic (H) layers are more active in mineralization (Flyes and McGill 1986). In acidic coniferous forests Persson and Wirén (1995) demonstrated that 32-74% of the net N mineralization occurs in the organic horizon and the top 10 cm of the mineral horizon. Assuming uniform bulk density, Cassman and Munns (1980) estimated that 42% of the total N mineralized occurs in the 0-18 cm depth interval of a silty loam soil in California. Ketilson (2004) examined differences in LFH and mineral soil of a boreal forest. Upland plots exhibited higher mineralization in the LFH horizon (2.8 g N m<sup>-2</sup>) than in the mineral horizon down to 10 cm (0.35 g N

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 $m^{-2}$ ). To the contrary, Offord (1999) determined that specific net N mineralization rates were higher in the Ae horizon than in the organic horizon.

#### 2.6 Time

A strong relationship exists between the evolution of vegetative communities and the evolution of soils (Lanoue 2003). The succession of an upland boreal forest generally follows one of two patterns: 1) after a fire aspen and birch develop then spruce; or 2) white spruce will invade with or without hardwoods (Van Cleve and Viereck 1981). Succession following various disturbances can influence the rate of mineralization in boreal forests. Van Cleve et al. (1993) compared different successional stages in Alaska. In all of the successional stages examined, the rate of mineralization and nitrification increased following vegetation clearing. Differences in species cover, thus differences in the quality of the litter produced, may directly impact the rate of mineralization in different successional stages. It is generally accepted that soil nutrients change during ecological succession, however the magnitude and direction of these changes vary.

Stand age is found to influence nitrogen characteristics within the LFH layer. Flyes and McGill (1986) examined mineralization rates in different stand ages (45-140 years old) in the boreal forest of central Alberta. Results showed that in the LFH layer, only the mineralization rate constant (k) was affected by age. During pedogenesis the L:FH ratio decreases increasing the rate constant for the total forest floor. Côté et al. (2000) examined nitrogen mineralization between a 50-and a 124-year old stand. On an OC weight basis the mineral soil from the oldest stand had the highest rate of N mineralization (4.54 mg g<sup>-1</sup> OC in the older stand and 3.7 mg g<sup>-1</sup> OC in the younger stand) while the forest floor layer showed similar N mineralization rates in the old and young stands (both 7.23 mg g<sup>-1</sup> OC). However, on an areal basis N mineralization rates were higher in the old stands in both the forest floor and the mineral soil layer. It is hypothesized that these results are due to a higher "quality" of soil organic matter in older stands compared to younger stands.

After a disturbance, net N mineralization typically increases for a short duration, and then decreases over the long term (Walley et al. 1996; Monleon et al. 1997; Giardina and Rhodes 2001). Van Cleve et al. (1993) reported an increase in mineralization rates after the vegetation was cleared. The total annual net N mineralization in the forest floor increased by 2.4 and 1.6 times in a poplaralder and white spruce stand, respectively, possibly due to an increase in temperature following harvesting. Similarly, Carmosini et al (2003) examined net N mineralization in mature and 2.5-year-old logged trembling aspen stands in Alberta. Cumulative net N mineralization rates over the growing season resulted in higher net N mineralization in the logged stand as compared to the mature stand. Higher mineralization rates after clear-cutting have also been reported by Matson and Vitousek (1981), and Vitousek and Andariese (1986). Walley et al. (1996) found that in Prince Albert, Saskatchewan, mineralization rates four years after disturbance (burning or clear cutting) were lower than in the native forest.

#### 2.7 Synthesis

The factors that affect net nitrogen mineralization may be described using the equation S = f(cl, o, r, p, t). This chapter provided a review of the literature explaining how each factor: climate, organisms, relief, parent material, and time influences net N mineralization rates. Climate was divided into temperature, moisture and the moisture content-soil temperature interaction. Net N mineralization has an optimal temperature and moisture, which is ecosystem dependant. For example, the optimal temperature and moisture for mineralization in the Arctic is different from what would be expected in a tropical region. The mineralization-temperature relationship may be described using the Arrhénius equation and the Q<sub>10</sub> factor. Orthic Gray Luvisols soils have an average Q<sub>10</sub> equal to 2.0, however, this number varies among ecosystems.

Atmospheric fixed nitrogen can be taken up by either plants or soil microorganisms. Microbes complete the cycle by transforming the organic nitrogen into inorganic forms (ammonium and nitrate) available for further uptake. The microbes involved in the mineralization process are primarily autotrophic nitrifiers and heterotrophic ammonifiers, which produce inorganic forms of nitrogen at different optimal moisture and temperature.

The type of vegetation on a site influences the net N mineralization rates through leaf litter composition (lignin, amount of N, lignin:N, mass). Although the actual influence of leaf litter quality is questionable, it is generally found that different types of vegetation (i.e. coniferous vs. deciduous forests) produce different mineralization responses.

Few studies have reported relief as having an influence on net N mineralization. Parent material influences the texture of each horizon. Mineralization occurs mainly in the organic horizon and decreases with depth. Mineralization has been found to be negatively correlated with clay and positively correlated with silt + clay content. Finally, ecological succession following disturbance involves changes in the vegetative cover, which leads to a change in net N mineralization in the associated soil. Generally, after a disturbance mineralization rates increase for the short term and then decrease in the long term as the soil enters a phase of recovery.

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# CHAPTER 3. NITROGEN MINERALIZATION IN RECLAIMED FOREST FLOOR ANDPEAT-MINERAL MIX CAPPING A PLEISTOCENE CLAY DEPOSIT

# Introduction

Northern Alberta lies within the Boreal forest region. In boreal forests, the most commonly limiting nutrient is nitrogen, as it is mainly present in unavailable organic forms (Kimmins 1996). Microbial mineralization rates, which are influenced by organic matter quality and microclimatic conditions, control the release of nitrogen into inorganic forms (ammonium and nitrate) that are directly available to plants. Nitrogen mineralization rates are often low in undisturbed boreal forests, and have been shown to further decrease, in the long term, after disturbances such as logging and burning (Walley et al. 1996, Monleon et al. 1997; Giardina and Rhodes 2001). Oil sand mining is an important resource in northern Alberta, raising concerns about impacts to nitrogen cycling during the disturbance and reclamation procedure. Practices such as stockpiling soil before reclamation, mixing materials and reclaiming with large equipment have been shown to alter soil properties such as bulk density, organic matter content and microbial biomass (Kaiser et al. 1991; Grigal 2000; Mummey et al. 2002). Another key component in reclamation is the quality of the organic capping used in the reconstructed soils. All of these factors may affect nitrogen mineralization fluxes and need to be more closely examined.

The Athabasca oil sands deposit around Fort McMurray, northeastern Alberta, is the largest oil deposit in the world, with an estimated reserve of one trillion barrels of bitumen (Kimball et al. 2000). Current bitumen extraction is focused on shallow oil sand deposits where surface mining is feasible. The impacted area to date is about  $150 \text{ km}^2$ , and it has been predicted that by the year 2023, the disturbance may be as much as 10 times the currently affected area (AEP 1998).

Following exploitation, the challenge of land reclamation entails the creation of a soil-like profile suitable for plant growth using tailings sand, mature fine tails, overburden (lean oil sand, glacial till, glacio-lacustrine materials,

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muskeg, etc.), composite tails and reclamation material, with a peat-mineral mix typically used as a surface treatment (Fung and Macyk 2000). More recently, salvage operations have used the forest floor (LFH) stripped from pre-mining areas as an alternate treatment for soil reclamation. In addition to providing an excellent seed bank for native plant species (Granström 1981; Qi and Scarratt 1998), this organic layer is a store of nutrients promoting microbial activity including organic matter decomposition and mineralization (Fyles and McGill 1986; Van Cleve et al. 1993).

Chemical, physical, and biological properties of peat-mineral mixes have been characterized by Li et al. (2003) and Lanoue (2003). Yet, no one has investigated and compared characteristics of soils reclaimed with the standard reclamation technique (i.e. peat-mineral mix) and the more recent approach, using reclaimed LFH. This study has two main objectives: first, to compare the properties of soils reclaimed with LFH to those reclaimed with the traditional peat-mineral mix; and secondly, to compare reclaimed areas with LFH to an undisturbed forest ecosystem. It is hypothesized that adding an LFH organic treatment to the reclaimed land will increase nitrogen release by mineralization as compared to the peat-mineral mix organic treatment. Furthermore, because the reclaimed LFH areas are so young, it is hypothesized that they will foster less favorable conditions for mineralization than the LFH at an established natural forest site.

# Site Characteristics and History

The study area is located approximately 40 km north of Fort McMurray, northeastern Alberta (57° N latitude and 111°W longitude). The area is characterized by long and cold winters (January average temperature is –18.8°C), short and warm summers (July average temperature is +16.8°C), and an average annual precipitation of 455 mm (Environment Canada 2002). Fort McMurray is located in the Boreal forest region, with white spruce (*Picea glauca* (Moench) Voss), black spruce (*Picea mariana* (Mill.) BSP), trembling aspen (*Populus tremuloides* Michx.), balsam poplar (*Populus balsamifera* L.), white birch (*Betula*  *papyfrifera* Marsh.), and jack pine (*Pinus banksiana* Lamb.) as the main vegetative species (Fung and Macyk 2000). The majority of soils in this area have developed on glacial and post-glacial deposits (Turchenek and Lindsay 1982). Gray Luvisols are associated with till and lacustrine deposits, while Dystric Brunisols typically developed in coarser parent material such as outwash glaciofluvial and eolian sands (Turchenek and Lindsay 1982; Lanoue 2003). Organic materials overlying glacial deposits cover about 60 percent of the area. The Fibrisol and Mesisol great groups of the Organic order are the most common soils in the area, and are composed of undecomposed to moderately decomposed peat (Turchenek and Lindsay 1982).

Syncrude Canada Ltd., one of the major oil sand operators in the Athabasca region, is the largest producer of crude oil, from oil sands, in the world (Syncrude Canada Ltd. 2001). In areas where the oil sand deposit is less than 45 m below the surface, Syncrude uses surface mining to access the oil-impregnated deposit (Fung and Macyk 2000). Underlying the organic LFH or peat materials are Pleistocene deposits which contain mineral materials (called secondary material), with high pH and clay content, Cretaceous overburden material that is a non-sodic/non-saline material salvaged from directly above the oil sands, and tailings sand which is the remaining material after the oil has been extracted from the sand (Lanoue 2003). See appendix A (Figure A-1) for a schematic diagram.

Surface soils and near-surface geological materials, overburden and secondary material, are salvaged and placed as the reconstructed soil cover on the newly-reconstructed landforms. In addition to being used to reclaim disturbed areas, suitable overburden materials are used to build storage areas and terraced dykes.

Organic materials used for land reclamation in this study include mesic peat mixed with the underlying mineral material, extracted from a nearby peatland, and forest floor (LFH) material salvaged from an upland aspen dominated forest that was deforested in 1996, stripped to a depth of 7.8 cm in 1998 and windrowed with vegetation litter for approximately 20 hours (Qualizza *per. com.*). Reclaimed sites included in this study were located at the Syncrude Canada Ltd. Mildred Lake Mine site. Three different reclamation treatments had been previously applied at this site: (i) peat-mineral mix (15 cm of peat-mineral mix over 35 cm of secondary material) established in winter 1999 on a gentle west-facing slope (ii)LFH over peat-mineral mix (11 cm of LFH over 18 cm of peat-mineral mix and 35 cm of secondary material) established in summer of 1998 on the same gentle west-facing slope and (iii) LFH over secondary (18 cm of LFH over 23 cm of secondary material) established in winter 1999, level. All materials were placed on tailings sands from which oil had been extracted during processing. See appendix A (Figure A-2) for a schematic diagram.

The reclaimed LFH sites were not fertilized, however, the peat-mineral mix was fertilized to 500-600 kg/ha of 10-30-15-4 mix (N-P-K-S) and was planted to barley in the summer of 1998. An undisturbed upland aspen dominated forest, 4 km south of the site from which the LFH had been extracted, was also included as a comparison to the reclaimed sites (LFH average depth is 5.1 cm).

# Methods

#### Field Methods

Three 10 m x 10 m replicate plots were established randomly at each of the four study sites, and sampling was completed using a randomized block design at the following three dates: June 2003, July 2003, and May 2004.

At each sampling location two aluminum cores (7 cm diameter x 7 cm deep) were driven into the ground with a 2 cm distance between cores. The soil from one of the cores was taken to the laboratory for initial analysis while the soil from the other core was placed in a polyethylene bag (15 m thick) and buried in the LFH over peat-mineral mix site for a 30-day field incubation (Eno 1960). In June and July 2003, four pairs of samples were extracted from each plot. In May 2004, 12 pairs of samples were extracted per plot and composited into four pairs prior to analysis, in an attempt to reduce the spatial variability that had been observed at the sites in 2003. Bulk density was determined on four samples per plot using an aluminum core (7 cm diameter x 4 cm deep).

A reciprocal transfer experiment was also conducted. Soil samples from the reclaimed LFH sites were incubated *in situ* (source) as well as incubated into the natural forest (host) site. Likewise, soil samples from the natural forest site were incubated *in situ* as well as in each of the reclaimed LFH sites.

#### Laboratory Experiment

In August 2004, six samples were taken from each plot using the same core sampler as mentioned above (7 cm diameter x 7 cm deep), composited into three samples per plot and sieved to 4 mm. Twenty grams of soil from each composite sample was placed in 1 L Mason jars with a septa on the top and incubated at 20°C. Each composite sample was divided into a control sample, where the moisture content was not changed, as well as into three additional samples where moisture contents were adjusted to 30%, 45%, and 60% by weight. Sixty percent was chosen as the highest moisture content because it was the average moisture content of the natural forest site at the time of sampling. The experiment was duplicated with the same composite samples to include one set of soil samples to be used for the initial measurements of  $NH_4^+$  and  $NO_3^-$  and microbial biomass C and N, and one set for the final measurement four weeks later. Wetting soil yields a short-term increase in mineralization rates (Fierer and Schimel 2001), thus the soil was incubated for two weeks to allow the soil to stabilize before the initial soil set was extracted for determination of microbial biomass and net N mineralization. See appendix B (Figure B-3) for a schematic diagram.

The soils were weighed weekly to ensure that the correct moisture content was maintained and then  $CO_2$  measurements were taken with a syringe through the septa on the Mason jar, and analyzed by a gas chromatograph (5890 series II Hewlett Packard, Wilimington, Delaware). To avoid development of an anoxic environment the Mason lids were removed for aeration for two hours after  $CO_2$ concentration measurements were taken.

#### Laboratory Analysis

All samples were sieved to 4 mm prior to analysis, except for bulk density, where unsieved samples were directly dried for 48 hours at 70°C and then weighed. Moisture content was determined by oven drying the soil samples for 48 hours at 70°C. Measurement of the pH was conducted using a glass electrode in a 1:4 field moist soil:0.001M CaCl<sub>2</sub> suspension (Karla and Maynard 1991). Total C and N were air dried, ground with a ball grinder, and then measured using a CN elemental analyzer (NA 1500, Carlo-Erba, Italy). Soil texture was determined using the hydrometer method with a pre-treatment of 30% H<sub>2</sub>O<sub>2</sub> to remove organic matter (Karla and Maynard 1991). Net nitrogen mineralization rates for the field and laboratory experiments were calculated using the following formula:  $(NO_3^- + NH_4^+)_{incubated} - (NO_3^- + NH_4^+)_{initial}$  where  $(NO_3^- + NH_4^+)_{incubated}$ corresponds to the sum of nitrate and ammonium concentrations in the incubated bag (field) or Mason jar (laboratory) after incubation and  $(NO_3^- + NH_4^+)_{initial}$ corresponds to the sum of concentrations in the initial sample. Samples were extracted using 0.5M K<sub>2</sub>SO<sub>4</sub> (1:10 soil:K<sub>2</sub>SO<sub>4</sub>) as described by Mulvaney (1993). Ammonium and nitrate contents of the extracts were analyzed using a Technicon Auto Analyzer II (Technicon Industrial systems, Tarrytown, New York).

Microbial biomass C and N were determined from dissolved organic nitrogen (DON) and dissolved organic carbon (DOC) concentrations using the chloroform fumigation-extraction method where microbial biomass C=DOC<sub>fumigated</sub>-DOC<sub>unfumigated</sub>/K<sub>c</sub> and microbial biomass N=DON<sub>fumigated</sub>- $DON_{unfumigated}/K_n$  with a correction factor of K<sub>c</sub> and K<sub>n</sub>=0.45 (Jenkinson et al. 2004). Two soil samples were weighed to 10 g (dry weight basis); one was extracted directly using 0.5M K<sub>2</sub>SO<sub>4</sub> and the other was fumigated with chloroform for 24 hours and then extracted (Horwath and Paul 1994). In June and July 2003, DON was determined using the persulfate oxidation method where the organic nitrogen and NH<sub>4</sub><sup>+</sup> in a sample are oxidized into NO<sub>3</sub><sup>-</sup> and compared to an initial, non-oxidized sample (Cabrera and Beare 1993). DON= NO<sub>3</sub><sup>-</sup> after oxidation- $(NO_3^{-} + NH_4^{+})_{initial}$ . In 2003, dissolved organic carbon (DOC) was analyzed using a Soluble Carbon Analyzer (Astro 2001 II, Texas, USA). In May 2004, DOC and TN (total soluble nitrogen) were quantified directly using a Shimadzu TOC-VTN instrument (Mandel Scientific Company Inc. Ontario, Canada). DON was calculated as DON = TN -  $(NO_3^+ + NH_4^+)$ . A more detailed account of the methodology may be found in appendix B.

Respiration rates from week two were calculated from the  $CO_2$ concentrations by the gas chromatograph (5890 series II Hewlett Packard, Wilimington, Delaware) and divided into microbial biomass C to obtain  $qCO_2$ .

