

Sulphur Fertilization of Lodgepole Pine: A Stable Isotope Tracer Study (E.P. 886.15) Establishment Report

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Paul Sanborn, Rob Brockley, Bernhard Mayer,
Misuk Yun, and Joerg Prietzel



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PREFACE

This Technical Report constitutes the establishment report for the British Columbia Ministry of Forests E.P. 886.15, "Sulphur Fertilization of Lodgepole Pine: A Stable Isotope Tracer Study." The intent is to preserve data of permanent value to the researchers and forestry practitioners who will interpret and apply the results of this study.

This project grew out of the Ministry of Forests research program on fertilization of lodgepole pine in the British Columbia interior, conducted principally under E.P. 886, "Fertilizing and Thinning in Interior Forests," led by Rob Brockley (Kalamalka Research Station). Recognition of widespread sulphur (S) deficiencies in interior lodgepole pine stands resulted in establishment of an array of additional fertilizer trials during the 1990s that examined responses to various rates and forms of S fertilizer. However, Ministry researchers recognized that a deeper understanding of fertilizer S behaviour in managed conifer stands would require new approaches, and stable isotope tracer methods showed considerable promise.

After initial contacts with the stable isotope group at the University of Calgary in 1995 and 1996, Dr. Bernhard Mayer joined Paul Sanborn, then with the Prince George Forest Region, and Rob Brockley in conducting pilot studies of the feasibility of using stable isotope methods to trace the fate and behaviour of fertilizer S in plot-scale field experiments. The present study is the outcome of this team's initial collaboration.

For further information on the status of this project, contact either Paul Sanborn (sanborn@unbc.ca) or Rob Brockley (Rob.Brockley@gems7.gov.bc.ca). The data files on which this report is based are archived in the Ministry of Forests Research Branch, Growth and Yield database.

ABSTRACT

Widespread sulphur (S) deficiencies have been detected in lodgepole pine (*Pinus contorta* Dougl. var. *latifolia* Engelm.) stands in the Sub-Boreal Spruce biogeoclimatic zone of central interior British Columbia. Field experiments in this region have shown that addition of sulphate-S to nitrogen (N) fertilization treatments rapidly increases foliar S concentrations, and usually improves tree growth responses relative to N-only treatments. However, there is an insufficient scientific basis for choosing this S form over more slowly available elemental S-based fertilizers.

To address this knowledge gap, this study was begun in 2001 to compare the behaviour of sulphate-S and elemental-S fertilizers in an area-based fertilizer trial, using stable isotope tracer methods to examine the fate and transformations of fertilizer S. Fertilizer treatments were applied to two lodgepole pine stands, near Fraser Lake (Holy Cross site) and in the Bowron River valley (Kenneth Creek site), in fall 2002. This establishment report reviews background literature relevant to this study, details the experimental design and methods used, and documents the initial soil and stand conditions at the time the experimental treatments were installed.

Pre-treatment analyses indicated that mineral soils at these sites have low total S concentrations, which are typical of the British Columbia central

interior and are among the lowest reported in the temperate and boreal zones worldwide. Concentrations of other total and (or) available soil macronutrients (N, Ca, Mg, K, P) were usually higher at the Holy Cross site than at the Kenneth Creek site. Lodgepole pine foliar analyses indicated that S deficiency was more pronounced at the former site. Ratios of background S stable isotopes in lodgepole pine foliage and soils differed sufficiently from those of the applied S fertilizers to make a tracer experiment feasible.

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Dr. Jim Beaton assisted us in many ways; his authoritative knowledge of fertilizer properties and behaviour improved this project.

This report benefited considerably from reviews by Dr. Doug Maynard (Canadian Forest Service) and Les Herring. Dr. Barbara Kishchuk (Canadian Forest Service) kindly allowed us reproduce a diagram from her dissertation.

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This study, begun in 2001, addresses the behaviour of sulphur (S) fertilizers in the soils and vegetation of lodgepole pine (*Pinus contorta* Dougl. var. *latifolia* Engelm.) stands, which represent significant portions of the Sub-Boreal Spruce biogeoclimatic zone, and may be relevant to a wider area of the British Columbia central interior. The study builds upon previous S fertilization research undertaken by the British Columbia Ministry of Forests, and strengthens the scientific basis for fertilization of lodgepole pine. Such advances are timely, given the pending timber supply shortfalls created by the current mountain pine beetle (*Dendroctonus ponderosae* Hopkins) outbreak in this region. Fertilization will likely be one of the few methods available for accelerating the growth of the remaining immature lodgepole pine resource.

This section reviews five major areas of background relevant to this research, and describes details of pilot studies carried out in 1997 to test the feasibility of using stable isotope methods in S fertilization research in the British Columbia interior.

1.1 Sulphur Forms and Behaviour in Forest Soils

Knowledge of S distribution, forms, and behaviour in forest soils expanded greatly during the 1980s and 1990s, through the substantial research devoted to understanding the ecological impacts of acid deposition, particularly in eastern North America and western Europe, and to a lesser extent in Alberta. Although a comprehensive 1992 review of forest S dynamics noted the rarity of S deficiencies in forests (Mitchell et al. 1992), this may reflect the particular attention that researchers gave to areas of excess S supply from anthropogenic sources. This has implications for designing treatments to ameliorate nutrient deficiencies of forests in low-S environments. Research findings on the fate of anthropogenic S inputs in already S-rich soils may not be completely relevant to fertilizer behaviour in S-deficient soils.

The pathways of S transformation in ecosystems involve both organic and inorganic forms, changes in oxidation state, microbial and enzymatic processes, interactions with inorganic solid phases through adsorption/desorption, and trace gas fluxes to the atmosphere (Figure 1). Despite this apparent complexity, many of these forms and processes are of little significance in the functioning of aerobic forest soils in unpolluted environments, which will be the focus of this review.

The range of total S concentrations found in a survey of mostly temperate-zone forest soils was approximately 50–800 mg · kg⁻¹ in mineral soils and 1000–2000 mg · kg⁻¹ in forest floors (Mitchell et al. 1992). For most surface soils of temperate, humid, and semi-humid regions, which would include the more intensively studied forest soils of temperate and boreal zones, 90% or more of total soil S is in organic forms (Zhao et al. 1996). Organic S is conventionally segregated into two pools: (1) ester sulphates and (2) carbon-bonded S. The ester sulphates are operationally defined as the fraction reducible to H₂S by a reagent including hydriodic acid (HI). This group consists primarily of organic ester sulphates (–C–O–S–), inorganic sulphate, sulphamates (–C–N–S–), and other less common compounds. Ester sulphate concentrations are usually calculated as the HI-reducible S fraction, less any inorganic sulphate (either removed from the sample by leaching, or determined separately). The C-bonded S fraction consists of defined compounds, such as the S-containing amino acids, sulpholipids, and sulphonic acids, as