# Statistical Methods

Measurements from each plot within a site were averaged and then pooled (n=3) to calculate net N mineralization rate, microbial C and N, pH, moisture content, and total C and N. A one-way ANOVA PROC MIXED model was used to determine the significance of the treatment. Due to the small n value (n=3) it was not possible to check for the normality of the data, thus a conservative multiple comparison test, Tukey, was used with  $\alpha$ =0.05. A correlation analysis using data from all plots (n=48) was used to define relationships between all of the field measurements listed above. For the reciprocal transfer experiment, differences between buried in situ and transplanted samples were also tested using a one-way ANOVA and a Tukey multiple comparison test at  $\alpha$ =0.05. The respiration rates from the laboratory experiment were analyzed by calculating the cumulative CO<sub>2</sub> efflux of the last four weeks of the incubation, i.e. after the stabilization period. Respiration rates, microbial biomass C and N and mineralization rates from this experiment were analyzed using an ANOVA PROC GLM model and Tukey multiple comparison test at  $\alpha$ =0.05 (n=3). Regressions were completed using all samples of each material (n=24). Standard errors are shown through the text in parentheses after the mean. All tests were performed using SAS version 8.01 (SAS institute Inc. NC, USA).

#### Results

#### Soil Characteristics at the Reclaimed and Natural Sites

The reclaimed treatments had a significantly (p=0.05) higher bulk density, pH, and total C:N ratio than the natural forest site LFH (Table 3-1). There were no significant differences in physical and chemical properties among surfaces of reclaimed treatments except for a lower pH but higher total C in the peat-mineral mix as compared to the LFH treatments. All reclaimed sites had a silty loam

texture in the top 7 cm, and had an average temperature for May 2004 that was 5°C higher than in the natural forest (Table 3-1).

The reclaimed sites had a significantly lower moisture content than the natural forest site for each sampling month (Table 3-2). There were no significant differences in moisture contents among treatments during any month.

# In situ Net N Mineralization Rates

No statistically significant differences in net N mineralization were found between sites, but there was a trend for the natural forest to yield a higher mineralization rate than the reclaimed soils (Figure 3-1). The natural forest and the peat-mineral mix treatment showed similar net N mineralization rates at all sampling times. The reclaimed LFH sites showed the highest net N mineralization rates in June. The reciprocal transfer experiment showed no difference in mineralization rates when incubated in host and source sites (Table 3-3). Because of differences between bulk densities of the natural and reclaimed sites, net N mineralization was also calculated on an areal basis (mg N m<sup>-2</sup>). On an areal basis, net N mineralization rates tended to be higher in the reclaimed sites due to the higher bulk density and thicker horizons (Table 3-4). Net N mineralization rates on an areal basis were consistently higher in the LFH over secondary compared to the LFH over peat-mineral mix site.

#### In situ Microbial Biomass C and N

Microbial biomass C and N in the initial samples (i.e. prior to incubation) were always significantly lower in the reclaimed treatments as compared to the natural forest, with the exception of microbial N at the start of the experiment in May 2004 (p=0.0632) (Table 3-5). After incubation microbial biomass C and N were also significantly lower in the reclaimed treatments as compared to the natural forest with the exception of July 2003 when there was no difference between treatment and natural forest when incubated into the LFH over secondary material (microbial biomass C p=0.065, microbial biomass N p=0.127). During the field experiment, microbial biomass C ranged between 321 and 890 mg kg<sup>-1</sup> at

the reclaimed sites, while it ranged between 1573 and 2870 mg kg<sup>-1</sup> within the natural forest LFH. Microbial biomass N followed the same trend as microbial biomass C. The overall range of microbial biomass N was 23-118 mg N kg<sup>-1</sup> at the reclaimed sites, while the significantly higher range at the natural forest site was between 88 and 307 mg N kg<sup>-1</sup>. There was no significant difference in microbial biomass C and N between the different reclaimed materials, except on three accounts: June 2003 where, for the initial samples, the peat-mineral mix had a significantly lower microbial biomass N; July 2003, where, for the initial samples, the LFH over peat-mineral mix had a significantly higher microbial biomass N; and May 2004, where, for the samples incubated into LFH over peatmineral mix the peat-mineral mix had a significantly lower microbial biomass compared to the other reclaimed treatments (Table 3-5). Similarly to net N mineralization, microbial biomass C and N did not differ significantly when incubated in different materials, as shown by the reciprocal transfer results (Table 3-5). Regardless of where the samples were incubated, they always showed statistically higher microbial C and N for the natural LFH as compared to the reclaimed materials.

Each month showed a correlation between moisture content and microbial C, microbial N and pH; between microbial C and microbial N and DON; between total C/N and bulk density and between microbial N and DON. Table 3-6 shows one month (June 2003), which is representative of each month tested. Net N mineralization did not correlate with any other property measured.

#### Effects of Moisture Manipulation in Reclaimed Soils

Measured  $CO_2$  concentrations peaked between week one and three for all incubated materials, leveling after week three (Figure 3-2). Hence cumulative respiration rates were calculated after the steady state occurred (weeks 3-6) and examined. Cumulative respiration rates remained statistically higher in the natural forest than in the reclaimed treatments at all moisture contents, however the difference between the natural LFH and the reclaimed treatments decreased with increasing moisture (Figure 3-3). The regression analysis determined that respiration rates significantly increased with the addition of moisture (p=<0.0001 in all treatments). Within the reclaimed treatments, the peat-mineral mix material had the lowest respiration rate at all moisture content levels, and was significantly lower than the respiration rate found in the LFH over secondary material over entire the moisture gradient (Figure 3-3). The LFH over secondary material also had a higher respiration rate than the LFH over peat-mineral mix, however it was not significantly higher at 45% and 60%.

Net N mineralization rates did not change significantly with moisture content, however there was a trend of increasing net N mineralization with increasing moisture (Table 3-7). Similarly to the respiration results, LFH over secondary generally had the highest net N mineralization rates (except at 30%) and peat-mineral mix had the lowest mineralization rate regardless of moisture content.

Interestingly, microbial biomass C and N responded differently to the increase in moisture content. Microbial biomass N showed a positive correlation with moisture for all reclaimed material (Figure 3-4). Microbial biomass N was similar between all treatments at the original moisture content and at 30% moisture content (Table 3-7). When the moisture content was increased to 45% and 60% microbial biomass N became significantly higher in the LFH over secondary than in the peat-mineral mix, while the LFH over peat-mineral mix showed an intermediate value between the peat mineral mix and the LFH over secondary treatments. Microbial biomass C showed no relationship with moisture content within any treatment. The peat-mineral mix had the lowest microbial biomass C regardless of the moisture content, while the LFH over secondary had the highest microbial C (Table 3-7).

Microbial biomass C/N displayed a trend of decreasing as moisture content increased (Table 3-8). Original microbial biomass C/N were 18-36, while at 60% moisture content the microbial biomass C/N dropped by 27%-50% to 13-15. Values for the respiration rate:microbial biomass C ( $qCO_2$ ), again did not show significant differences but showed a general trend of increasing with moisture content (Table 3-8).

# Discussion

#### Soil Characteristics at the Reclaimed and Natural Sites

Differences in soil properties (pH, bulk density, total C and N) between reclaimed and natural sites generally occur due to the extraction and placement of the treatments (Schwenke et al. 1999; Stolt et al. 2001). Bulk density is often higher on reclaimed sites, as shown in our study (Table 3-1), because of the compaction caused by large equipment driving over the soil during soil placement (Bradshaw 1983; Grigal 2000). Overburden material is associated with a high pH, thus due to mixing the alkaline overbuden with the organic material, the reclaimed material is more alkaline than the natural forest (Fung and Macyk 2000; Mummey et al. 2002). The higher moisture content and lower temperature in the natural site may be attributed to the forest canopy cover, which insulates the soil, slowing evaporation and providing shade that decreases the soil temperature.

The natural forest total C and N contents (Table 3-1) are comparable to the range found in other studies in western Canada boreal forest floors (total carbon 1.5-5.5 kg C m<sup>-2</sup> (Bhatti et al. 2002); total nitrogen 0.05 to 1.68 kg N m<sup>-2</sup> (Offord 1999; Carmosini et al 2003; Ketilson 2004). Nitrogen inputs to soils supporting aspen dominated stands, through litterfall, throughfall and stemflow can be quite high, averaging 22 to 32 kg ha<sup>-1</sup> yr<sup>-1</sup> (Peterson and Peterson 1992), hence a high total N was expected for the total forest soil included in this study. A high C:N ratio was also expected in the peat-mineral mix because although peatlands may have a high total nitrogen content, the amount of total carbon is so high that C:N ratios have been found to be greater in peatlands compared to upland forests (Devito et al. 1999). Low organic matter contents are typical of reclaimed treatments when incorporating materials of lower organic matter subhorizon into the topsoil (Mummey et al. 2002). This may be why the reclaimed LFH has a significantly higher C:N ratio.

# In situ Net N Mineralization Rates

Research on net N mineralization rates in the boreal forest has been extensive. The net N mineralization rates found in this study were consistent with values reported during May to July field incubations in other boreal forest soils, ranging from 160 to 750 mg N m<sup>-2</sup> (Carmosini et al 2003; Ketilson 2004). The lower net N mineralization found in this study during the May incubation, also seen by Carmosini et al. (2003), may be due to lower soil temperature and reduced aeration following saturation during spring thawing (Stanford et al. 1973; Stottlemyer and Toczydlowski 1999; Bhatti et al. 2002).

Although not statistically different, lower net N mineralization rates were found in the reclaimed sites as compared to the natural forest (Figure 3-1). This may be related to the higher soil C:N ratios, which promote immobilization at the reclaimed sites (Paré and Van Cleve 1993; Côté et al 2000). The natural forest generally had a higher standard error and coefficient of variance compared to the reclaimed materials. The high spatial variability within the sites, especially the natural forest site, may be the reason that no statistical differences were found.

The reciprocal transfer experiment was conducted to differentiate between temperature effects and the potential influence of the material composition (i.e. initial moisture content and biochemical make-up) on net N mineralization rates. Microclimate, through differences in canopy cover, has been previously demonstrated to influence biological processes during a reciprocal transfer experiment (Coûteaux and Raubuch 1998). Our study showed no changes in net N mineralization rates when transplanting the soil into a host location, still showing higher net N mineralization rates in the natural forest material, regardless of the site of incubation. This suggests that either the quality of the organic substrate incubated was more important in controlling net N mineralization rates than was temperature (Nadelhoffer et al. 1991) or that the change in temperature (~5°C cooler in the natural forest) was not high enough to cause a change in net N mineralization rates. Conversely, an increase in net N mineralization rate between 9°C and 15°C has been reported (Nadelhoffer et al. 1991; Stottlemyer and Toczydlowski 1999).

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## In situ Microbial Biomass C and N

Other studies have found a change in microbial biomass after a disturbance (Visser et al. 1983; Breland and Hansen 1996; Mummey et al. 2002). For instance, Mummey et al. (2002) found that 20 years after reclamation of a surface mine (reclaimed with stockpiled topsoil), microbial biomass C was still 56% lower in the reclaimed soil than in the undisturbed soil. In our study, the lower microbial biomass C and N found in the reclaimed sites compared to the natural forest site may be a result of lower moisture content or lower dissolved organic nitrogen (DON), which are the only factors positively correlated with microbial C and microbial N each month (Table 3-6). Other field studies have reported a positive relationship between moisture content and microbial biomass C and N (Bohlen et al. 2001; Chen et al. 2003; Hannam and Prescott 2003). Increasing the moisture content to an optimal capacity provides adequate water availability for microbial metabolism, as well as increases diffusion of substrates (Zamen and Chang 2004).

#### Effects of Moisture Manipulation in Reclaimed Soils

Moisture content and soil temperature are the two main environmental factors affecting biological properties (Keith et al. 1997; Koizumi et al. 1999). Because no correlation was found during our field incubation between temperature and mineralization rate or between temperature and microbial biomass, the laboratory experiment study focused on manipulating the moisture content. Samples were incubated at room temperature (20°C), however the interactive influence of temperature and moisture should not be forgotten. For instance, Knoepp and Swank (2002) reported that when temperature is limiting moisture content has little effect on net N mineralization rates. Optimal temperatures for net N mineralization rates have been recorded anywhere between 15 and 35°C (Kowalenko and Cameron 1976). It is possible that conducting the laboratory incubation at a higher temperature would have resulted in higher net N mineralization rates. Net N mineralization rate showed a trend of increasing with an increase in moisture content. The strongest relationship between moisture content and net N mineralization rates was seen in the LFH over secondary material (p=0.064, r=0.39). A positive correlation between moisture content and net N mineralization rates have been reported in the literature (Kowalenko and Cameron, 1976; Carmosini et al. 2003; Zaman and Chang 2004). Optimum net N mineralization rates have been recorded at 10-35% moisture content by weight (Stanford and Epstein 1973).

At the initial (original) moisture content, respiration rates were higher in the natural LFH than in the reclaimed sites (Figure 3-3). Respiration rates have been shown to decrease following a disturbance (Visser et al. 1983; Chang and Trofymow 1996; Lindo and Visser 2003). This has been linked to soil compaction reducing air filled porosity. A decrease in air filled porosity may result in low oxygen availability. Respiration rates of the peat-mineral mix (original moisture content) found in this study (0.009 mg C  $g^{-1} d^{-1}$  or 47 mmol m<sup>-2</sup>) are lower than *in situ* respiration measurements previously recorded in a similar reclaimed peat-mineral mix site at Syncrude (380 mmol m<sup>-2</sup>) (Li et al. 2003). Natural forest respiration rates measured in this experiment  $(0.075 \text{ mg C g}^{-1} \text{ d}^{-1} \text{ or } 1.05 \text{ g C m}^{-2} \text{ d}^{-1})$  were similar to a field experiment examining boreal forest respiration rates found in Alaska (0.85-1.08 g C m<sup>-2</sup> d<sup>-1</sup>) (Ruess et al. 1996). It is difficult to directly compare respiration rates in this study with other studies because of differences in measurement procedures, incubation duration, root inclusion or exclusion, and changes of respiration rates over time and season.

Positive relationships between soil moisture and respiration can be found in the literature (Orchared and Cook 1983; Orchared et al. 1992; Koizumi et al. 1999). Koizumi et al. (1999) has reported an optimal gravimetric water content of 35% in peat fields to reach the highest respiration rate. This is 13.5% greater than the average moisture content of the reclaimed peat-mineral mix when the respiration measurements were taken (Table 3-2). However, our study found a significant increase in respiration rates up to 45% moisture content in peatmineral mix and LFH over peat-mineral mix and 30% moisture content in LFH over secondary. After these moisture contents respiration rates continued to increase, not significantly, from 45% to 60%. These data suggest that the optimal value of soil gravimetric moisture content is between 30% and 45% in the reclaimed treatments.

In the field, a positive correlation between moisture content and microbial C and N was found. In the laboratory, however, only a significant relationship between microbial N and moisture content occurred. One possible explanation for this may be a shift in the microbial community structure with the increase in moisture. Microbial biomass C:N provides a general index of the type of microorganisms present. That is, soil bacteria generally have a C:N ratio of 4:1 to 6:1 while fungi have a higher C:N ratio of 10:1 to 12:1 (Tate 1995). The microbial biomass C:N ratio decreased as the moisture content was increased (Table 3-8). This suggests a change in the microbial community from a fungal dominated community to a bacteria/fungal community (Tate 1995). This is further displayed in the soil respiration rates:microbial biomass C ratios ( $qCO_2$ ). As the fungal:bacteria ratio increases, the  $qCO_2$  decreases possibly due to a higher efficiency of substrate carbon utilization by fungi (Sakamoto and Oba 1994).

No significant differences were found in respiration rates between the peatmineral mix and the reclaimed LFH during the field experiment. However, the laboratory results showed a dramatic difference. At most moisture contents, respiration rates and microbial biomass C were statistically higher in the reclaimed LFH over secondary material than in the peat mineral mix. LFH over peat-mineral mix showed an intermediate value between the peat mineral mix and the LFH over secondary treatments. At the original and 30% moisture content, microbial biomass N was similar in all reclaimed treatments; however, when moisture was increased to 45% and 60% microbial biomass N became statistically higher in the reclaimed LFH treatments compared to the peat-mineral mix. The stronger response of the LFH material may be due to the type of microbial community found in the natural LFH treatments or to a possible difference in organic matter composition.

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## Conclusions

There were no statistical differences between the various treatments using the two amendments, LFH and peat-mineral mix, when mineralization rates and microbial biomass C and N were measured *in situ*. The reciprocal transfer and field incubation into the LFH over peat mineral mix showed a trend of lower net N mineralization rates and lower microbial biomass C and N in the reclaimed sites as compared to the natural forest site. The lack of statistical differences in net N mineralization rates may be due to the large spatial variability, especially at the natural forest. This variability should be further examined using with a stratified sampling strategy.

A correlation analysis performed on the field data found a positive relationship between moisture content and microbial C and N, suggesting that microbial activity may be moisture limited in the reclaimed soils. A laboratory experiment further examining this relationship was conducted by increasing the moisture content of the reclaimed treatments to a soil moisture content equivalent to the natural forest site at the time of sampling. When moisture was added all reclamation materials displayed an increase in mineralization rate, microbial biomass N and respiration rate. Microbial biomass C did not change with moisture. This may be due to a change in microbial community structure with added water. Within the reclaimed LFH sites, the LFH over secondary material showed a higher mineralization rate, respiration rate, and microbial biomass C and N than the LFH over peat-mineral mix. The peat-mineral mix always had the lowest mineralization, microbial biomass C and N and respiration.

Increasing the moisture content of the reclaimed LFH treatments *in situ* may establish a greater amount of available nitrogen providing positive feedback through microbial activity. An experiment manipulating soil moisture *in situ* is required to test this hypothesis.

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# TABLES AND FIGURES

	Bulk density (Mg·m <sup>-3</sup> )	рН	C (mg kg <sup>-1</sup> )	C/N	Soil temperature May 2004 (°C)	Soil Texture
Natural Forest	0.29 (0.02) b	5.32 (0.05) c	170 (4) a	18.3 (0.9) b	7.8 (2.5) b	
Peat-mineral mix	0.81 (0.05) a	5.51 (0.05) b	100 (1) b	21.5 (0.9) a	12.8 (2.5) a	Silty loam
LFH over peat-mineral mix	0.88 (0.02) a	5.86 (0.02) a	50 (7) c	21.5 (0.8) a	13.0 (3.0) a	Silty loam
LFH over secondary	0.88 (0.04) a	5.79 (0.03) a	50 (2) c	21.3 (0.1) a	12.6 (2.5) a	Silty loam

Table 3-1: Selected properties for the topsoil layer (up to 7 cm except for bulk density 0-4 cm) at the natural and reclaimed sites.