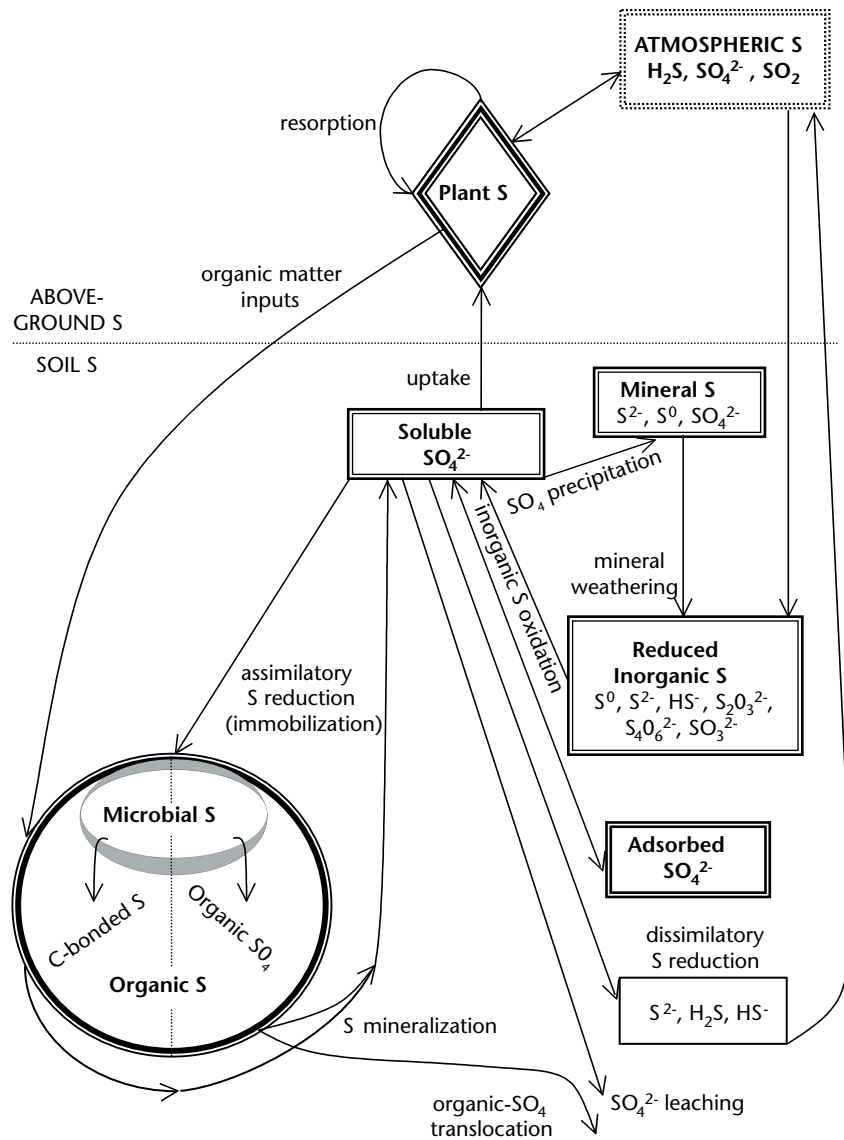


FIGURE 1 Conceptual model of sulphur cycling in soils (Kishchuk 1998).

well as poorly characterized components of humic substances. C-bonded S is usually operationally defined as the difference between total S and HI-reducible S. A method for direct determination of two components of C-bonded S, thiol-S (R-SH) and disulphide-S (R-S-S-R'), in plant tissues and forest floor materials was recently developed, but has not become a routine technique (Strehl and Prietzel 1999). C-bonded S is usually the predominant organic S fraction, accounting for 50–80% of total S (vs. 20–30% for ester sulphates) in a range of forest soils in northeastern North America (Mitchell et al. 1992).

Organic S is contributed to soils by both detrital plant materials and microbial sources. Although organic S in plant detritus consists mostly of proteins and amino acids (see Section 1.2), soil microbial biomass can contain a substantial proportion of other S compounds. Fungal production of sulphate esters, comprising approximately 40% of their total S, can be stimulated by addition of inorganic sulphate-S to culture media (Saggar et al. 1981); these compounds may act as an internal S reserve (Fitzgerald 1976). Overall,

microbial biomass S typically comprises about 2–3% of total soil S (Paul and Clark 1996). Although turnover rates of microbial biomass S seem not to have been documented in the literature, based on analogies with C and N this soil S pool is likely to actively cycle soil S, as shown conceptually in Figure 1. There are few published estimates of microbial biomass S in forest soils: Prietzel et al. (2001) found that microbial S comprised 1.8–5.6% of organic S in forest floors and surface (0–10 cm depth) mineral soils at two sites in the Black Forest (Germany), while Chen et al. (2001) found a greater contrast between forest floors and mineral soils, with the microbial S accounting for up to 11.38% of total S in the latter.

While most studies of forest nutrition emphasize the role of forest floors and the uppermost mineral soil, deeper mineral horizons often contain a substantial reserve of organic S, although the extent of its participation in nutrient cycling is poorly understood. For example, Luvisols in a boreal aspen forest in Saskatchewan contained 61% of their organic S reserves in the B and C horizons (Huang and Schoenau 1996). This organic S appears to originate through leaching of nutrient-rich soluble organic matter from the upper horizons (Schoenau and Bettany 1987).

Soil inorganic S consists primarily of sulphate-S, both adsorbed and water-soluble. Sulphate adsorption, which is reversible in varying degrees, is more likely to be an important S retention mechanism in deeper soil horizons with lower concentrations of organic matter and more abundant Fe and Al oxides. Adsorbed sulphate tends to be a larger proportion—as much as one-third to two-thirds—of soil total S in forests receiving higher rates of atmospheric S deposition (Johnson 1984). A vast literature has addressed the controls and modelling of sulphate adsorption processes, including such issues as its importance relative to organic S formation in S retention by forest soils (see Johnson and Mitchell 1998).