NOTE: Values are means (±SE). Different letters in a given column indicate statistical differences at  $\alpha$ = 0.05.

Table 3-2: Moisture content (% weight) for the topsoil layer (up to 7 cm) at the natural and reclaimed sites.

	June	July	May	August
Natural Forest	76.7 (8.8) a	63.5 (8.2) a	187.2 (15.8) a	60.0 (13) a
Peat-mineral mix	39.6 (7.4) b	28.8 (0.2) b	51.7 (9.6) b	21.5 (4.4) b
LFH over peat-mineral mix	22.6 (0.8) b	29.0 (3.6) b	43.5 (3.4) b	24.9 (0.6) b
LFH over secondary	26.1 (2.6) b	20.6 (0.8) b	36.8 (2.5) b	19.6 (0.5) b
P-Value	0.042	0.024	<0.0001	<0.0001

NOTE: Values are means ( $\pm$ SE). Different letters in a given column indicated statistical differences at  $\alpha$ = 0.05.

	Natural forest			LFH over peat-mineral mix			LFH over secondary		
	June /03	July /03	May /04	June /03	July /03	May /04	June /03	July /03	May /04
Natural forest	29.6 (22.2)	13.2 (12.1)	14.8 (32.7)	25.7 (6.1)	30.2 (9.1)	19.7 (13.2)	25.7 (6.1)	24 (18.2)	11.6 (14.0)
LFH over peat-mineral mix	18.6 (0.4)	1.4 (0.4)	-0.1 (1.7)	10.9 (6.2)	3.1 (3.9)	2.4 (2.0)			
LFH over secondary	5.3 (0.4)	1.4 (0.4)	3.6 (0.5)				3.6 (1.6)	19.6 (3.9)	20.2 (8.1)

**Table 3-3:** Net N mineralization rates (mg N kg<sup>-1</sup> 30 days<sup>-1</sup>) during the reciprocal transfer incubation. Incubation location in the top row, material incubated in the first column.

NOTE: Values arc means (±SE). --- indicates reciprocal transfer did not occur between the material and site.

**Table 3-4:** Net N mineralization rates (mg N m<sup>-2</sup> 30 days<sup>-1</sup>) during the LFH over peat-mineral mix incubation. Incubation location in the top row, material incubated in the first column.

	LFH over peat-mineral mix					
	June /03	July /03	May /04			
Natural forest	393 (91) b	463 (138) a	301 (202) a			
LFH over peat-mineral mix	1079 (616) ab	303 (381) a	242 (202) a			
LFH over secondary	2886 (852) a	830 (1090) a	360 (282) a			
Peat-mineral mix	616 (153) ab	9950 (810) a	696 (427) a			
P-value	0.039	0.890	0.705			

NOTE: Values are means (±SE).

	Microbial biomass C (mg kg <sup>-1</sup> )					Microbial biomass N (mg kg <sup>-1</sup> )				
	Initial	Natural forest	LFH over Peat-mineral mix	LFH over secondary	P- value	Initial	Natural forest	LFH over peat-mineral mix	LFH over secondary	P- value
June 2003	3			\						<u> </u>
Natural forest	1787 (145) a	1985 (283) a	2045 (394) a	2002 (180) a	0.8884	99 (15) a	124 (14) a	88 (15) a	99 (34) a	0.377
LFH over peat-mineral mix	873 (133) b	669 (78) b	728 (97) b		0.4778	48 (4) b	29 (4) b	32 (3) b		0.076
LFH over secondary	789 (161) b	622 (68) b	622 (68) b	716 (39) b	0.7016	41 (3) b	32 (5) b	40 (4) b	33 (1) b	0.286
Peat-mineral mix	679 (71) b		650 (108) b		0.3897	25 (3) c		32 (3) b		0.352
P-value	0.029	0.010	0.009	0.012		0.004	0.005	<0.001	0.005	
July 2003	3									
Natural forest	2870 (337) a	2525 (283) a	2434 (422) a	1996 (373) a	0.1121	130 (22) a	114 (33) a	113 (34) a	97 (26) a	0.118
LFH over peat-mineral mix	827 (91) b	704 (38) b	612 (87) b		0.0986	40 (6) b	29 (8) b	27 (7) b		0.055
LFH over secondary	784 (198) b	890 (82) b	662 (80) b	1115 (186) a	0.3087	25 (5) c	36 (4) b	29 (3) b	35 (2) a	0.060
Peat-mineral mix	872 (126) b		650 (108) b		0.3852	23 (3) c		44 (16) b		0.395
P-value	<0.001	0.005	0.003	0.065		0.002	0.036	0.012	0.127	
May 200	4									
Natural forest	1675 (416) a	2125 (394) a	1692 (184) a	1573 (117) a	0.4933	214 (61) a	307 (30) a	269 (26) a	246 (17) a	0.354
LFH over peat-mineral mix	532 (134) b	614 (66) b	611 (31) b		0.7462	99 (19) a	116 (19) b	118 (7) b		0.694
LFH over secondary	435 (94) b	501 (15) b	528 (50) b	801 (59) b	0.0128	104 (11) a	116 (6) b	112 (6) b	1 38 (9) b	0.110
Peat-mineral mix	440 (213) b		321 (37) b		0.5807	71 (24) a		48 (16) c		0.110
P-value	0.025	0.01	<0.001	0.048		0.063	<0.001	<0.001	0.017	

Table 3-5: Microbial biomass C (MBC) and microbial biomass N (MBN) (mg kg<sup>-1</sup>) in the initial samples as well as in samples following the reciprocal transfer experiment. Incubation location is in the top row, material incubated in the first column.

NOTE: Values are means ( $\pm$ SE), --- indicates soil was not incubated in that material. Different letters in a given column indicated statistical differences at  $\alpha$ = 0.05.

	Microbial C	Microbial N	DON	pH	Bulk density	C/N
Moisture content	p=0.0004, r= 0.49	p<0.0001, r= 0.54	N.S.	p<0.0054, r= -0.4	N.S.	N.S.
Microbial C		p<0.0001, r= 0.86	p<0.0077, r= 0.38	N.S.	N.S.	N.S.
Microbial N			p<0.0219, r= 0.33	N.S.	N.S.	N.S.
DON				N.S.	N.S.	N.S.
PH					N.S.	N.S.
Bulk density						p=0.0001, r=0.53
C/N						

 Table 3-6: Correlation analysis for June 2003. Results shown are typical of each month tested.

Note: N.S. = non significant at  $\alpha$ =0.05

Material	Original	30%	45%	60%	Regression P-value
Microbial C (mg kg <sup>-1</sup> )				······	
LFH over secondary	1057 (128) a	1236 (116) a	1159 (101) a	1296 (144) a	0.265
LFH over peat-mineral mix	594 (151) ab	565 (141) b	825 (96) a	648 (140) b	0.490
Peat-mineral mix	294 (30) b	351 (37) b	424 (68) b	387 (156) b	0.393
P-value	0.010	0.030	< 0.001	<0.001	
Microbial N (mg kg <sup>-1</sup> )					
LFH over secondary	53 (23) a	62 (20) a	77 (14) a	92 (20) a	0.027
LFH over peat-mineral mix	28 (7) a	34 (3) a	42 (5) ab	49 (4) ab	0.002
Peat-mineral mix	16(1) a	22 (1) a	27 (1) b	29 (1) b	0.002
P-value	0.238	0.124	0.019	0.022	
Net N mineralization (mg kg <sup>-1</sup> 28day <sup>-1</sup> )					
LFH over secondary	3 (2) a	1 (0) a	6 (2) a	7 (3) a	0.064
LFH over peat-mineral mix	2 (1) a	2 (1) a	2 (1) ab	3 (1) a	0.938
Peat-mineral mix	0 (1) a	0(1)a	l (0) b	3 (2) a	0.113
P-value	0.448	0.499	0.047	0.201	

Table 3-7: Tukey analysis of sites within the same moisture content for microbial C, microbial N and net N mineralization rates from the laboratory incubation experiment.

Note: Values are means ( $\pm$ SE). Different letters in a given column indicated statistical differences at  $\alpha$ = 0.05.

	Moisture Content (%)	Microbial biomass C/N	$q \text{CO}_2 (d^{-1})$
Peat-mineral mix	Original	18.7(1.4) a	0.03 (0.01) a
	30%	16.3 (1.7) a	0.05 (0.01) a
	45%	15.4 (1.9) a	0.06 (0.01)a
	60%	13.2 (5.2) a	0.12 (0.08) a
	P-value	0.550	0.106
LFH over peat-mineral mix	Original	25.7 (12.0) a	0.04 (0.011) a
-	30%	16.4 (3.3) a	0.05 (0.013) a
	45%	19.9 (1.8) a	0.06 (0.007) a
	60%	13.7 (3.2) a	0.07 (0.010) a
	P-value	0.612	0.438
LFH over secondary	Original	35.4 (19.4) a	0.02 (0.003) a
-	30%	26.9 (10.9) a	0.03 (0.002) a
	45%	15.9 (2.2) a	0.04 (0.002) a
	60%	14.9 (2.3) a	0.04 (0.002) a
	P-value	0.637	0.358

Table 3-8: Microbial C/N and  $qCO_2$  for each treatment over various gravimetric moisture contents from the laboratory incubation experiment.

Note: Values are means ( $\pm$ SE). Different letters in a given column indicated statistical differences at  $\alpha$ = 0.05.

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**Figure 3-1**: Net N mineralization rates (mg N kg<sup>-1</sup> 30 days<sup>-1</sup>) during the LFH over peatmineral mix incubation.



Figure 3-2:  $CO_2$  concentrations (mM) for each treatment with altered moisture contents over the six-week lab incubation. Natural forest (original moisture content) is shown on each graph as a reference point.



**Figure 3-3:** Cumulative respiration rates (weeks 3-6) at various moisture contents from the laboratory incubation experiment. Standard error bars are shown. Different letters indicate statistical differences between treatments as compared to the natural LFH, within a given moisture content.



Figure 3-4: The relationship between microbial biomass N and moisture content for each treatment during the laboratory incubation.

# CHAPTER 4. NITROGEN MINERALIZATION IN RECLAIMED FOREST FLOOR AND PEAT-MINERAL MIX TREATMENTS CAPPING LEAN OIL SAND

# Introduction

The Athabasca oil sand region, located in northeastern Alberta, contains one of the largest reserves of hydrocarbons in the world, estimated at almost one trillion barrels of bitumen (Kimball et al. 2000). Current bitumen extraction is focused on shallow oil sand deposits where surface mining is feasible. During the surface mining process, overburden materials (organics, lean oil sand, glacial till, glacio-lacustrine materials, muskeg, mineral soil, etc.) are salvaged and stockpiled for use during the reclamation phase (Fung and Macyk 2000). To date, the total cumulative land disturbance is over 15,000 hectares (Oil Sands Environmental Research Network 2004).

In 2000, Suncor Energy Inc., one of the main oil sand operators in the Athabasca region, established the "Steepbank North Dump" capping study to examine the effectiveness of various reclamation techniques. Nitrogen was considered to be generally limiting in reclaimed mine soils, restricting plant productivity (Bendfeldt et al. 2001). Organic treatments were used on the assumption that there would be an increase total nitrogen content and nitrogen availability in reclaimed soils.

Peat, abundant in the Athabasca region, improves the physical conditions of the soil, increases the moisture-holding capacity of sandy soils and available nutrient supply to plants (iron and phosphorus) (Lucas et al. 1965; Allison 1973). The effectiveness of LFH in reclamation is currently being examined as an alternative to peat. In addition to providing a seedbank/propagules source for native plant species (Granström 1981; Qi and Scarratt 1998), the LFH layer is a store of nutrients and may promote microbial activity in reclaimed soils (Fyles and McGill 1986; Van Cleve et al. 1993). Peat and LFH properties have been compared in undisturbed ecosystems (Clarholm and Rosswall 1980; Devito et al. 1999), however only rarely have they been characterized following placement at reclaimed sites (Lanoue 2003; Li et al. 2003). No one has investigated and compared characteristics of soil reclaimed with the standard reclamation technique (i.e. peat-mineral mix) and the more recent approach, using reclaimed LFH.

The purpose of this paper was to examine and contrast selected physical, chemical and biological properties of soils with these two organic treatments, LFH and peat-mineral mix, and to compare the treatment properties to those of a nearby, undisturbed forest. It was hypothesized that the properties of the reclaimed sites would have a lower net N mineralization and microbial biomass C and N than the natural site. Furthermore, within the reclaimed sites the peatmineral mix was expected to lead to lower microbial biomass and net N mineralization than the reclaimed LFH.

## Site Characteristics and History

The study sites were located approximately 40 km north of Fort McMurray, northeastern Alberta (57° N latitude and 111°W longitude). Long and cold winters (January average temperature is –18.8°C), short and warm summers (July average temperature is +16.8°C), and an average annual precipitation of 455 mm characterize the area. Fort McMurray is located in the Boreal forest region, vegetated by white spruce (*Picea glauca* (Moench) Voss), black spruce (*Picea mariana* (Mill.) BSP), trembling aspen (*Populus tremuloides* Michx.), balsam poplar (*Populus balsamifera* L.), white birch (*Betula papyfrifera* Marsh.) and jack pine (*Pinus banksiana* Lamb.) (Fung and Macyk 2000).

The peat-mineral mix and forest floor (LFH) materials used to cap the lean oil sand overburden were extracted from the Athabasca River valley and deposited directly onto the Steepbank North Dump site in 2000 (AMEC Earth & Environmental *unpubl*.). The LFH was stripped to 20 cm from a west-facing slope dominated by low bush cranberry and aspen, although a small area also included blueberry, aspen, and white spruce. Thickness of the LFH stripped from the pre-mining area was less than 20 cm, thus some of the mineral substrate was mixed into the treatment; the collective treatment is referred to as LFH in this paper. The peat-mineral mix (mesic peat) was salvaged from the base of the same slope and supported coniferous swamp and graminoid marsh (AMEC Earth & Environmental *unpubl.*). The Steepbank North Dump site was planted to annual barley (*Hordeumm vulgare* L.) during the spring of 2000, and then fertilized in 2000 to 24-25-8 (N-P-K) at 300 kg/ha broadcast and 2001 to 2003 to 32-16-5 (N-P-K) at 250 kg/ha. In 2000 the area was hand planted to a mix of white spruce and aspen at a rate of 2076 and 132 stems/ha, respectively (AMEC Earth & Environmental *unpubl.*). It was a western-facing, very strong slope (approximately 22°).

Three reclamation treatment techniques were implemented to cap the lean oil sand overburden (overburden with less than 8% bitumen). In one treatment, a 20 cm depth of peat-mineral mix was placed directly over lean oil sand, while in the second treatment 20 cm of LFH was used. The third treatment included 5 cm LFH over 15 cm of peat-mineral mix. See appendix A-3 for schematic diagram. The LFH (average depth of 5.1 cm) of a nearby, undisturbed upland aspen dominated forest was also included for comparison to the reclaimed sites. **Methods** 

#### **Field Methods**

In July 2003, three 10 m x 10 m plots were randomly selected within each reclaimed and natural site. The LFH over peat-mineral mix where plots were established in May 2004. Two field experiments were conducted in July 2003 and May 2004, both using the buried-bag technique (Eno 1960). In the first experiment duplicate sets of soil samples from each plot: one set was brought to the laboratory as an initial sample, while the other was placed in a polyethylene bag and incubated for one month in the LFH over lean oil sand site before being brought back to the laboratory for analysis. Samples were cored from the organic layer of the natural forest and treatments, up to a depth of 7 cm. In July 2003, four sets of duplicate samples were taken from each plot, while in May 2004, 12 sets were sampled and then composited into four sets prior to analysis, in an attempt to reduce the spatial variability that had been observed at the sites in
2003. Bulk density was determined on four samples per plot using an aluminum corer (7 cm diameter x 4 cm deep).

The second field experiment was a reciprocal transfer experiment between the natural and reclaimed LFH sites. Three sets of four soil cores were extracted from each site; one set was brought to the laboratory for initial analysis, one set was buried *in situ* (in source material), and the final set was incubated into a host material (reclaimed LFH host for the natural site and natural LFH host for the reclaimed material). Again, in May 2004, 12 sets were sampled and then composited into four sets prior to analysis

#### Laboratory Analysis

Soil samples brought back to the laboratory were immediately sieved to 4 mm. Several analyses were conducted to examine moisture content, pH, soil texture, total C and N, net N mineralization, and microbial biomass C and N.

Moisture content was determined by oven drying 5 g of soil at 70°C until a constant weight was obtained. The pH was measured using a glass electrode in a 1:4 field moist soil:0.001M CaCl<sub>2</sub> suspension. Soil texture was determined using the hydrometer method with a pre-treatment of 30%  $H_2O_2$  to remove organic matter (Karla and Maynard 1991). To determine total C and N, samples were air-dried, ground with a ball grinder, and analyzed by Dumas combustion using a CN Elemental Analyzer (NA 1500 Carlo-Erba, Italy). Bulk density was quantified by drying soil cores of a known volume for 48 hours at 70°C (Karla and Maynard 1991).

Net N mineralization rates (mg N kg<sup>-1</sup> soil) were calculated by subtracting the amount of ammonium and nitrate in the field-incubated sample with that in the initial sample. Ammonium and nitrate were extracted using a  $0.5M K_2SO_4$ solution and analyzed with a Technicon Auto Analyzer II (Technicon Industrial Systems, New York, USA).