Most Canadian studies of sulphate adsorption have involved Podzolic soils from Ontario and Quebec, and have characterized its pH-dependence, reversibility, and relationships with common soil properties. Neary et al. (1987) characterized sulphate retention in Podzolic forest soils in central Ontario, finding significant correlations between adsorbed sulphate and extractable Fe and Al. In Quebec, Courchesne and Hendershot (1989) demonstrated the strong pH-dependence of sulphate adsorption by Podzolic forest soils, with maximum adsorption occurring between pH 3.5 and 4.5. The sulphate adsorption behaviour of British Columbia forest soils has received much less attention, apart from an unpublished study of Podzolic and Brunisolic coastal soils (Sanborn and Ballard 1990). As with similar soils in eastern Canada, strong pH dependence of sulphate adsorption was observed, although the limitations of simple batch adsorption experiments were noted. In particular, natural forest floor leachates contain soluble organic matter that can dramatically reduce sulphate adsorption (Gobran and Nilsson 1988).

In most productive temperate and boreal forest soils, sulphate salts such as gypsum ($\text{CaSO}_4 \cdot \text{H}_2\text{O}$) will be found only at depths below the rooting zone, and usually in drier climates. Precipitation of Al-containing sulphates may occur in some acidic forest soils (Johnson and Mitchell 1998). Inorganic non-sulphate S forms are seldom measured, and are usually assumed to be unimportant in the S cycle of aerobic forest soils.

Since organic S forms predominate in most forest soils, the supply of plant-available sulphate-S is largely controlled by turnover of organic S through mineralization and immobilization processes mediated by microbes,

as well as extracellular enzymes. In their model of soil nutrient cycling, McGill and Cole (1981) tried to account for the difficulty in predicting S mineralization behaviour of soils from simple C/S ratios. They distinguished between biological mineralization (nutrient release from organic materials driven by the microbial search for energy) and biochemical mineralization (driven by specific nutrient requirements, and conducted by extracellular enzymes). Based on these two mineralization pathways, ester sulphates and C-bonded S were predicted to behave differently in both short-term nutrient cycling processes and long-term soil development.

This model has influenced interpretations of several incubation experiments in which C and S supplies have been manipulated or radioisotopic tracers employed (David et al. 1983; Maynard et al. 1983; Ghani et al. 1991, 1992; Dail and Fitzgerald 1999; Houle et al. 2001). One implication of the model is that ester sulphates should act as an elastic storage form of retained S, responsive to changes in external S supply or internal soil S availability—a matter of considerable relevance to the fate and retention of anthropogenic S inputs. Field evidence for this hypothesis is mixed, with studies demonstrating pronounced differences between sites in the relative importance of ester sulphate formation and inorganic S retention processes in response to similar rates of S addition (Prietz et al. 2001). It is also important to distinguish between rates of short-term mineralization/immobilization and the role of organic S formation in long-term net accumulation patterns (Houle et al. 2001). Soil ester sulphates are not a single, homogeneous fraction, and recently formed components are considerably more labile in Podzolic soils (Lou and Warman 1992).

Volatilization losses of S during prescribed burning or wildfires may reinforce inherently low S reserves in some forest ecosystems. Long-term nutrient uptake and recycling through litterfall and litter decomposition tend to concentrate an increasing proportion of ecosystem S in the vegetative biomass and forest floor—pools that are more vulnerable to fire-induced losses. Field measurements of S volatilization losses from burning vegetation in various ecosystems have found that relative to N, proportional S losses can be higher (Ewel et al. 1981), lower (Delmas 1982), or identical (Raison 1980). Combustion experiments under more controlled conditions have shown that the proportion of total S lost increases with temperature (Evans and Allen 1971; Tiedemann 1987). Losses of S from Douglas-fir (*Pseudotsuga menziesii* [Mirb.] Franco) foliage can exceed 90% at 600°C (Sanborn and Ballard 1991). Potential S losses from biomass may also be related to the relative amounts of various S chemical forms, which in turn can be influenced by both soil S availability and interactions with N supply. A higher proportion of inorganic sulphate in plant tissues, because of high soil S availability and (or) low N availability (Section 1.2), results in correspondingly lower volatilization losses across a range of combustion temperatures (Sanborn and Ballard 1991).

In interior British Columbia mineral soils, total S concentrations are usually below 100 mg · kg⁻¹ (Table 1), clearly at the bottom of the documented range for temperate and boreal forest soils (Kishchuk and Brockley 2002). However, the greater abundance of C-bonded S than ester sulphate-S in both mineral soils and forest floors at both these interior and coastal sites is similar to general patterns reported for forest soils. The much higher mineral soil S concentrations at a Vancouver Island site (Table 1) may reflect naturally higher atmospheric inputs from the marine atmosphere (Klinger and Erickson 1997), which would be blocked from interior sites in the rain shadow of

TABLE 1 Sulphur fraction data for forest soils in three regions of British Columbia^a

Fraction	Prince George			Nelson			Vancouver Island	
	F	B	C	F	B	C	Forest floor	0–20 cm
Total S (mg · kg ⁻¹)	1200	64	61	1000	65	60	740	240
C-bonded S (% of total S)	78.5	68.4	77.5	89.1	70.0	58.3	78.1	63.5
Ester SO ₄ -S (% of total S)	17.6	26.7	20.6	8.3	26.3	40.4	20.2	33.2

^a Mean values are reported for F, B, and C horizons in the former Prince George (six sites) and Nelson (four sites) forest regions (Kishchuk 1998), and for forest floors and surface (0–20 cm depth) mineral soils near Port Alberni, Vancouver Island (Sanborn and Ballard 1990).

the Coast Mountains. Despite the geological complexity of British Columbia, the contribution of bedrock composition to the S content of soil parent materials has not been systematically studied. An indirect indication comes from regional variation in surface water geochemistry within the Fraser River watershed (Cameron et al. 1995). Dissolved sulphate concentrations are highest in the sedimentary terrain of the headwaters, and S stable isotope data suggest that dissolution of sedimentary sulphate minerals is the source. Sulphate concentrations are lower in most downstream tributaries draining the central and southern Interior plateaus, with isotopic data suggesting that oxidation of sulphide minerals is a major S source.

1.2 Sulphur Nutrition of Conifers

In conifers and other plants, S is taken up in sulphate form and undergoes assimilatory reduction primarily in the leaves (Marschner 1993) as the first step in the synthesis of S-containing amino acids. Proteins comprise the predominant pool of S in plant tissues, resulting in an organic S/N ratio that is characteristic of the species, and ranges from 0.025 in legumes to 0.032 in grasses (gram-atom basis) (Dijkshoorn and Van Wijk 1967). In conifers, this relationship was first documented in *Pinus radiata* in Australia, with a gram-atom organic S/total N ratio of 0.030 in foliage and seeds. Interactions between S and N nutrition were interpreted from foliar analyses: inadequate N supply in the presence of abundant S tended to result in accumulation of excess sulphate-S, whereas the presence of little or no sulphate-S may indicate an S deficiency (Kelly and Lambert 1972). Plants in S-rich environments accumulate excess S mostly as inorganic sulphate-S. However, soluble organic S can also increase, principally as the peptide glutathione (Rennenberg 1984), though it still forms a very small proportion of total S involved in internal transport, as in the case of *Picea abies* (L.) Karst (Köstner et al. 1998).

These relationships were elaborated for Douglas-fir in a series of papers by Turner et al. (1977, 1979, 1980) and Turner and Lambert (1980) that examined the responses of foliar sulphate-S to manipulation of N availability in fertilization trials, and the utility of foliar sulphate-S as a predictor of response to N fertilization. The N fertilization decreased sulphate-S and increased organic S concentrations in foliage at sites that received elevated atmospheric S inputs from air pollution (Turner et al. 1980). Based on this close linkage between N and S, and the plasticity of sulphate-S in relation to N supply, Turner et al. (1977) suggested that sulphate-S concentration would reliably predict potential response of Douglas-fir to urea fertilization.