Microbial biomass C and N were determined from dissolved organic nitrogen (DON) and dissolved organic carbon (DOC) concentrations using the chloroform fumigation-extraction method where microbial biomass C=DOC<sub>fumigated</sub>-DOC<sub>unfumigated</sub>/K<sub>c</sub> and microbial biomass N=DON<sub>fumigated</sub>-DON<sub>unfumigated</sub>/K<sub>n</sub> with a correction factor of K<sub>c</sub> and K<sub>n</sub>=0.45 (Jenkinson et al. 2004). Two soil samples were weighed to 10 g (dry weight basis); one was extracted directly using 0.5M K<sub>2</sub>SO<sub>4</sub> and the other was fumigated with chloroform for 24 hours and then extracted (Horwath and Paul 1994). In June and July 2003, DON concentration was determined using the persulfate oxidation method where the organic nitrogen and NH<sub>4</sub><sup>+</sup> in a sample are oxidized into NO<sub>3</sub><sup>-</sup> and compared to an initial, non-oxidized sample (Cabrera and Beare 1993). DON= NO<sub>3</sub><sup>-</sup> after oxidation<sup>-</sup> (NO<sub>3</sub><sup>-</sup> + NH<sub>4</sub><sup>+</sup>)<sub>initial</sub>. In 2003, dissolved organic carbon (DOC) concentration was analyzed using a Soluble Carbon Analyzer (Astro 2001 II, Texas, USA). In May 2004, DOC and TN (total soluble nitrogen) were quantified directly using a Shimadzu TOC-VTN instrument (Mandel Scientific Company Inc. Ontario, Canada). DON was calculated as DON = TN - (NO<sub>3</sub><sup>-</sup> + NH<sub>4</sub><sup>+</sup>). A more detailed account of the methodology may be found in appendix B.

#### **Results and Discussion**

The LFH layer at the undisturbed forest site contained a significantly (p=0.05) lower bulk density, pH and total C:N ratio and a higher total C than the reclaimed treatments (Table 4-1). Within the reclaimed sites, the LFH over lean oil sand had a significantly higher bulk density and total C:N ratio but a lower pH and total C than the other two treatments. Texture of the mineral component also varied among treatments, from a sand for the LFH over lean oil sand, to a loam for the peat-mineral mix (Table 4-1).

Substantial differences between the natural and reclaimed sites were expected due to the extraction and placement of the treatments (Stolt et al. 2001). Higher compaction, as measured by bulk density, may be attributed to the passing of large equipment (Bradshaw 1983; Grigal 2000). Soil texture in reclaimed mine soils is important for nutrient retention and CeC (Bendfeldt et al. 2001) and soil pH may influence restoration success (Mummey et al. 2002). Higher pH values have been reported in amended soils in other surface mines compared to undisturbed soils, possibly due to mixing the treatment with the alkaline subsurface (Bendfeldt et al. 2001). The higher C:N ratio observed at the LFH over lean site may be due to the presence of hydrocarbons.

Net N mineralization rates were not statistically different (p=0.05) when materials from all sites were incubated at the LFH over lean oil sand site (Figure 4-1). Devito et al. (1999) compared properties of undisturbed upland forest surface soils and peatland soils and found a higher mineralization rate in the forests. The lack of statistical differences between disturbed and undisturbed sites as reported in our study agrees with Carmosini et al. (2003) who rarely found significant differences in net N mineralization between disturbed (logged) and undisturbed sites. On an areal basis, net N mineralization rates tended to be higher in the reclaimed sites due to the higher bulk density and thicker horizons, but were not significantly different (data not shown). The lack of significant difference among sites in this study may be due to the large spatial variability, especially at the natural LFH site.

The reciprocal transfer experiment did not show statistical differences between the two locations of incubation (source and host), except in the May 2004 reciprocal transfer with LFH over lean oil sands, incubated in source and natural LFH (p=0.029) (Table 4-2). This suggests that either quality of soil organic matter may be more important in controlling net N mineralization rates than temperature or the change in soil temperature (~7°C) between the shaded natural forest and the non-shaded reclaimed materials may have been too small to influence net N mineralization rates. However, net N mineralization rates have been found to increase after temperatures were increased above 9°C (Nadelhoffer et al. 1991; Stottlemyer and Toczydlowski 1999). It is unknown why the LFH over lean oil sands showed a significant difference in May 2004.

The natural LFH showed a significantly higher microbial biomass C and N as compared to the reclaimed treatments for both months of study (Table 4-3). There were no statistical differences between the reclaimed treatments. Similarly, no difference in microbial biomass between host and source incubation was found during the reciprocal transfer experiment (data not shown). Wardle (1998) compiled data from several authors examining microbial biomass C and N from

grassland, forest and arable soils. He concluded that the more persistent and less disturbed ecosystems had a higher microbial biomass C and N. Mummey et al. (2002) found that 20 years after reclamation of a surface mine (reclaimed with stockpiled topsoil), microbial biomass C was still 56% lower in the reclaimed soil than in the undisturbed soil.

The higher microbial biomass may occur due to a lower bulk density and pH or a higher total C (Table 4-1). Many studies have reported a positive correlation between microbial biomass C and N and moisture content (e.g. Bohlen et al. 2001; Chen et al. 2003; Hannam and Prescott 2003). An optimal moisture content allows the transport of nutrients to microorganisms, movement of mobile microbes to substrates, and provides water for metabolic processes of microbes and other organisms (Zaman and Chang 2004). The lower pH would favour fungi growth, which produces a greater amount of microbial biomass. As soil compaction increases porosity decreases in turn decreasing aeration, infiltration and available water (Rab 1994; Kozlowski 1999; Williamson and Neilsen 2000). Some studies found that a higher compacted area may have a lower microbial biomass (Kaiser et al. 1991; Šantrůčková et al. 1993) while other studies found no correlation (Chen et al. 2003; Li et al. 2004).

#### Conclusions

This study provided new information on selected soil properties for peatmineral mix and LFH treatments in surface mine reclamation in the Athabasca oil sand region in northeastern Alberta. No significant differences in mineralization rates between the natural forest and reclaimed materials were measured. Also, there was no difference found from the reciprocal transfer experiment suggesting that either the quality of soil organic matter may be more important in controlling net N mineralization rates than temperature, or the change in temperature was not large enough to alter net N mineralization despite the fact that the natural forest was cooler than the reclaimed areas. Microbial biomass C and N were lower in the reclaimed areas likely due to an interaction of lower moisture content and higher bulk density associated with the disturbance. Controlled laboratory experiments manipulating moisture and bulk density of the soil materials should be conducted to further characterize factors controlling N fluxes at these sites.

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**Tables and Figures** 

.

	Bulk density (Mg·m <sup>-3</sup> )	PH	Total C (g· kg <sup>-1</sup> )	C:N ratio	Temperature May 2004 (°C)	Soil Texture
Natural LFH	0.31 (0.02) c	5.3 (0.1) c	170 (4) a	18.3 (0.9) c	7.8 (2.5) b	-
Peat-mineral mix	0.89 (0.05) b	6.7 (0.1) a	80 (20) b	29.1 (0.5) b	14.8 (2.9) a	Loam
LFH over peat-mineral mix	0.89 (0.03) b	6.6 (0.1) a	60 (4) b	28.8 (0.8) b	16.5 (3.1) a	Sandy Loam
LFH over lean oil sand	1.22 (0.05) a	5.9 (0.1) b	40 (1) c	48.0 (4.5) a	15.8 (3.0) a	Sand
P-value	< 0.001	<0.001	0.0009	0.001	< 0.001	

Table 4-1: Physical and chemical characteristics of the natural LFH and reclaimed treatments (organic layer up to 7 cm, bulk density 0-4 cm).

NOTE: Values are means with standard errors in parentheses. Different letters in a given column indicate statistical differences ( $\alpha$ = 0.05) between sites. SAS one-way ANOVA with Tukey analysis.

Table 4-2: Net N mineralization rates (mg N kg<sup>-1</sup>) after 30-day incubation (organic layer up to 7 cm) in original and host materials.

Original material	Host material	Net N mineralizat	ion (mg N kg <sup>-1</sup> )
		July 2003	May 2004
Natural LFH	Natural LFH	13.2 (12.1) a	14.8 (32.8) a
	LFH over peat-mineral mix	-	49.4 (13.2) a
	LFH over lean oil sand	30.5 (16.4) a	13.6 (19.6) a
	P-value	0.598	0.501
LFH over peat-mineral mix	Natural LFH	-	-6.8 (8.3) a
	LFH over peat-mineral mix	-	-4.1 (8.2) a
	P-value		0.8229
LFH over lean oil sand	Natural LFH	10.4 (9.4) a	5.7 (1.9) b
	LFH over lean oil sand	17.2 (5.6) a	14.8 (2.0) a
	P-value	0.259	0.029

NOTE: Values are means with standard errors in parentheses. No samples were extracted from the

LFH over peat-mineral mix in July 2003 (indicated by -). Different letters in a given column indicate statistical differences ( $\alpha$ = 0.05) of original material incubated in different host material sites. SAS one-way ANOVA

	Microbial C (mg kg <sup>-1</sup> )	Microbial N (mg kg <sup>-1</sup> )	Gravimetric Moisture Content (% weight)
July 2003			
Natural LFH	1678 (337) a	130 (22) a	63.5 (8.2) a
LFH over lean oil sand	402 (121) b	15 (6) b	14.0 (1.4) b
Peat-mineral mix	662 (166) b	37 (21) b	34.3 (7.2) b
LFH over peat-mineral mix	N/A	N/A	N/A
P-value	0.0004	0.0085	0.0370
May 2004			
Natural LFH	1675 (42) a	215 (61) a	187.2 (5.8) a
LFH over lean oil sand	174 (37) b	40 (8) b	17.3 (1.2) b
Peat-mineral mix	141 (30) b	59 (20) b	39.9 (7.7) b
LFH over peat-mineral mix	156 (30) b	29 (7) b	15.8 (2.7) b
P-value	0.0019	0.0116	<0.0001

Table 4-3: Microbial biomass C and N (mg kg<sup>-1</sup>) in July 2003 and May 2004 (organic layer up to 7 cm).

NOTE: Values are means with standard errors in parentheses. Different letters in a given column indicate statistical differences ( $\alpha$ = 0.05) between sites. SAS one-way ANOVA with Tukey analysis.



Figure 4-1: Net N mineralization rates (mg N kg<sup>-1</sup>) during a 30-day incubation period at the LFH over lean oil sand site (organic layer up to 7 cm). Error bars represent standard error (n=3).

#### 5.1 Summary

Two main organic treatments are used in reclamation following oil sand mining: LFH horizon material stripped from upland forests, and a mix of peat and mineral material, referred to as peat-mineral mix. The main objectives of this study were to compare the chemical, biological and physical characteristics of these two organic treatments in reclaimed sites, as well as to compare the reclaimed LFH sites to a neighboring, undisturbed boreal forest. Microbial biomass C and N and net N mineralization rates were measured, as well as a series of parameters that has been found to influence them (soil temperature, moisture, pH, and total C and N) in an effort to understand the factors controlling nitrogen availability in these soils.

## 5.1.1 A Comparison of Peat-Mineral Mix and Reclaimed Forest Floor Capping a Pleistocene Clay Deposit

In situ net N mineralization and microbial biomass C and N were measured during an incubation experiment comparing different surface soil treatments: peat mineral mix, LFH over secondary, LFH over peat mineral mix, and LFH in a natural forest. There were few differences between the reclaimed materials. Results showed that the natural forest LFH had a higher mineralization rate than the reclaimed material, however the difference was not statistically significant as a result of high spatial variability within the sites. Microbial biomass nitrogen and carbon were statistically higher in the natural LFH compared to the reclaimed sites. A positive correlation existed between microbial biomass, dissolved organic nitrogen (DON) and moisture content. A laboratory experiment manipulating moisture content resulted in an increase of net N mineralization rates and microbial biomass N with higher moisture content, but microbial biomass C was not affected.

## 5.1.2 Nitrogen Mineralization in Reclaimed Forest Floor and Peat-Mineral Mix Treatments Capping Lean Oil Sand

Results of an *in* situ buried-bag experiment indicated differences between the reclaimed LFH over lean oil sand and the other treatments, specifically a higher bulk

density and C:N ratio, and a lower pH and total C for the reclaimed LFH over lean oil sand. These differences may be attributed to a thicker organic horizon at this site, or to mixing of the lean oil sand with the LFH, as suggested by the observed sandy texture; in turn the mixed-in lean oil sand possibly affected soil properties, resulting for instance in a wider C:N ratio. No significant differences in net N mineralization rates were found between the natural forest and the reclaimed LFH sites. The reciprocal transfer experiment did not alter net N mineralization rates, suggesting that either soil organic matter quality was more important than temperature in controlling net N mineralization rates or alternatively that the change in temperature was not large enough to alter these rates. The reclaimed materials showed a significantly lower microbial C and N compared to the natural forest, likely due to their lower moisture content and higher bulk density. There were no differences in microbial biomass C or N between the reclaimed treatments.

#### 5.2 Project Limitations/Future Research

One of the biggest limitations in this project is the lack of replication of the reclaimed sites, resulting in pseudoreplication. The experimental sites were established before this study began, hence this aspect of the field experimental design cannot be modified. Yet, increasing the replicates of each treatment type would be beneficial. Soil properties were quite variable within the three constructed plots at each site. To reduce the variability found in 2003, more samples were extracted in 2004 and then composited prior to analysis, however, the variability was still quite high. More plots should be constructed within each site and more samples from each plot should be analyzed to better characterize the variability within the sites.

Laboratory experiments do not mimic the complex environment found in the field, and may cause stress to soil microbes. Therefore it is essential that relationships found in the laboratory also be tested in the field. Altering the moisture content of the reclaimed material *in situ* would provide a more accurate account of the response of respiration rates, net N mineralization rates, and microbial biomass compared to the laboratory experiments conducted at controlled room temperature.

Manipulating soil moisture *in situ* may reveal additional differences between the reclaimed treatments.

The short experiment duration, both in the field and the laboratory is also a limitation of this study. Sampling was conducted during the main growing season, however monitoring the sites over the full year and through several years, would provide a more complete account of the nutrient cycling occurring in these soils.

Finally, characterizing the microbial community structure may provide a concrete explanation as to why the microbial biomass C did not respond to moisture additions during the laboratory experiment. In this regard, analysis of the ester-linked phospholipid fatty acids (PLFA) composition of the different soils, or genetic profiling through PCR-DGGE analysis of ribosomal RNA genes (rDNA) should be performed.

#### **5.3 Recommendations**

It is obvious that the natural and reclaimed LFH materials have different properties. The reclaimed soils are clearly moisture limited, as shown by an increase in net N mineralization rates, respiration rates, and microbial biomass N with the addition of water. This study did not pinpoint the exact problem associated with the moisture retaining problem found in the reclaimed soils, however some general suggestions can be provided. Mixing the surface organic layer with the mineral horizon (20 cm at Suncor and 7.8 cm at Syncrude) may have negative effects on soil properties. Ketilson (2005) has found lower moisture contents in the mineral horizon, compared to the forest floor in an upland boreal forest. Mixing these two horizons may alter moisture content and soil organic matter quality (Mummey et al. 2002). The topsoil also has a more active microbial community, higher total N and a higher amount of mineralizable N (Schwenke et al. 1999). These properties are partially lost, or diluted, when the organic material is mixed with the underlying mineral horizon. The higher moisture content observed at the natural forest site may also be partially due to shading by the forest canopy that reduces evaporation losses.

Compaction, due to large equipment passing over the site during reconstruction of the soil, is often found to decrease porosity and aeration, infiltration and available water (Kozlowski 1999; Grigal 2000; Williamson and Neilsen 2000). Placement of organic treatments onto the reclaimed sites with smaller equipment may yield higher microbial biomass and net N mineralization by decreasing this compaction. Another important property influencing soil moisture is soil texture. Soil texture at one of the Suncor sites is sand, which has a low inherent water holding capacity (Hausenbuiller 1985). Using mineral substrates with more favorable textures, such as a loam, may provide a greater water holding capacity in these soils. Finally, over the medium to long-term, increasing soil organic matter content by stimulating vegetation inputs should also decrease bulk density and improve available water capacity (Bradshaw 1983).

The moisture manipulating experiment conducted under laboratory conditions clearly shows that the LFH over secondary material found at Syncrude has more favorable characteristics (i.e. higher microbial activity, microbial biomass C and N, and net N mineralization), compared to the other reclaimed materials. If adding an LFH treatment successfully increases nutrient cycling on reclaimed lands it will have considerable benefits in allowing disturbed areas to become self-sustaining ecosystems more rapidly. Organic matter composition should be further characterized and related to carbon and nitrogen availability within each treatment. Lastly, it should not be forgotten how juvenile the soils in the reclaimed areas are compared to the natural LFH site. Continued monitoring is recommended as they develop further in the future.

#### **5.4 Conclusions**

Results showed that *in situ*, the peat-mineral mix and reclaimed LFH treatments were not significantly different in the properties tested. An increase in moisture content had a positive effect on net N mineralization rates and microbial biomass N of each reclaimed treatment. This suggests that the reclaimed materials are not acting to their full potential under current field conditions. Laboratory results from this study further indicate that under the right circumstances (i.e. increased soil moisture) the reclaimed LFH may have a higher microbial biomass, respiration rate, and net N mineralization rate compared to the reclaimed peat-mineral mix.

#### 5.5 Literature Cited

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### Appendix A – Reclamation material



Figure A-1: Approximate pre-disturbance profile found in the Athabasca oil sand region (modified from Lanoue 2003). Note: not to scale.



Figure A-2: Schematic diagram of three reclamation treatments used at Syncrude Canada Ltd.



Figure A-3: Schematic diagram of three reclamation treatments used at Suncor Energy Inc.

### **Reclamation Terminology** Oil Sands Environmental Research Network

- Composite tails- After the water is removed from fine tails and are composed of 30% fine tails, and 70% tailings sand, mixed with gypsum.
- Fine tails 5 10% of the tailing sand (suspended silt and clays), mainly consisting of water as well as clay and bitumen (in small proportions).
- Lean oil sand- oil sand that is not economically feasible to mine consisting of less then 7-8% bitumen.
- Muskeg- a bog formed by an accumulation of sphagnum moss, leaves, and decayed matter.
- Overburden- surface material above the mineable oil sands including lean oil sand, muskeg, shale and clay tills.
- Tailing sand -granular mineral deposit mainly consisting of sand, as well as a small proportion of silt and clay, left after water and sludge removal during the oil removal process.

#### **Reference:**

Oil Sands Environmental Research Network. 2004. OSERN [online] Available from http://www.osern.rr.ualberta.ca. [modified 2004, cited 17 February 2005].

### **Appendix B. Laboratory Methods**

Sieve soil samples to 4mm for all analysis except when specified

Moisture content

- 1. Weigh a tin (W<sub>1</sub>),. Record weight then zero scale
- 2. Measure soil (about 5g) into tin (W<sub>2</sub>), record exact number to two decimal places.
- 3. Dry to constant weight at 70°C
- 4. Place tins in dessicator for two hours to cool down, then re-weigh  $(W_3)$ .

oven dry (g) =  $(W_3)-(W_1)$ 

moisture (g) =  $(W_2)$ - $(W_3)$ 

5. Calculate the moisture content (% by weight)= moisture/oven dry)x 100

Initial extraction for DON, DOC, NH<sub>4</sub><sup>+</sup> and NO<sub>3</sub><sup>-</sup>

- 1. Weigh out 10 g dry weight of soil in a nalgene bottle or a whirl pack shaker bag
- 2. Add 100 ml of 0.5M K<sub>2</sub>SO<sub>4</sub>
- 3. Put in shaker on high for 1 hour
- 4. Let the mixture settle (about 15mins)
- 5. Pre leach a quartered Whatman #42 paper (or P3 paper- 15 cm diameter) that is sitting in a small funnel. Preleach with K<sub>2</sub>SO<sub>4</sub> Let dry then pour the liquid content of the nalgene bottle/whirl pack bag in the Whatman paper lining the funnel
- 6. Let sit until liquid filters through the paper
- 7. Analyze for  $NH_4^+$  and  $NO_3$ , DON and DOC
- 8. Repeat with buried samples and analyze for  $NH_4^+$  and  $NO_3^-$
- 9. Net N mineralization =  $(NH_4^+ + NO_3^-)_{buried} (NH_4^+ + NO_3^-)_{initial}$

Fumigation extraction

- 1. Take 10 g of soil (dry weight) and put it into a 50 ml beaker
- 2. Label the beakers with a lead pencil
- 3. Line the dessicator with moist paper towels
- 4. Put beakers in the dessicator along with a beaker containing chloroform and antibumping agents
- 5. Cover the dessicator with the greased lid
- 6. Use the vacuum pump to boil the choloroform
- 7. Make sure the dessicator is closed and cover with a garbage bag for 24 hours
- 8. After 24 hours take off the garbage bag and take out the paper towel and the chloroform
- 9. Hook the pump to the dessicator and pump five or six times to get all the chloroform out of the soil
- 10. Extract soil as mentioned above
- 11. Analyze for DON and DOC

pН

- 1. Weigh out 5 g of air dry soil in 50ml beakers
- 2. Add 20ml of 0.001 CaCl<sub>2</sub> to the soil in the beaker and mix carefully with a stirring stick
- 3. Let stand for one hour, stir four times in first 30 mins.
- 4. Swirl the suspension in the beaker and insert the electrodes into the suspension. Place the electrodes directly in the sedimented soil.
- 5. Read pH and record
- 6. Between pH readings, rinse the electrodes with distilled water

### Bulk Density

Do not sieve.

- 1. Know the volume of soil extracted (by corer)
- 2. Remove all plant roots, wood chunks etc.
- 3. Dry in oven at 70°C for 48 hours
- 4. Weigh

Loss On Ignition (LOI)

- 1. Dry samples at 70°C for 48 hours
- 2. Grind samples using a ball grinder
- 3. Place 1-2 g of soil (record exact amount of soil) in a tin and heat at 70°C for 24 hours
- 4. Place in dessicator to dry, then reweigh
- 5. Heat crucibles in muffle furnace at 550°C for two hours. Let cool for two hours in furnace then two hours in dessicator.
- 6. Reweigh
- 7. Calculate LOI % = ((weight<sub>70</sub>-weight<sub>550</sub>)/weight<sub>550</sub>)100%

Total N and Total C

- 1. Dry samples at 70°C for 48 hours
- 2. Grind samples
- 3. Place 1-2 g of soil in vials
- 4. Send to laboratory for the Dumas combustion method

DON- Alkaline Persulfate oxidation method

- 1. Take solutions from  $0.5M K_2SO_4$
- 2. Make the reagent (0.375M NaOH): dissolve 15g NaOH in 600ml of dionized water, add 52g Persulfate, 21.2 g of boric acid ->volume to 1L
- 3. Pipette 5ml of the standards or samples into a labeled culture tube then add 5ml of reagent
- 4. Cover quickly using Teflon-lined screw caps
- 5. Put the tubes on a rack and put the rack and tubes in a steal sink
- 6. Wrap with an autoclave bag
- 7. Place in autoclave for 45mins at 121°C, with a drying time of 15 minutes
- 8. Analyze for nitrate
- 9. Calculate DON as NO<sub>3</sub> oxidized NO<sub>3</sub> initial

### Particle Size Distribution-

Pre-treatment:

- 1. Transfer 50 g of soil to a 1L beaker
- 2. Weigh the beaker and the soil
- 3. In the fume hood add 20ml H<sub>2</sub>O<sub>2</sub> (30%). Keep adding until the violent reaction stops
- 4. Wait until it stops frothing, wash the sides with distilled water
- 5. Put it on hotplate (around 100°C)
- 6. If you have excess water left in the beaker after boiling, you want to set the sample to sit overnight. In the morning siphon the excess liquid out of the beaker and then place it in the oven.
- 7. Put in the oven at 105°C overnight
- 8. Break down the particles with a rubber pestle and mortar
- 9. Sieve to 2mm and oven dry again at 105°C overnight
- 10. Weigh at the end
- 11. Ready for the particle size distribution

### Hydrometer method

- 1. Transfer 50g (or what ever is left from your pre-treatment) to a metal cup
- 2. Add 400ml of water
- 3. Add 50 ml Calgon solution
- 4. Stir on milkshake machine for 15 minutes
- 5. Transfer soil solution into cylinder. Add water to 1L mark
- 6. Make a blank consisting of 50 ml Calgon and 950 ml water
- 7. Insert plunger; plunge to close to the bottom really fast for 2 minutes
- 8. Put the hydrometer reading into the solution after 30 sec, 1 min, 2 hours, and 24 hours.
- 9. Adjust for temperature

### **Reference:**

Cabrera, M.L., and Beare, M.H. 1993. Alkaline persulfate oxidation for determining total nitrogen in microbial biomass extracts. Soil Science Society of America Journal **57**: 1007-1012.

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### Experiment comparing 0.5M K<sub>2</sub>SO<sub>4</sub> and 2M KCl

 $0.5M \text{ K}_2\text{SO}_4$  and 2M KCl are two neutral salt solutions that are used to extract  $\text{NH}_4^+$  and  $\text{NO}_3^-$  from soils. KCl is generally used when differentiating between  $\text{NO}_3^-$  and  $\text{NO}_2^-$  or when wanting to store samples in the refrigerator for a few months without

precipitation, as typically occurs with  $K_2SO_4$ . On the other hand,  $K_2SO_4$  is typically used as an extractant to determine microbial biomass C and N. Due to the low amount of  $NO_2^-$  in soils, and rapid analyses of the samples after a study was designed to compare the two extractants 5M  $K_2SO_4$  and 2M KCl to see if  $K_2SO_4$  could be used to determine available  $NH_4^+$  and  $NO_3^-$ .

Methodology

Initial extraction for NH4<sup>+</sup> and NO3<sup>-</sup>

- 1. Weigh out two 5 g samples (dry weight) from the same subsample placed in a nalgene bottle
- 2. Add 50ml of 0.5M K<sub>2</sub>SO<sub>4</sub> to one sample bottle and 50 ml 2M KCl to the other bottle
- 3. Put on shaker on high for 1 hour. Let the mixture settle (about 15 mins)
- 4. Pre leach a quartered Whatman #42 paper (or P3 paper- 15 cm diameter) that is sitting in a small funnel. Preleach with K<sub>2</sub>SO<sub>4</sub> (for K<sub>2</sub>SO<sub>4</sub> extracts) or KCl (for KCl extracts). Let dry then pour the solution from nalgene bottle in the filter paper lined funnel.
- 5. Let sit until liquid filters through the paper
- 6. Analyze for  $NH_4^+$  and  $NO_3^-$

Results (n=12)



Figure B-1: Correlation analysis between KCl and K<sub>2</sub>SO<sub>4</sub> examining NH<sub>4</sub><sup>+</sup> and NO<sub>3</sub><sup>+</sup>

### Conclusion

Results from the 0.5M  $K_2SO_4$  and 2M KCl extractions are very well therefore  $K_2SO_4$  was used to determine available  $NH_4^+$  and  $NO_3^-$  as well as microbial biomass C/N.

name	K2S0	4 fum	KClu	unfum
	N03 (ppm)	NH4 (ppm)	N03 (ppm)	NH4 (ppm)
L/S 1i	0.67	2.28	0.73	0.62
L/S 1ii	0.68	2.01	0.81	0.57
L/S 1iii	1.34	2.07	1.9	0.54
L/S 1iv	0.5	1.62	0.8	0.6
L/P 1i	0.42	2.16	0.53	0.73
L/P 1ii	0.36	2.12	0.48	0.58
L/P 1iii	0.23	1.27	0.31	0.49
L/P 1iv	0.28	1.5	0.33	0.63
PM 1i	0.28	0.55	0.2	0.37
PM 1ii	0.3	1.67	0.34	0.52
PM 1iii	1.05	1.09	1.78	0.48
PM 1iv	0.39	0.59	0.36	0.42
NLFH 1i	0.53	2.57	0.29	0.81
NLFH 1ii	0.55	9.25	0.35	3.71
NLFH 1iii	0.52	5.86	0.36	2
NLFH 1iv	0.58	8.4	0.29	2 <u>.81</u>
L/S 2i	1.11	1.75	1.21	0.64
L/S 2ii	0.75	1.9	1.19	0.68
L/S 2iii	0.79	1.74	0.74	0.48
L/S 2iv	0.36	1.35	0.27	0.49
L/P 2i	0.39	1.53	0.45	0.36
L/P 2ii	3.1	3.65	4.52	0.62
L/P 2iii	0.28	1.97	0.49	0.57
L/P 2iv	0.33	2.01	0.4	0.43
PM 2i	0.26	1.06	0.43	0.37
PM 2ii	0.45	0.95	0.76	0.38
PM 2iii	0.42	1.02	0.49	0.32
PM 2iv	0.35	1.11	0.47	0.33
NLFH 2i	0.22	5.2	0.32	2.2
NLFH 2ii	0.23	10.7	0.24	4.89
NLFH 2iii	0.28	9.91	0.29	3.69
NLFH 2iv	2.75	1.06	0.24	1.53
L/S 3i	1.21	3.9	2.6	0.56
L/S 3ii	0.41	1.81	0.53	0.45
L/S 3iii	0.32	2.2	0.5	0.45
L/S 3iv	1.47	3.51	2.71	0.56
L/P 3i	0.27	2.36	0.54	0.69
L/P 3ii	0.45	4.33	0.44	0.82
L/P 3iii	0.29	2.07	0.46	0.8
L/P 3iv	0.62	1.84	0.33	0.83

#### Raw data

name	K2S0	4 fum	KCI unfum							
	N03 (ppm)	NH4 (ppm)	N03 (ppm)	NH4 (ppm)						
PM 3i	0.42	0.89	0.51	0.34						
PM 3ii	0.3	1.24	0.38	0.4						
PM 3iii	0.28	1.12	0.38	0.36						
PM 3iv	0.3	1.95	0.48	0.39						
NLFH 3i	0.27	6.62	0.24	1.61						
NLFH 3ii	0.34	10.06	0.32	1.37						
NLFH 3iii	0.16	7.16	0.31	3.88						
NLFH 3iv	0.17	6.73	0.26	2.43						

#### MICROBIAL BIOMASS C/N ratios

Microbial biomass C/N were different between years. June and July 2003 microbial biomass C/N values were 17 to 38 in the initial samples, which is representative of each sampling analysis (Table 6). In May 2004, microbial biomass ranged between 4 and 8. None of the microbial biomass C/N ratios were significantly different, however in May 2004, the more reliable date of sampling, microbial biomass C/N was highest in the natural forest LFH material.

Microbial biomass C/N values for the initial microbial biomass

	June 2003	July 2003	May 2004
Natural LFH	18.6 (1.9)	22.5 (1.7)	8.2 (1.4)
LFH over peat-mineral mix	28.9 (5.1)	37.8 (6.7)	5.2 (0.8)
LFH over secondary	19.0 (3.5)	17.7 (2.5)	4.1 (0.5)
Peat mineral mix	29 (15.0)	21.1 (1.6)	5.9 (1.5)
P-value	0.162	0.064	0.140

Note: Values are means  $(\pm SE)$ .

The large difference in microbial biomass N, and therefore microbial biomass C/N between 2003 and 2004 is probably due to the change in laboratory methodology. In 2003, the persulfate oxidation method was used, while in 2004 a direct nitrogen measurement was taken. The persulfate may not have oxidized the sample completely, providing a low nitrogen value, thus microbial biomass N and microbial biomass C/N should not be compared between years.



Figure B-2: Laboratory analysis for field experiment

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Figure B-3: Methods for laboratory experiment

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### **APPENDIX C - Syncrude**

### GPS coordinates of sampling plots (square):

Material	Plot	Corner 1	Corner 2	Corner 3	Corner 4
LFH over secondary	1	57° 06.065'N	57° 06.063'N	57° 06.057'N	57°06.058'N
		111°40.258'W	111°40.248'W	111°40.249'W	111°40.260'W
	2	57° 06.049'N	57° 06.044'N	57° 06.046'N	57°06.050'N
		111°40.271'W	111°40.273'W	111°40.283'W	111°40.279'W
	3	57° 06.061'N	57° 06.060'N	57° 06.055'N	57°06.055'N
		111°40.273'W	111°40.265'W	111°40.268'W	111°40.276'W
Peat-mineral mix	1	57° 06.165'N	57° 06.167'N	57° 06.171'N	57° 06.171'N
		<u>111°38.926'W</u>	111°38.937'W	111°38.935'W	111°38.926'W
	2	57° 06.159'N	57° 06.159'N	57° 06.164'N	57° 06.164'N
		111°38.920'W	<u>111°</u> 38.928'W	111°38.926'W	111°38.918'W
	3	57° 06.172'N	57° 06.172'N	57° 06.179'N	57° 06.178'N
		111°38.924'W	_111°38.935'W	111°38.934'W	111°38.923'W
LFH over peat-mineral	1	57°06.137'N	57°06.136'N	57°06.141'N	57°06.141'N
mix		111°38.995'W	111°38.006'W	111°38.005'W	111°38.996'W
	2	57°06.138'N	57°06.139'N	57°06.142'N	57°06.143'N
		111°38.978'W	111°38.982'W	111°38.981'W	111°38.973'W
	3	57°06.134'N	57°06.135'N	57°06.140'N	57°06.140'N
		111°38.937'W	111°38.948'W	111°38.948'W	111°38.936'W
Natural forest	1	57°06.4570'N	57°06.565'N	57°06.558'N	57°06.563'N
		111°41.715'W	111°41.722'W	111°41.723'W	111°41.728'W
	2	57°06.5570'N	57°06.556'N	57°06.560'N	57°06.556'N
		111°41.703'W	_111°41.700'W	111°41.714'W	111°41.712`W
	3	57°06.4577'N	57°06.581'N	57°06.573'N	57°06.579'N
		111°41.769'W	111°41.759'W	111°41.766`W	111°41.756'W

#### Abbreviations

Natural Forest LFH – NLFH LFH over peat-mineral mix – LP LFH over secondary – LS Peat-mineral mix – PM