1.3 Sulphur in U.S. Pacific Northwest and British Columbia Forests

After the early work of Turner and co-workers, it was not until the late 1980s that the S nutrition of forests in British Columbia and the adjacent U.S. Pacific Northwest began to receive increased attention. This reflected the growing maturity of forest fertilization research in this region, and concomitant needs to understand inconsistent responses to N addition and identify other limiting nutrients. Simultaneously, a much greater understanding of S dynamics in forest ecosystems was being gained through the upsurge of research on the effects of acid deposition in Europe and eastern North America. Presentations to three forest fertilization symposia in the late 1980s and early 1990s (Chappell and Miller [compilers] 1988; Lousier et al. [editors] 1991; Chappell et al. [editors] 1992) summarized the somewhat limited and fragmentary picture available at that time. In their regional overviews of forest nutrition and fertilization response, Brockley et al. (1992) and Mika et al. (1992) cited initial evidence for S deficiencies and positive responses to S fertilization in some inland portions of the Pacific Northwest and British Columbia. Areas of particular concern included the pumice soil region of central Oregon (Will and Youngberg 1978) and the British Columbia central interior (Brockley 1991). Although fertilization trials in the Pacific Northwest had occasionally included S in fertilizer prescriptions, their experimental designs did not detect specific responses to this element, as in the case of Cochran's studies of lodgepole pine (1979) and white fir (*Abies concolor* [Gord. & Glen.] Lindl.) (1991) in central Oregon.

Studies of fertilization and nutrient status in coastal and Cascade Range conifer stands have provided mixed evidence for S deficiencies and fertilization responses. For example, in coastal Washington and Oregon, Blake et al. (1988) found that soil sulphate-S concentrations were useful in identifying Douglas-fir stands likely to respond better to N + S than to N-only treatments. But subsequent work found that the predictive ability of soil sulphate-S was much less clear (Blake et al. 1990). On Vancouver Island, Weetman et al. (1997) found no additional benefit from inclusion of S in a Douglas-fir optimum nutrition field experiment. This result was consistent with observed foliar S concentrations and N/S ratios relative to the established criteria of Ballard and Carter (1986). Carter et al. (1998) found that both mineral soil and foliar sulphate-S concentrations did not improve prediction of N fertilizer response in Douglas-fir in coastal British Columbia, although they noted that foliar sulphate-S concentrations did not appear to be growth-limiting. In the case of western hemlock (*Tsuga heterophylla* [Raf.] Sarg.), Radwan and DeBell (1980) and Radwan and Shumway (1983) found no clear evidence that S availability influenced productivity or fertilization response.

More recent fertilization studies at inland sites in the Pacific Northwest have found stronger evidence that S may limit response to N fertilization. Tiedemann et al. (1998) found that urea fertilization ($350 \text{ kg N} \cdot \text{ha}^{-1}$) of an Oregon grand fir (*Abies grandis* [Dougl.] Forbes) stand depressed soil S availability, and recommended that S be included in fertilization treatments for sites with mineral soil S concentrations below 0.008%. In mixed conifer stands in northeast Oregon and north-central Washington, Garrison et al. (2000) found that after N, S was the most commonly deficient nutrient.

In both interior British Columbia and northern Alberta, S deficiencies in agricultural, range, and forest soils, particularly Luvisols, have been reported. The first reported S deficiencies were recorded on recently cleared Luvisols in northern Alberta in the 1920s (Bentley et al. 1955). Freyman and van Ryswyk

(1969) found positive responses to S fertilization of native rangeland at sites near Kamloops and Williams Lake. Beaton and MacRae (1967) reported that lodgepole pine fertilization treatments in the Okanagan Valley gave better basal area responses when S was included. Even earlier evidence was cited by Beaton and Soper (1986), whose compilation of response data for forage and grain crops included sites (Fort Fraser, McBride) across a geographical and climatic range similar to that of the present study.