C:N		21.42	20.76	22.30	20.42	20.13	22.20	21.84	20.41	21.55	20.93	21.56	22.23	19.69	26.13	20.54	20.54	21.76	19.81	20.60	19.32	21.64	21.25	22.84	24.22	21.28	18.40	20.24	20.21	19.46
Soluble Organic Nitrogen	(mg/kg)	1.07	0.97	1.32	1.03	3.71	1.44	1.06	1.00	2.26	4.00	1.59	0.93	1.06	0.88	0.71	0.75	0.62	0.82	2.46	1.00	0.62	0.82	2.46	1.00	0.59	0.77	1.17	0.69	0.77
Soluble Organic Carbon	(mg/kg)	24.80	24.40	26.80	26.80	121.20	27.20	18.00	28.80	34.00	91.60	31.60	29.60	26.80	27.60	20.80	32.40	18.00	29.20	41.60	20.00	20.00	21.20	22.40	23.60	39.20	42.00	42.80	42.00	29.20
Moisture content	(% weight)	26.33	21.45	30.51	21.03	22.93	21.43	17.08	18.90	26.88	17.78	20.71	23.30	25.17	26.47	16.89	23.43	20.95	33.50	27.92	25.19	23.72	31.71	39.72	35.62	49.53	52.74	59.22	51.62	40.93
Net N mineralization	(mg/kg)	42.10	39.40	45.20	00.6	14.70	21.00	19.90	9.70	-7.90	13.00	36.60	44.20	25.20	11.70	-0.50	13.30	9.00	23.00	0.40	50.90	-11.20	5.60	5.70	-2.30	3.00	4.20	18.10	2.90	0.40
Microbial N	(mg/kg)	36.67	49.78	57.56	40.44	13.78	44.44	47.11	37.78	48.00	18.89	70.67	30.67	50.89	55.78	34.89	31.56	25.11	36.22	73.78	43.56	42.00	60.67	47.33	74.22	10.00	43.78	27.11	8.44	46.00
Microbial C	(mg/kg)	746.67	604.44	1004.44	568.89	71.11	746.67	791.11	577.78	2062.22	480.00	1111.11	702.22	951.11	1057.78	675.56	586.67	595.56	551.11	871.11	693.33	977.78	1137.78	995.56	1377.78	631.11	1031.11	737.78	533.33	666.67
pq	(g/cm3)	0.79	0.94	0.98	0.75	0.73	0.65	0.78	0.88	0.85	0.76	0.64	0.81	1.30	0.81	0.93	0.82	0.79	0.88	0.76	0.76	0.73	0.80	0.64	0.67	0.85	0.82	0.88	0.83	0.69
Чd		5.80	5.70	5.80	5.60	5.65	5.90	6.80	5.73	5.50	5.60	5.96	6.02	6.00	5.70	5.88	5.50	5.78	5.98	5.81	6.01	5.87	5,99	5.99	5.08	4.70	5.50	5.60	4.65	5.82
name		L/S 1i	L/S 1ii	L/S 1iii	L/S 1iv	L/S 2i	L/S 2ii	L/S 2iii	L/S 2iv	L/S 3i	L/S 3ii	L/S 3iii	L/S 3iv	L/P 1i	L/P 11	UP 111	L/P 1iv	L/P 2i	L/P 2ii	UP 2iii	L/P 2iv	L/P 3i	L/P 3ii	L/P 3iii	L/P 3iv	PM 1i	PM 1ii	PM 1iii	PM 1iv	PM 2i

name	ph	bd	Microbial C	Microbial N	Net N mineralization	Moisture content	Soluble Organic Carbon	on Soluble Organic Nitrogen		
		(g/cm3)	(mg/kg)	(mg/kg)	(mg/kg)	(% weight)	(mg/kg)	(mg/kg)		
PM 2ii	5.53	0.58	444.44	26.89	1.20	37.63	46.80	0.85	20.47	
PM 2iii	5.78	0.61	284.44	22.67	7.60	33.17	51.20	0.89	21.33	
PM 2iv	5.45	0.72	764.44	24.44	1.60	36.75	35.60	0.73	22.30	
PM 3i	<b>6</b> .96	1.10	942.22	31.11	3.30	19.60	23.60	2.26	23.05	
PM 3ii	5.99	0.92	782.22	21.33	8.70	35.82	28.00	4.00	26.54	
PM 3iii	5.67	0.80	480.00	8.44	3.00	22.55	30.40	1.59	24.05	
PM 3iv	5.49	0.86	853.33	24.44	7.40	35.40	31.20	0.93	18.62	
NLFH 1i	4.80	0.19	1475.56	60.67	63.60	29.35	58.80	1.56	19.22	
NLFH 1ii	5.32	0.33	2204.44	120.67	9.80	106.99	41.60	3.04	17.98	
NLFH 1iii	5.10	0.30	2186.67	108.89	4.20	53.64	44.40	2.28	22.05	
NLFH 1iv	5.50	0.55	2195.56	128.89	48.10	61.17	46.80	2.58	20.85	
NLFH 2i	5.31	0.40	648.89	115.56	60.00	93.00	39.60	0.81	21.84	
NLFH 2ii	5.36	0.36	1546.67	92.67	0.50	120.81	36.80	1.72	15.26	
NLFH 2iii	5.59	0.27	2186.67	126.00	11.00	88.68	44.00	3.35	15.65	
NLFH 2iv	5.72	0.18	2897.78	151.56	29.70	64.38	40.40	2.83	15.52	
NLFH 3i	5.30	0.21	577.78	18.22	33.10	74.31	31.20	0.72	17.51	
NLFH 3ii	5.34	0.17	933.33	33.56	0.30	108.43	38.80	0.77	17.51	
NLFH 3iii	5.74	0.20	1635.56	84.00	55.60	106.90	39.60	1.57	17.87	
NLFH 3iv	5.58	0.21	2951.11	146.22	39.10	76.10	47.20	1.77	18.52	

an C:N		21.42	20.76	22.30	20.42	21.76	19.81	20.60	19.32	23.05	26.54	24.05	18.62	20.13	22.20	21.84	20.41	21.64	21.25	22.84	24.22	19.22	17.98	22.05	20.85	21.55	20.93	21.56	22.23	21.28
Soluble Organic Nitrog	(mg/kg)	1.26	0.95	0.62	0.85	0.89	0.85	0.72	0.73	1.10	0.81	0.76	0.73	1.02	0.56	0.81	0.91	0.78	1.38	0.94	0.98	0.54	0.57	0.72	0.75	1.20	1.11	0.82	0.83	
Soluble Organic Carbon	(mg/kg)	21.20	20.20	16.60	21.20	19.20	20.40	21.20	18.40	107.60	26.60	23.60	18.40	21.40	21.40	22.80	29.20	17.00	26.40	22.00	19.00	20.00	15.80	17.60	21.20	27.40	48.20	29.40	27.00	01 00
Moisture content	(% weight)	19.87	23.26	16.57	21.89	25.13	17.51	23.04	22.69	18.27	26.12	15.37	17.23	26.03	28.14	21.43	28.54	26.44	23.09	26.56	22.93	48.61	27.20	25.98	43.10	33.17	23.45	30.66	20.40	7145
Net N mineralization	(mg/kg)	14.50	24.10	35.80	9.10	-30.10	-19.90	21.80	0.50	13.50	6.80	-4.20	1.90	-9.80	1.70	4.40	6.20	-21.70	-3.60	19.90	-2.70	16.30	5.60	2.40	18.10	1.40	-5.70	9.90	-1.60	00 1 1
Microbial N	(mg/kg)	51.56	52.44	30.89	55.11	53.56	60.89	39.11	48.22	15.33	54.00	32.44	31.11	45.11	21.78	65.33	44.44	66.22	30.00	51.56	46.22	22.44	31.78	28.89	26.67	29.33	34.67	18.89	36.22	00.00
Microbial C	(mg/kg)	886.67	724.44	462.22	922.22	591.11	1515.56	1086.67	1360.00	0.00	862.22	306.67	686.67	686.44	333.33	1108.89	1235.56	982.22	775.56	1202.22	940.00	717.78	684.44	502.22	753.33	619.33	1617.78	555.56	1102.22	4040 00
pq	(g/cm3)	0.79	0.94	0.98	0.75	0.79	0.88	0.76	0.76	1.10	0.92	0.80	0.86	0.73	0.65	0.78	0.88	0.73	0.80	0.64	0.67	0.19	0.33	0.30	0.55	0.85	0.76	0.64	0.81	0.05
hq		5.81	5.54	5.34	5.90	6.08	5.48	5.51	5.26	6.12	5.76	6.24	6.05	6.10	5.55	6.07	5.87	5.70	5.60	5.66	5.73	5.89	5.71	6.38	5.66	5.70	5.55	5.75	5.65	с 72
name		L/S 1i	L/S 1ii	L/S 1iii	L/S 1iv	L/S 2i	L/S 2ii	L/S 2iii	L/S 2iv	L/S 3i	L/S 3ii	L/S 3iii	L/S 3iv	L/P 1i	L/P 1ii	L/P 1iii	L/P 1iv	L/P 2i	L/P 2ii	L/P 2iii	L/P 2iv	L/P 3i	L/P 3ii	L/P 3iii	L/P 3iv	PM 1i	PM 1ii	PM 1III	PM 1iv	10 M O

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# July 2004

name	ph	bd	Microbial C	Microbial N	Net N mineralization	Moisture content	Soluble Organic Carbon	on Soluble Organic Nitrogen		
		(g/cm3)	(mg/kg)	(mg/kg)	(mg/kg)	(% weight)	(mg/kg)	(mg/kg)		
PM 2ii	5.27	0.82	1257.78	6.22	-14.10	21.52	33.60	0.77	18.40	
PM 2iii	5.30	0.88	722.22	10.89	-1.20	34.72	25.00	0.88	20.24	
PM 2iv	5.17	0.83	880.00	23.56	12.10	37.13	30.60	1.04	20.21	
PM 3i	5.00	0.40	875.56	18.89	18.00	38.31	53.80	1.19	21.84	
PM 3ii	6.18	0.36	515.56	25.78	26.60	21.65	30.20	0.75	15.26	
PM 3iii	5.26	0.27	508.89	15.78	20.40	31.71	25.00	0.62	15.65	
PM 3iv	6.03	0.18	597.78	24.22	22.00	18.65	24.20	0.82	15.52	
NLFH 1i	5.52	1.30	1930.67	122.67	15.90	60.32	48.20	1.74	19.69	
NLFH 1ii	5.90	0.81	2477.78	107.56	40.70	38.15	35.20	1.17	26.13	
NLFH 1iii	4.95	0.93	3191.11	99.33	-6.10	48.05	49.00	1.47	20.54	
NLFH 1iv	5.08	0.82	1411.11	72.89	140.00	69.12	25.60	1.02	20.54	
NLFH 2i	5.31	0.69	3055.56	112.89	60.40	61.05	22.40	1.26	19.46	
NLFH 2ii	5.36	0.58	4344.44	154.89	10.20	94.82	35.80	2.23	20.47	
NLFH 2iii	5.34	0.61	5406.67	276.22	48.60	91.67	55.80	4.92	21.33	
NLFH 2iv	5.50	0.72	2170.00	146.22	-17.00	72.12	44.40	5.28	22.30	
NLFH 3i	5.17	0.21	3591.11	121.78	-52.50	47.84	66.40	2.71	17.51	
NLFH 3ii	4.77	0.17	3784.44	126.89	-52.70	77.19	65.80	4.13	17.51	
NLFH 3iii	4.77	0.20	2417.78	104.89	119.20	52.81	25.00	1.71	17.87	
NLFH 3iv	4.93	0.21	2057.78	112.00	56.20	48.48	67.00	1.86	18.52	

# May 2004

name	ph	bd	Microbial C	Microbial N	Net N mineralization	Moisture content	Soluble Organic Carbon	Soluble Organic Nitrogen	C:N	
		(g/cm3)	(mg/kg)	(mg/kg)	(mg/kg)	(% weight)	(mg/kg)	(mg/kg)		
L/S 1i	5.94	0.79	331.78	97.78	10.80	18.85	12.51	1.45	21.42	
L/S 1ii	5.90	0.94	287.33	94.44	1.40	35.76	13.92	1.64	20.76	
L/S 1iii	5.75	0.98	432.89	104.44	8.10	41.90	11.81	1.58	22.30	
L/S 1iv	5.87	0.75	435.56	118.67	3.90	36.50	16.52	2.09	20.42	
L/S 2i	5.98	0.79	303.78	80.22	-1.20	34.97	15.38	2.00	21.76	
L/S 2ii	5.28	0.88	249.78	74.67	-4.70	35.62	20.16	1.76	19.81	
L/S 2iii	5.62	0.76	410.00	104.44	1.00	37.50	12.98	2.11	20.60	
L/S 2iv	5.70	0.76	282.89	75.56	0.30	34.04	13.80	2.14	19.32	
L/S 3i	6.11	1.10	464.67	100.89	0.90	36.73	18.36	1.95	23.05	
L/S 3ii	5.80	0.92	700.89	125.33	3.00	49.15	14.28	1.97	26.54	
L/S 3iii	5.90	0.80	657.56	124.89	3.50	38.19	10.31	1.47	24.05	
L/S 3iv	5.66	0.86	657.56	140.44	5.00	42.66	14.76	1.75	18.62	
L/P 1i	6.18	0.73	190.00	45.56	7.50	23.03	16.37	1.26	20.13	
L/P 1ii	5.90	0.65	301.78	76.00	-2.20	42.89	18.66	1.70	22.20	
L/P 1iii	6.04	0.78	272.44	62.89	-4.40	40.33	18.09	1.61	21.84	
L/P 1iv	6.34	0.88	292.00	79.78	-3.40	43.75	16.44	1.77	20.41	
L/P 2i	6.36	0.73	725.11	137.33	-1.90	43.99	23.13	1.89	21.64	
L/P 2ii	6.24	0.80	683.78	128.89	6.00	44.04	13.79	1.67	21.25	
L/P 2iii	6.00	0.64	599.11	115.11	0.60	42.06	14.73	1.69	22.84	
L/P 2iv	5.95	0.67	686.22	148.00	1.80	45.36	18.30	1.65	24.22	
L/P 3i	5.08	0.19	586.67	89.11	5.80	42.90	13.71	1.67	19.22	
L/P 3ii	5.47	0.33	498.44	70.22	7.90	60.96	15.09	1.60	17.98	
L/P 3iii	5.77	0.30	912.44	147.33	1.90	53.02	15.29	1.71	22.05	
L/P 3iv	5.18	0.55	636.67	90.22	9.70	39.82	14.37	2.04	20.85	
PM 1i	5.43	0.85	1.33	38.44	20.60	26.68	16.32	1.53	21.55	
PM 1ii	5.62	0.76	160.89	49.11	9.10	53.61	21.50	1.78	20.93	
PM 1iii	5.23	0.64	272.89	79.11	20.10	49.46	30.14	2.82	21.56	

# May 2004

name	ph	bd	Microbial C	Microbial N	Net N mineralization	Moisture content	Soluable Organic Carbon	Soluable Organic Nitrogen	C:N
		(g/cm3)	(mg/kg)	(mg/kg)	(mg/kg)	(% weight)	(mg/kg)	(mg/kg)	
PM 1iv	5.62	0.81	194.44	51.11	1.70	46.20	22.47	2.00	22.23
PM 2i	4.96	0.85	877.11	107.56	1.60	78.83	26.16	1.94	21.28
PM 2ii	5.16	0.82	746.22	93.56	8.60	67.95	25.40	1.90	18.40
PM 2iii	5.12	0.88	684.22	95.78	3.10	64.97	22.20	1.79	20.24
PM 2iv	5.90	0.83	1118.67	171.11	-1.90	70.63	32.40	2.10	20.21
PM 3i	5.52	0.40	206.22	44.00	3.06	47.45	32.66	2.18	21.84
PM 3ii	5.32	0.36	2.22	22.22	-0.80	23.89	32.21	1.11	15.26
PM 3iii	5.23	0.27	671.78	20.22	0.40	40.22	27.98	1.70	15.65
PM 3iv	5.88	0.18	346.44	75.11	4.00	50.79	30.09	1.79	15.52
NLFH 1i	5.44	1.30	1730.00	99.33	-7.12	196.40	62.73	2.52	19.69
NLFH 1ii	5.50	0.81	1950.00	237.11	-1.22	213.82	55.80	6.15	26.13
NLFH 1iii	5.29	0.93	1730.00	179.33	-6.06	201.60	51.30	3.56	20.54
NLFH 1iv	5.16	0.82	1330.00	276.89	-0.76	139.45	23.93	1.23	20.54
NLFH 2i	5.41	0.69	3914.00	419.78	-3.10	153.63	81.05	4.66	19.46
NLFH 2ii	5.48	0.58	2308.67	376.89	-37.10	154.10	103.63	3.92	20.47
NLFH 2iii	5.44	0.61	2450.67	340.67	121.60	176.96	48.13	2.77	21.33
NLFH 2iv	5.36	0.72	1349.11	170.67	2.40	154.21	33.23	2.72	22.30
NLFH 3i	5.19	0.21	1840.00	65.33	69.80	181.65	32.10	2.95	17.51
NLFH 3ii	5.34	0.17	1319.33	96.44	62.00	159.00	25.93	2.42	17.51
NLFH 3iii	5.33	0.20	1557.78	104.22	-5.70	174.10	37.90	3.80	17.87
NLFH 3iv	5.45	0.21	2300.00	207.11	41.50	213.45	38.20	1.62	18.52

Date	NLFH 1	_S I		PM	NLFH L	.s	LP	PM
May 17/04	5.9	10.2	10.4	10.1 May 27/04	9.3	12.3	14.3	14.2
May 17/04	4.5	10.5	10.0	9.2 May 27/04	8.8	13.2	15.5	14.5
May 17/04	5.5	10.8	10.2	9.8 May 27/04	9.9	14.2	13.1	15.7
May 17/04	4.6	10.9	10.3	9.9 May 27/04	9.8	14.7	13.6	14.0
May 17/04	6.3	10.2	9.9	9.5 May 27/04	8.4	15.6	14.6	15.2
May 17/04	5.4	11.0	9.8	9.7 May 27/04	8.8	15.2	14.2	13.4
May 17/04	6.7	10.3	9.6	9.9 May28/04	7.2	9.7	10.2	10.8
May 17/04	6.4	10.4	9.9	10.0 May28/04	6.5	9.5	10.5	10.2
May 17/04	6.9	9.9	10.2	10.0 May28/04	6.2	10.6	10.8	11.1
May 17/04	5.3	10.2	10.5	10.1 May28/04	6.6	10.2	10.2	11.2
May 17/04	5.5	10.0	10.8	9.9 May28/04	8.6	9.8	9.9	10.4
May 17/04	5.4	10.6	10.7	9.9 May28/04	8.7	10.1	9.8	10.0
May 18/04	8.5	10.3	10.1	10.2 May28/04	7.2	9.8	9.6	10.7
May 18/04	8.2	12.3	10.1	9.0 May28/04	7.7	9.2	9.7	10.3
May 18/04	7.8	12.2	10.2	9.3 May28/04	8.4	9.8	10.4	11.3
May 18/04	7.6	10.2	10.4	9.1 May28/04	9.2	9.5	10.0	11.1
May 18/04	6.2	10.9	10.9	10.9 May28/04	7.8	9.1	10.2	11.2
May 18/04	6.2	11.3	11.3	11.3 May28/04	8.8	9.9	10.2	11.4
May 18/04	6.7	11.3	11.3	11.3 June 1/04	10.1	14.6	12.9	13.7
May 18/04	5.9	11.2	11.2	11.2 June 1/04	8.2	12.0	11.8	13.1
May 18/04	8.4	9.8	11.8	11.8 June 1/04	7.5	12.0	12.0	13.0
May 18/04	9.2	11.6	10.6	10.6 June 1/04	7.7	13.2	12.3	13.2
May 18/04	9.1	10.3	9.3	9.3 June 1/04	10.2	11.3	13.4	14.0
May 18/04	8.1	11.3	9.4	9.4 June 1/04	9.1	11.8	11.6	14.2
May 19/04	7.7	11.5	10.5	10.0 June 1/04	9.1	12.0	13.5	12.2
May 19/04	5.8	10.9	12.3	9.9 June 1/04	7.5	12.3	13.2	12.3
May 19/04	2.9	11.4	10.4	9.7 June 1/04	7.7	14.4	11.8	13.2
May 19/04	3.4	10.3	14.2	9.1 June 1/04	9.5	12.2	13.2	13.5
May 19/04	8.3	13.1	11.6	10.9 June 1/04	8.8	12.4	13.2	14.6
May 19/04	7.0	11.0	10.6	10.4 June 1/04	8.2	14.2	12.7	13.8
May 19/04	6.6	9.7	10.3	10.3 June 3/04	11.8	15.4	16.1	15.4
May 19/04	6.2	9.0	11.9	10.7 June 3/04	11.0	14.9	14.1	14.1
May 19/04	9.5	13.5	9.5	9.8 June 3/04	10.1	14.8	14.3	15.0
May 19/04	8.3	13.4	10.1	9.6 June 3/04	10.2	15.3	14.3	13.4
May 19/04	7.4	12.4	12.0	9.7 June 3/04	11.3	14.8	15.9	13.5
May 19/04	9.2	15.3	12.9	9.4 June 3/04	11.2	14.2	15.9	14.1
May 20/04	3.3	18.0	11.9	13.6 June 3/04	11.6	14.3	15.5	14.3
May 20/04	4.3	19.3	14.8	9.9 June 3/04	12.2	14.6	15.3	15.3
- May 20/04	2.5	17.4	14.0	12.2 June 3/04	12.0	16.0	16.0	14.8
- May 20/04	3.0	14.2	14.4	9.6 June 3/04	11.0	14.3	15.2	16.9
May 20/04	7.3	11.2	14.5	12.7 June 3/04	9.4	15.3	15.1	16.2
May 20/04	7.0	12.6	15.5	11.5 June 3/04	9.9	15.3	14.3	12.0
May 20/04	5.7	9.9	13.3	14.6 June 5/04	12.8	15.9	17.2	21.5
May 20/04	6.8	12.3	13.3	10.4 June 5/04	12.3	17.4	18.2	21.3
May 20/04	7.7	12.3	13.0	15.3 June 5/04	10.1	18.2	18.1	17.0
Date	NLFH	LS	LP I	PM	NLFH I	LS	LP	РМ
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May 20/04	4.9	10.5	11.2	12.0 June 5/04	12.2	18.5	17.7	17.2
May 20/04	5.4	15.5	11.3	15.7 June 5/04	14.1	18.2	20.8	18.5
May 20/04	5.5	11.8	12.5	15.0 June 5/04	11.1	16.3	20.3	17.5
May 21/04	3.6	9.9	13.3	11.7 June 5/04	11.3	18.8	19.1	17.0
May 21/04	3.0	9.8	9.4	9.9 June 5/04	13.3	18.6	19.3	19.1
May 21/04	2.8	8.5	12.1	9.6 June 5/04	12.3	16.8	20.2	16.8
May 21/04	1.3	9.4	11.0	10.3 June 5/04	11.3	15.4	20.4	18.4
May 21/04	5.8	9.3	10.5	13.5 June 5/04	14.4	15.7	20.9	18.3
May 21/04	6.5	12.6	11.1	12.5 June 5/04	13.4	16.3	19.1	16.3
May 21/04	4.9	10.1	9.7	10.9 June 7/04	8.5	10.2	12.2	12.3
May 21/04	4.4	13.1	10.1	12.5 June 7/04	7.5	11.8	12.0	12.2
May 21/04	6.3	12.3	12.2	14.6 June 7/04	7.7	11.3	12.5	12.2
May 21/04	5.6	11.3	11.3	10.6 June 7/04	7.1	11.3	12.3	12.4
May 21/04	6.7	11.7	11.7	10.5 June 7/04	8.9	10.9	12.0	12.1
May 21/04	4.9	10.7	11.0	14.1 June 7/04	8.3	11.4	12.1	11.6
May22/04	3.6	10.8	11.4	12.1 June 7/04	8.4	11.3	12.1	12.3
May22/04	3.5	10.9	11.9	11.3 June 7/04	8.3	11.3	12.3	11.4
May22/04	3.5	8.9	10.0	12.3 June 7/04	10.9	11.4	11.4	13.1
May22/04	2.1	9.3	10.4	11.7 June 7/04	10.1	12.5	12.0	13.4
May22/04	5.2	8.7	10.9	9.6 June 7/04	9.9	12.1	12.0	13.2
May22/04	5.2	9.2	10.3	11.7 June 7/04	9.3	11.4	12.4	12.6
May22/04	5.6	9.7	7.5	12.0 June 9/04	9.8	15.8	15.3	15.5
May22/04	5.1	9.8	9.3	10.3 June 9/04	9.2	15.3	16.3	15.9
May22/04	10.1	12.0	10.6	10.7 June 9/04	9.1	15.3	15.7	14.6
May22/04	7.2	9.5	9.7	11.0 June 9/04	9.0	15.3	15.3	15.3
May22/04	6.5	9.6	9.1	10.6 June 9/04	10.6	16.7	15.1	17.2
May22/04	7.5	10.0	11.2	13.8 June 9/04	9.7	15.3	16.8	17.6
May 23/04	5.2	9.7	10.3	9.9 June 9/04	9.2	16.3	15.7	16.3
May 23/04	5.1	9.9	10.4	10.1 June 9/04	9.1	15.7	15.3	14.3
May 23/04	2.9	8.4	10.3	10.0 June 9/04	11.1	16.6	17.7	18.3
May 23/04	4.5	9.0	11.4	10.6 June 9/04	10.1	15.3	17.4	17.7
May 23/04	7.1	10.2	13.3	12.3 June 9/04	9.8	16.1	16.7	18.3
May 23/04	5.2	10.3	12.3	8.9 June 9/04	9.9	17.0	16.3	16.7
May 23/04	5.5	10.3	9.9	11.9 June 11/04	10.5	14.0	17.0	14.7
May 23/04	5.2	9.4	10.3	11.3 June 11/04	9.9	14.1	18.0	15.0
May 23/04	7.5	9.5	9.7	11.7 June 11/04	9.8	14.3	18.3	14.6
May 23/04	6.5	8.7	9.6	10.1 June 11/04	10.1	14.3	18.3	15.3
May 23/04	4.3	9.1	10.2	12.5 June 11/04	10.9	14.3	20.3	14.8
May 23/04	4.6	12.0	10.4	11.3 June 11/04	11.1	14.7	21.3	14.4
May 24/04	6.2	13.4	10.2	9.8 June 11/04	10.3	14.7	21.1	14.5
May 24/04	3.5	10.5	11.5	10.2 June 11/04	10.4	14.3	20.3	14.6
May 24/04	2.4	9.0	11.0	10.2 June 11/04	12.0	14.1	20.2	14.7
May 24/04	4.0	9.2	9.0	10.3 June 11/04	10.0	14.4	20.3	14.4
May 24/04	6.8	10.3	9.3	10.9 June 11/04	11.6	14.5	20.3	14.9
May 24/04	5.2	11.0	10.5	12.4 June 11/04	11.7	14.3	20.1	15.3
May 24/04	5.3	10.9	11.2	11.4 June 13/04	10.8	15.8	14.3	15.3
May 24/04	4.0	12.0	10.3	11.2 June 13/04	11.4	14.4	14.3	15.2

Date	NLFH I	LS I	_P	PM	NLFH	LS	LP	PM
May 24/04	8.5	10.7	12.3	12.3 June 13/04	9.1	14.3	13.5	14.3
May 24/04	6.1	10.0	11.9	11.9 June 13/04	10.1	16.5	14.3	14.3
May 24/04	6.2	12.2	11.3	12.1 June 13/04	11.7	14.7	12.6	13.8
May 24/04	5.5	12.3	11.2	12.0 June 13/04	11.4	15.4	12.5	14.6
May 25/04	8.9	10.9	11.7	12.1 June 13/04	10.2	15.8	12.8	14.6
May 25/04	5.2	10.8	12.7	10.5 June 13/04	9.2	15.2	12.3	14.4
May 25/04	4.9	12.0	12.1	10.4 June 13/04	9.2	16.9	11.8	14.3
May 25/04	5.3	10.1	10.9	11.0 June 13/04	8.9	15.3	11.0	13.7
May 25/04	7.7	12.7	12.1	12.0 June 13/04	9.5	15.5	11.3	14.7
May 25/04	9.7	11.8	11.1	9.9 June 13/04	9.2	15.3	11.3	14.4
May 25/04	7.7	10.8	10.2	10.2 June 15/04	9.5	14.3	14.3	14.3
May 25/04	6.5	10.1	10.9	12.2 June 15/04	6.7	14.1	14.3	14.6
May 25/04	7.7	10.7	10.7	10.4 June 15/04	6.8	15.5	14.7	16.2
May 25/04	6.8	11.4	10.2	11.8 June 15/04	8.3	15.8	13.1	15.2
May 25/04	6.4	13.2	12.4	14.8 June 15/04	7.4	13.5	14.1	14.6
May 25/04	6.5	12.2	12.2	12.2 June 15/04	8.4	13.5	14.3	14.1
May 27/04	6.8	12.2	15.4	14.1 June 15/04	9.4	13.6	14.7	14.4
May 27/04	6.3	13.7	16.2	13.4 June 15/04	7.3	16.3	14.3	14.0
May 27/04	6.2	13.5	14.2	14.4 June 15/04	8.8	14.1	14.3	14.8
May 27/04	5.3	13.0	14.9	15.2 June 15/04	8.4	14.4	14.9	13.5
May 27/04	10.2	14.8	13.6	14.8 June 15/04	8.9	14.5	14.7	13.2
May 27/04	9.5	12.8	13.9	14.5 June 15/04	8.3	14.3	14.3	13.5

#### **APPENDIX D - Suncor**

Material	Plot	Corner 1	Corner 2	Corner 3	Corner 4
LFH over lean oil	1	57° 00.419'N	57° 00.416'N	57° 00.421'N	57°00.424'N
sands		111°26.979'W	111°26.985'W	111°26.994'W	111°26.986'W
	2	57°00.411'N	57°00.410'N	57°00.414'N	57°00.416'N
		111°26.958'W	111°26.967'W	111°26.971'W	111°26.965'W
	3	57°00.430'N	57°00.426'N	57°00.432'N	57°00.432'N
		111°26.964'W	111°26.972'W	111°26.977'W	111°26.972'W
Peat-mineral mix	1	57°00.471'N	57°00.473'N	57°00.473'N	57°00.476'N
		111°27.015'W	111°27.021'W	111°27.026'W	111°27.017'W
	2	57°00.498'N	57°00.490'N	57°00.500'N	57°00.503'N
		111°27.011'W	111°27.014'W	111°27.023'W	111°27.015'W
	3	57°00.515'N	57°00.517'N	57°00.517'N	57°00.517'N
		111°27.012'W	111°27.012'W	111°27.013'W	111°27.020'W
LFH over peat-	1	57°00.451'N	57°00.550'N	57°00.546'N	57°00.549'N
mineral mix		111°27.119'W	111°27.129'W	111°27.124'W	111°27.115'W
	2	57°00.564'N	57°00.567'N	57°00.566'N	57°00.560'N
		111°27.106'W	111°27.110'W	111°27.118'W	111°27.115`W
	3	57°00.552'N	57°00.553'N	57°00.558'N	57°00.555'N
		111°27.090'W	111°27.076'W	111°27.083'W	111°27.093'W
Natural forest	1	57°06.4570'N	57°06.565'N	57°06.558'N	57°06.563'N
		111°41.715'W	111°41.722'W	111°41.723'W	111°41.728'W
	2	57°06.5570'N	57°06.556'N	57°06.560'N	57°06.556'N
		111°41.703'W	111°41.700'W	111°41.714'W	111°41.712'W
	3	57°06.4577'N	57°06.581'N	57°06.573'N	57°06.579'N
		111°41.769'W	111°41.759'W	111°41.766'W	111°41.756'W

### GPS coordinates of sampling plots (square):

#### Abbreviations

Natural Forest LFH – NLFH Peat-mineral mix – SunC LFH over lean oil sands – SunL LFH over peat-mineral mix - SunF

## July 2003

name	ph	bd	microbial C	microbial N	net N mineralization	Moisture content	Soluble Organic Carbon	Soluble Organic Nitrogen	C:N
		(g/cm3)	(mg/kg)	(mg/kg)	(mg/kg)	(% weight)	(mg/kg)	(mg/kg)	
NLFH 1i	5.52	0.19	1930.67	122.67	21.40	60.32	48.20	1.74	19.22
NLFH 1ii	5.90	0.33	2477.78	107.56	7.50	38.15	35.20	1.17	17.98
NLFH 1iii	4.95	0.30	3191.11	99.33	27.10	48.05	49.00	1.47	22.05
NLFH 1iv	5.08	0.55	1411.11	72.89	22.50	69.12	25.60	1.02	20.85
NLFH 2i	5.31	0.40	3055.56	112.89	119.00	61.05	22.40	1.26	21.84
NLFH 2ii	5.36	0.36	4344.44	154.89	83.90	94.82	35.80	2.23	15.26
NLFH 2iii	5.34	0.27	5406.67	276.22	51.40	91.67	55.80	4.92	15.65
NLFH 2iv	5.50	0.18	762.22	146.22	-2.40	72.12	44.40	5.28	15.52
NLFH 3i	5.17	0.21	3591.11	121.78	-32.20	47.84	66.40	2.71	17.51
NLFH 3ii	4.77	0.17	3784.44	126.89	-75.70	77.19	65.80	4.13	17.51
NLFH 3iii	4.77	0.20	2417.78	104.89	138.50	52.81	25.00	1.71	17.87
NLFH 3iv	4.93	0.21	2057.78	112.00	45.00	48.48	67.00	1.86	18.52
SunL 1i	6.15	0.98	1198.00	11.11	13.80	12.92	14.20	0.72	30.24
SunL 1ii	6.26	0.91	631.11	24.89	16.90	19.80	23.80	0.85	32.25
SunL 1iii	6.23	0.96	555.56	30.44	31.60	14.81	27.20	1.04	41.29
SunL 1iv	6.29	0.72	177.78	20.22	8.80	19.08	21.40	0.73	66.70
SunL 2i	6.13	0.95	277.78	4.44	-32.20	13.92	21.00	1.99	46.75
SunL 2ii	6.04	0.89	273.33	4.00	-75.70	10.71	23.20	1.25	50.01
SunL 2iii	5.85	0.92	113.33	2.22	138.50	11.43	21.40	1.25	79.00
SunL 2iv	6.25	1.12	444.44	2.89	45.00	13.11	18.40	1.21	51.63
SunL 3i	6.40	1.03	233.33	18.22	58.40	13.69	21.20	0.71	43.50
SunL 3ii	6.21	1.08	342.22	28.89	52.70	12.08	20.20	0.64	40.39
SunL 3iii	6.30	1.04	346.67	25.11	-12.10	13.43	21.00	0.59	49.83
SunL 3iv	6.15	1.05	231.11	12.00	7.10	12.59	18.00	0.55	44.28
SunC 1i	6.72	0.85	328.00	10.44	1.80	23.54	37.40	0.73	29.42
SunC 1ii	6.76	0.93	800.00	15.33	7.90	37.93	38.80	0.78	26.74
SunC 1iii	6.81	0.79	1942.22	51.11	33.70	56.41	67.60	1.40	28.76

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# July 2003

name	ph	bd	microbial C	microbial N	net N mineralization	Moisture content	Soluble Organic Carbon	Soluble Organic Nitrogen	C:N
		(g/cm3)	(mg/kg)	(mg/kg)	(mg/kg)	(% weight)	(mg/kg)	(mg/kg)	
SunC 1iv	6.66	0.79	742.22	4.89	6.70	25.95	35.00	0.90	31.11
SunC 2i	6.94	0.63	880.00	2.22	-2.40	20.72	35.40	1.34	30.83
SunC 2ii	6.88	1.03	573.33	106.89	-1.90	16.93	31.60	1.12	30.95
SunC 2iii	6.84	0.87	360.00	111.33	14.60	19.40	32.60	1.02	30.94
SunC 2iv	6.85	0.88	766.67	94.89	6.90	26.82	37.00	1.72	28.28
SunC 3i	6.63	0.82	391.11	13.11	43.00	19.34	32.80	0.88	26.42
SunC 3ii	6.94	0.76	293.33	10.22	102.10	16.28	32.40	0.76	30.08
SunC 3iii	6.50	0.67	406.67	19.78	51.60	30.58	42.00	0.76	31.16
SunC 3iv	6.72	0.80	460.00	8.89	44.90	117.81	40.80	0.87	25.95

### May 2004

name	ph	bd	Microbial C	Microbial N	Net N mineralization	Moisture content	Soluble Organic Carbon	Soluble Organic Nitrogen	CN
		(g/cm3)	(mg/kg)	(mg/kg)	(mg/kg)	(% weight)	(mg/kg)	(mg/kg)	
NLFH 1i	5.4	4 0.1	9 1088.6	7 99.33	-1.60	196.40	62.73	14.15	i 19.22
NLFH 1ii	5	.5 0.3	33 1189.7	8 237.11	-11.60	213.82	55.80	11.80	) 17.98
NLFH 1iii	5.2	29 0.3	30 1169.33	3 179.33	-47.20	201.60	51.30	14.40	) 22.05
NLFH 1iv	5.1	16 0.5	5 1485.7	8 276.89	-22.80	139.45	5 <b>23.9</b> 3	8.33	3 20.85
NLFH 2i	5.4	41 0.4	10 3914.0	0 419.78	3 24.90	153.63	81.05	16.1	5 21.84
NLFH 2ii	5.4	48 0.3	36 2308.6	7 376.89	-49.70	154.10	) 103.63	15.73	3 15.26
NLFH 2iii	5.	44 0.2	27 2450.6	7 340.67	12.70	176.90	6 48.13	3 10.08	3 15.65
NLFH 2iv	5.	36 O. <sup>-</sup>	18 1349.1	1 170.67	7 70.40	154.21	33.23	3 7.90	) 15.52
NLFH 3i	5.	19 0.2	21 1104.2	2 65.33	3 40.00	181.65	5 <b>32.1</b> 0	) 7.75	5 17.51
NLFH 3ii	5.	34 0. <sup>-</sup>	17 1319.3	3 96.44	4 63.40	191.76	<b>3 25.9</b> 3	6.65	5 17.51
NLFH 3iii	5.	33 0.5	20 1557.7	8 104.22	2 33.10	268.71	1 37.90	) 10.50	) 17.87
NLFH 3iv	5.	45 0.2	21 1164.8	9 207.11	I 51.20	213.45	5 38.20	) 12.00	) 18.52
SunF 1i	e	5.8 1.0	08 48.4	4 32.22	2 15.30	9.09	77.27	5.69	<b>35.00</b>