The Ministry of Forests Research Program has been principally responsible for documenting the widespread occurrence of S deficiencies in British Columbia interior conifer stands. In a compilation of research results for 18 lodgepole pine fertilization installations in four biogeoclimatic zones, Brockley (1996) reported that 17 installations responded to N + S treatments, compared with only 11 that responded to N-only treatments. Three-year relative diameter responses were 38% for N + S versus 23% for N-only. These trials included S additions in two contrasting forms, sulphate-S as $(\text{NH}_4)_2\text{SO}_4$ and elemental S (S^0), with the former providing more rapid elevation of foliar S concentrations after 1 year (Brockley and Sheran 1994). Since S^0 must be microbially oxidized before becoming plant-available, these differing short-term foliar responses are not surprising. In the longer term, both S sources were equally effective in improving radial growth over 6 years (Brockley 2004).

This recent research has allowed refinement of the foliar nutrient criteria used to predict fertilization responses by lodgepole pine (Brockley 2000, 2001b). Deficiency thresholds for foliar S and sulphate-S have been lowered from those given by Ballard and Carter (1986), and this recent work has highlighted the importance of analytical methods in defining and applying interpretive criteria (Brockley 2001b). Pre-fertilization foliar N and sulphate-S concentrations and N/S ratios were useful in identifying stands that would respond to S in addition to N, and offer more immediate information for operational decisions than screening trials that assess first-year increases in fascicle mass (Brockley 2000).

Although low S concentrations in mineral soils are widespread across the British Columbia interior, the pattern of fertilization responses to S is less consistent, and research into the underlying controls of S availability in lodgepole pine stands began only recently. Kishchuk (1998) and Kishchuk and Brockley (2002) compared soils from both S-deficient and S-sufficient lodgepole pine stands, based on foliar analysis criteria and fertilization responses. They found that concentrations of total S and most S fractions were low in almost all cases. Based on correlations between foliar S and soil S fractions, this research suggested that S cycling through the ester sulphate-S in the mineral soil was a potential control of S availability and responsiveness to N fertilization.

Although most of the research has addressed lodgepole pine, S deficiency also appears to be a concern for other interior British Columbia conifer species. For both white spruce (*Picea glauca* [Moench] Voss) and Engelmann spruce (*Picea engelmannii* Parry), and their natural hybrids, the limited research suggests that S supply may influence the response to N fertilization (Brockley 1992; Swift and Brockley 1994). In the SBS biogeoclimatic zone of central British Columbia, Wang and Klinka (1997) examined 102 mature white spruce stands across a range of site conditions. They found a slight S deficiency in 26 cases, although they noted that the nutrient diagnosis standards of Ballard and Carter (1986) may not be suitable for mature spruce.

1.4 Sulphur Fertilizers: Forms and Behaviour in Soils

Little research has been done on the behaviour of S-containing fertilizers in forest soils, and inferences must be made from the much larger body of research on agricultural soils, as well as possible analogies between atmospheric deposition and fertilization. Particular considerations in forest fertilization include (1) reliance on high-cost aerial application, which favours infrequent applications and fertilizers with high nutrient concentrations; (2) the long-term nutrient requirements of a crop managed on a time scale of decades; (3) environmental requirements for minimizing nutrient losses to surface waters; and (4) application primarily to surface organic horizons, without incorporation in the underlying mineral soil by tillage.

Although many S-containing fertilizers have been developed, the need for granular solids suitable for aerial application means that only a few of the materials listed by Tisdale et al. (1985) and Hagstrom (1986) would be of practical value as S sources in forest fertilization in British Columbia (Table 2). The need for other nutrients present in a material would also be a consideration. For example, although normal superphosphate contains 13.9% S, other sources would be preferred, since there is little need to supply P to Canadian forest soils. In response to research findings of extensive S deficiencies in the British Columbia interior, operational fertilization treatments have often used a urea-ammonium sulphate blend (10% S) as a standard formulation. The materials listed in Table 2 fall into two groups: (1) sulphate salts, which are water-soluble (although gypsum is only sparingly soluble and needs to be finely divided); and (2) elemental S, which must be microbially oxidized to sulphate to become plant-available.