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May 2	004
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name	ph b	d	Microbial C	Microbial N	Net N mineralization	Moisture content	Soluble Organic Carbon	Soluble Organic Nitrogen	CI	N
	(9	g/cm3)	(mg/kg)	(mg/kg)	(mg/kg)	(% weight)	(mg/kg)	(mg/kg)		
SunF 1ii	6.74	0.73	3 223.33	3 3.11	-8.20	19.70	6 59.78	8 9	.77	26.97
SunF 1iii	6.24	1.08	3 66.00	D 15.11	2.70	6.3	0 43.16	6 2	.72	31.55
SunF 1iv	6.96	1.02	2 114.67	7 15.56	34.20	15.70	0 62.29	9 8	.93	27.98
SunF 2i	6.52	0.98	3 288.22	2 22.22	8.20	22.1	9 43.61	1 2	.54	27.12
SunF 2ii	6.6	0.91	1 256.22	2 44.67	' 8.10	26.6	5 21.65	5 2	.12	26.94
SunF 2iii	6.8	0.96	6 22.8	9 31.78	9.20	6.3	7 59.70	0 3	.75	27.09
SunF 2iv	6.48	0.72	2 13.50	6 25.56	5 21.30	29.3	1 56.90	0 4	.07	28.97
SunF 3i	6.35	0.6	5 61.7	8 31.11	3.90	0.73	2 52.56	6 2	.73	25.45
SunF 3ii	6.64	0.99	9 385.5	6 70.00	) 4.80	16.7	0 43.59	9 2	.45	34.55
SunF 3iii	6.63	0.87	7 317.7	8 38.67	8.60	25.0	7 37.6	8 2	.60	24.87
SunF 3iv	6.34	1.07	7 75.78	8 27.11	2.60	) 11.8	5 42.20	0 2	.84	28.82
SunL 1i	5.58	0.98	8 126.2	2 32.44	1 7.80	) 21.2	6 11.30	0 1	.73	30.24
SunL 1ii	5.63	0.9	1 186.4	4 32.67	7.30	) 31.2	9 9.8	5 2	.28	32.25
SunL 1iii	5.55	0.9	6 201.5	6 54.00	) 18.60	) 9.9	1 10.5	3 2	.05	41.29
SunL 1iv	5.57	0.73	2 221.3	3 50.00	) 11.90	) 7.1	2 11.2	5 1	.78	66.70
SunL 2i	5.9	0.9	5 102.4	4 19.33	3 21.70	) 16.7	4 16.8	5 2	.25	46.75
SunL 2ii	5.76	0.8	9 77.1	1 14.89	9 16.30	) 14.9	0 18.9	0 2	.45	50.01
SunL 2iii	5.94	0.9	2 97.5	6 17.11	I 18.60	35.1	0 16.6	5 2	.73	79.00
SunL 2iv	6.16	1.1	2 122.0	0 36.89	9 16.20	) 10.8	6 14.5	0 1	.95	51.63
SunL 3i	5.82	1.0	3 216.8	9 54.44	4 16.20	) 14.2	0 13.3	8 2	.10	43.50
SunL 3ii	5.3	1.0	8 204.6	7 45.78	3 15.30	) 14.1	8 13.0	5 2	2.00	40.39
SunL 3iii	6	1.0	4 321.1	1 41.78	3 14.20	) 17.6	3 13.8	0 2	.25	49.83
SunL 3iv	5.5	1.0	5 206.6	7 46.44	4 12.80	) 14.5	1 13.7	0 2	.18	44.28
SunC 1i	6.78	0.8	5 44.8	9 19.33	3 14.00	) 35.7	8 35.0	5 2	.88	29.42
SunC 1ii	6.66	0.9	3 52.6	7 15.33	3 15.80	) 35.3	3 37.8	8 2	.95	26.74
SunC 1iii	6.65	0.7	9 160.0	0 344.00	0 36.40	) 49.4	0 46.5	3 2	2.90	28.76
SunC 1iv	6.6	0.7	9 71.1	1 18.00	0 19.90	) 44.7	1 42.7	2 3	3.18	31.11
SunC 2i	6.47	0.6	3 53.5	6 55.78	8 8.90	) 37.6	6 46.8	0 2	2.15	30.83
SunC 2ii	6.45	1.0	3 356.0	0 38.22	2 4.80	) 24.1	7 34.1	7 2	2.10	30.95

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May	2004
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name	ph	bd	Microbial C	Microbial N	Net N mineralization	Moisture content	Soluble Organic Carbon	Soluble Organic Nitrogen	С	N
		(g/cm3)	(mg/kg)	(mg/kg)	(mg/kg)	(% weight)	(mg/kg)	(mg/kg) .		
SunC 2iii	6.5	4 0.87	7 136.00	0 37.56	i 13.30	16.21	1 34.1	3	2.27	30.94
SunC 2iv	6.	7 0.8	8 152.67	7 34.22	7.90	26.19	9 39.9	3	2.21	28.28
SunC 3i	6.5	2 0.82	2 35.11	l 6.00	12.00	33.51	1 36.7	8	3.42	26.42
SunC 3ii	6.54	4 0.76	6 192.89	9 45.78	17.70	52.34	4 43.0	5	3.68	30.08
SunC 3iii	6.	7 0.67	7 436.00	) 79.11	22.50	50.31	1 44.4	3	3.62	31.16
SunC 3iv	6.5	5 0.80	0.22	2 11.11	18.00	73.47	7 49.6	2	4.79	25.95

Date	NLFH S	SunL S	SunF S	SunC	NLFH :	SunL :	SunF :	SunC
May 17/04	5.9	14.1	16.9	15.2 May 27/04	9.3	15.8	14.1	15.4
May 17/04	4.5	16.4	16.3	13.3 May 27/04	8.8	16.0	14.0	15.5
May 17/04	5.5	15.1	16.4	17.5 May 27/04	9.9	17.5	15.4	13.7
May 17/04	4.6	15.8	16.6	15.6 May 27/04	9.8	14.8	12.7	13.4
May 17/04	6.3	17.7	20.7	15.6 May 27/04	8.4	15.3	14.7	14.1
May 17/04	5.4	19.5	19.9	15.7 May 27/04	8.8	15.3	13.3	14.1
May 17/04	6.7	16.9	19.4	15.3 May28/04	7.2	12.8	11.1	11.6
May 17/04	6.4	17.6	17.6	14.4 May28/04	6.5	11.4	12.9	11.8
May 17/04	6.9	15.1	14.4	11.8 May28/04	6.2	11.2	12.8	11.8
May 17/04	5.3	15.3	15.5	14.3 May28/04	6.6	12.2	11.3	12.2
May 17/04	5.5	15.6	14.4	14.5 May28/04	8.6	11. <b>1</b>	11.8	12.4
May 17/04	5.4	16.9	15.4	13.5 May28/04	8.7	11.0	13.1	12.3
Mav 18/04	8.5	14.3	16.2	15.8 Mav28/04	7.2	12.2	12.4	10.2
May 18/04	8.2	14.3	15.6	15.7 Mav28/04	7.7	12.0	12.2	11.2
May 18/04	7.8	13.3	17.2	15.1 May28/04	8.4	11.4	11.6	10.2
May 18/04	7.6	15.3	15.8	15.4 May28/04	9.2	112	11.4	10.3
May 18/04	62	15.3	16.6	16.3 May28/04	7.8	12.1	11.8	11.0
May 18/04	6.2	15.3	17.4	16.3 May28/04	88	11.3	12.2	10.1
May 18/04	6.7	14.4	15.4	16.7 June 1/04	10.1	16.3	19.8	17.3
May 18/04	59	15.3	16.4	15.5.June 1/04	82	15.7	20.3	18.7
May 18/04	8.4	14.1	16.4	14.1.June 1/04	7.5	18.3	17 4	16.6
May 18/04	0.4 Q 2	14.3	17.6	16.4 June 1/04	7.0	10.0	10.1	18.2
May 18/04	9.2 Q 1	15.0	17 4	16.4 June 1/04	10.2	17.3	18.1	17.0
May 18/04	9.1	14.5	11.4	15.5 June 1/04	0.2	17.0	10.1	19.0
May 10/04	77	14.0	10.4	16.3 June 1/04	9.1	10.4	10.9	10.2
May 19/04	1.1 E 9	15.5	19.4	16.3 June 1/04	9.1 7.5	10.1	10.0	10.0
May 19/04	5.0	10.0	10.9	17.2 June 1/04	7.5	19.2	10.0	10.2
May 19/04	2.9	10.5	17.0	17.2 June 1/04	1.1	17.9	17.9	19.0
May 19/04	3.4	14.5	10.1	13.0 June 1/04	9.5	18.3	18.1	17.7
May 19/04	8.3	18.3	16.5	17.3 June 1/04	8.8	18.9	19.5	17.4
May 19/04	7.0	17.4	15.0	17.5 June 1/04	8.2	19.0	18.3	17.6
May 19/04	6.6	17.9	16.7	17.4 June 3/04	11.8	18.9	18.2	15.8
May 19/04	6.2	16.5	15.3	16.9 June 3/04	11.0	20.5	18.7	15.6
May 19/04	9.5	16.3	15.3	13.4 June 3/04	10.1	17.0	19.0	16.1
May 19/04	8.3	15.3	14.5	11.3 June 3/04	10.2	22.4	18.8	15.5
May 19/04	7.4	13.8	13.9	13.4 June 3/04	11.3	14.5	16.7	15.6
May 19/04	9.2	15.3	16.3	14.7 June 3/04	11.2	14.9	17.0	16.4
May 20/04	3.3	16.1	16.9	15.3 June 3/04	11.6	17.2	16.4	17.6
May 20/04	4.3	19.4	19.3	9.8 June 3/04	12.2	17.8	19.4	15.5
May 20/04	2.5	19.1	16.4	19.3 June 3/04	12.0	13.2	17.3	16.8
May 20/04	3.0	17.5	16.6	15.6 June 3/04	11.0	16.6	16.4	17.5
May 20/04	7.3	18.7	18.7	15.5 June 3/04	9.4	16.6	17.6	16.5
May 20/04	7.0	22.5	22.5	17.7 June 3/04	9.9	16.3	17.4	16.4
May 20/04	5.7	18.4	18.4	11.4 June 5/04	12.8	20.7	30.3	22.5
May 20/04	6.8	16.4	16.4	11.5 June 5/04	12.3	19.8	27.0	20.0
May 20/04	7.7	13.0	12.0	12.8 June 5/04	10.1	24.7	21.7	18.3
May 20/04	4.9	16.3	15.1	14.1 June 5/04	12.2	23.1	22.2	19.1
May 20/04	5.4	15.3	15.4	11.5 June 5/04	14.1	22.4	21.7	24.5

Date	NLFH :	SunL	SunF :	SunC	NLFH	SunL	SunF	SunC
May 20/04	5.5	16.3	16.2	12.1 June 5/04	11.1	22.3	26.9	21.3
May 21/04	3.6	12.9	16.1	11.7 June 5/04	11.3	17.0	21.3	25.6
May 21/04	3.0	14.5	16.4	10.2 June 5/04	13.3	19.5	23.3	25.4
May 21/04	2.8	15.3	15.9	10.2 June 5/04	12.3	22.2	20.4	18.9
May 21/04	1.3	13.7	16.3	12.1 June 5/04	11.3	23.2	23.2	17.4
May 21/04	5.8	10.7	14.9	13.0 June 5/04	14.4	25.3	21.4	20.4
May 21/04	6.5	12.1	14.3	13.5 June 5/04	13.4	23.2	22.3	18.9
May 21/04	4.9	11.0	13.6	12.3 June 7/04	8.5	13.2	16.8	13.6
May 21/04	4.4	12.5	10.9	June 7/04	7.5	15.3	16.5	15.0
May 21/04	6.3	10.6	14.1	10.6 June 7/04	7.7	14.4	14.2	14.7
May 21/04	5.6	9.9	11.1	9.9 June 7/04	7.1	14.3	15.3	14.8
May 21/04	6.7	13.5	14.4	13.5 June 7/04	8.9	14.2	14.3	12.8
May 21/04	4.9	12.3	12.5	12.3 June 7/04	8.3	15.8	15.7	15.8
May22/04	3.6	11.5	12.6	10.8 June 7/04	8.4	14.9	14.2	14.6
May22/04	3.5	15.1	14.3	11.6 June 7/04	8.3	14.3	14.2	14.6
May22/04	3.5	15.1	12.3	10.9 June 7/04	10.9	12.9	14.3	13.3
May22/04	2.1	15.8	13.6	13.3 June 7/04	10.1	12.8	14.9	13.3
May22/04	5.2	11.3	11.8	12.2 June 7/04	9.9	14.4	15.8	13.7
May22/04	5.2	11.5	15.5	15.5 June 7/04	9.3	13.0	15.1	13.6
May22/04	5.6	11.8	14.6	11.8 June 9/04	9.8	19.0	24.6	18.4
May22/04	5.1	12.0	13.5	10.2 June 9/04	9.2	23.2	21.6	17.4
May22/04	10.1	12.3	11.5	8.6 June 9/04	9.1	21.8	23.9	17.6
May22/04	7.2	13.8	13.0	9.1 June 9/04	9.0	22.3	21.1	18.6
May22/04	6.5	12.6	11.2	9.6 June 9/04	10.6	21.4	22.4	18.2
May22/04	7.5	12.6	12.5	10.2 June 9/04	9.7	18.6	18.5	19.0
May 23/04	5.2	12.5	12.8	10.7 June 9/04	9.2	17.8	19.5	17.8
May 23/04	5.1	10.3	14.5	11.1 June 9/04	9.1	17.7	19.1	19.1
May 23/04	2.9	10.3	12.6	12.3 June 9/04	11.1	17.3	20.3	16.5
May 23/04	4.5	11.0	13.4	10.1 June 9/04	10.1	19.1	19.3	16.2
May 23/04	7.1	14.4	16.4	10.4 June 9/04	9.8	18.2	18.1	16.3
May 23/04	5.2	15.6	15.5	12.2 June 9/04	9.9	18.5	18.2	16.3
May 23/04	5.5	15.0	10.6	10.4 June 11/04	10.5	15.3	17.7	16.1
May 23/04	5.2	14.5	13.3	10.2 June 11/04	9.9	16.4	17.5	15.8
May 23/04	7.5	13.2	13.4	8.8 June 11/04	9.8	16.6	16.5	15.6
May 23/04	6.5	13.1	12.0	7.5 June 11/04	10.1	17.6	16.9	15.6
May 23/04	4.3	10.5	11.9	10.2 June 11/04	10.9	17.3	16.8	15.0
May 23/04	4.6	11.3	13.2	11.2 June 11/04	11.1	17.4	17.6	16.7
May 24/04	6.2	14.9	20.3	13.2 June 11/04	10.3	17.6	16.7	16.3
May 24/04	3.5	16.6	22.4	13.7 June 11/04	10.4	16.3	17.6	16.6
May 24/04	2.4	13.5	18.4	14.3 June 11/04	12.0	18.2	16.1	14.8
May 24/04	4.0	14.2	16.2	15.3 June 11/04	10.0	17.3	16.6	15.6
May 24/04	6.8	13.2	18.0	16.8 June 11/04	11.6	17.5	16.8	16.6
May 24/04	5.2	12.6	19.5	15.7 June 11/04	11.7	16.7	16.7	15.9
May 24/04	5.3	15.4	15.1	16.3 June 13/04	10.8	17.8	18.9	16.1
May 24/04	4.0	14.2	14.5	15.5 June 13/04	11.4	19.3	19.7	15.1
May 24/04	8.5	13.2	17.1	12.2 June 13/04	9.1	17.3	18.7	16.4
May 24/04	6.1	12.6	13.3	15.1 June 13/04	10.1	21.2	18.3	15.3

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May 24/04	6.2	15.4	12.1	14.8 June 13/04	11.7	14.5	17.7	17.6
May 24/04	5.5	13.2	14.1	14.5 June 13/04	11.4	14.9	15.9	16.6
May 25/04	8.9	14.6	17.3	14.4 June 13/04	10.2	17.2	16.4	16.3
May 25/04	5.2	18.0	18.5	13.3 June 13/04	9.2	17.8	19.2	15.9
May 25/04	4.9	20.7	17.9	13.3 June 13/04	9.2	16.2	17.7	16.1
May 25/04	5.3	19.2	18.2	14.0 June 13/04	8.9	17.3	16.3	16.3
May 25/04	7.7	14.3	16.6	14.9 June 13/04	9.5	15.3	16.6	15.3
May 25/04	9.7	14.8	17.1	12.8 June 13/04	9.2	15.3	17.8	15.3
May 25/04	7.7	12.9	18.3	12.8 June 15/04	9.5	14.9	17.2	15.4
May 25/04	6.5	13.2	17.3	13.2 June 15/04	6.7	15.4	16.4	15.4
May 25/04	7.7	17.8	14.9	17.8 June 15/04	6.8	16.4	15.6	14.0
May 25/04	6.8	16.3	12.4	16.2 June 15/04	8.3	14.5	15.3	13.9
May 25/04	6.4	17.5	12.6	14.9 June 15/04	7.4	15.2	15.2	14.1
May 25/04	6.5	15.3	13.3	15.1 June 15/04	8.4	15.5	15.4	15.3
May 27/04	6.8	16.6	17.8	14.9 June 15/04	9.4	15.9	16.2	14.9
May 27/04	6.3	18.5	16.6	13.8 June 15/04	7.3	15.5	14.9	15.6
May 27/04	6.2	16.1	16.4	13.3 June 15/04	8.8	15.9	16.3	15.2
May 27/04	5.3	16.2	16.5	12.9 June 15/04	8.4	13.9	15.2	14.5
May 27/04	10.2	18.0	15.7	15.4 June 15/04	8.9	16.4	16.4	15.3
May 27/04	9.5	15.4	15.2	15.3 June 15/04	8.3	16.4	15.2	15.6