Sulphate-S materials remain the predominant S sources in the fertilizer industry (Cecotti 1996). Although sulphates are more readily available to plants, rapidly increase foliar S concentrations, and give more immediate growth responses than elemental S sources (Noellemeyer et al. 1981; Solberg and Nyborg 1983; Gupta and McLeod 1984; Janzen and Bettany 1986; Karmanos and Janzen 1991), their lower S concentrations increase shipping and application costs. Also, the high mobility of sulphate fertilizers in soil may not provide long-term amelioration of soil S deficiencies. After 150 years of sulphate-S application as inorganic fertilizers to continuously cropped wheat plots at Rothamstead, U.K., at annual rates ranging from 52 to 249 kg S · ha⁻¹, very little net accumulation of S had occurred (Knights et al. 2000, 2001). Treatments that promoted organic C accumulation through addition of organic manures much more effectively retained fertilizer S, as was also found in an approximately 100-year-old Danish cropping experiment (Eriksen and Mortensen 1999).

TABLE 2 Sulphur-containing fertilizer materials potentially useful in forest fertilization in British Columbia (Tisdale et al. 1985; Hagstrom 1986)

Material	Formula	% S
Ammonium sulphate	(NH ₄) ₂ SO ₄	24.2
Gypsum (hydrated)	CaSO ₄ ·2H ₂ O	18.6
Potassium sulphate	K ₂ SO ₄	17.6
Elemental sulphur	S	95–100 (depending on formulation)
S-coated urea	CO(NH ₂) ₂ + S	Variable (10–20)

Although fertilizers based on elemental S contain much higher S concentrations, the delay resulting from biological oxidation to sulphate may result in slower initial uptake than with more available sulphate forms. Factors and processes influencing rates of elemental S oxidation in soils include fertilizer particle size, soil temperature, soil moisture regime, soil texture (Wainwright 1984; Watkinson and Bolan 1998), previous elemental S fertilization history (Lawrence et al. 1988), as well as freeze-thaw processes (Solberg et al. 1987). However, this slow release characteristic may benefit long-term S availability and uptake (Janzen and Karamanos 1991). More recent development of micronized elemental S products, with granules containing very finely divided primary particles, enable higher rates of oxidation and S supply to crops (Riley et al. 2000; Hu et al. 2002), but the behaviour of these new materials in forest soils has not been studied.

Research on S fertilizer behaviour in forest soils of the temperate and boreal zones is much less developed than for agricultural soils, principally because S deficiencies have not been widely recognized or treated operationally in forests. An older unpublished study did examine the interaction of S-containing fertilizers with a Vancouver Island forest floor during a 14-day aerobic incubation, and found little retention of sulphate from the more available fertilizer forms (ammonium sulphate, thiourea, ammonium sulphate + urea) (Beaton et al. 1969). Much lower rates of sulphate leaching occurred with elemental S amendments, but other possible fates of this added S were not examined. Research on forest soils near Alberta gas processing plants has examined soil properties in relation to elemental S deposition (Maynard et al. 1986; Gower et al. 1991), but the emphasis has been on environmental impacts and reclamation, rather than on S dynamics. European studies of elemental S oxidation in forest soils have examined the roles of environmental factors, especially moisture regime, and previous S deposition history (Lettl et al. 1981a, 1981b; Nevell and Wainwright 1987).

Although studies of forests receiving elevated S inputs from air pollution or localized elemental S particulate deposition can offer some insights, the dynamics of S added to soils with already high S levels may differ from those in S-deficient ecosystems. Watershed-scale experiments in the Black Forest of Germany have used sulphate-S applications at rates of $170 \text{ kg S} \cdot \text{ha}^{-1}$ (as $[\text{NH}_4]_2\text{SO}_4$ or MgSO_4). However, these treatments were in addition to existing soil S reserves exceeding $1000 \text{ kg} \cdot \text{ha}^{-1}$, in areas receiving atmospheric S deposition at rates approximately twice the net annual S uptake by the forest stand (Feger et al. 1991; Feger 1995). Note that the relative importance of processes such as organic S formation and sulphate adsorption would not necessarily be similar if such treatments were applied to forests with demonstrated growth responses to S fertilization, no anthropogenic S deposition, and natural S reserves of $500 \text{ kg} \cdot \text{ha}^{-1}$ or less. Such conditions occur across much of central interior British Columbia.

1.5 Application of Stable Isotope Methods to Sulphur Research in Forests

To trace the fate of fertilizer S in a forest ecosystem, we need to be able to distinguish the added S from the S that is already in the soil and vegetation. Isotopic tracer methods are uniquely effective for doing this, and use different instrumentation and experimental approaches, depending on whether radioactive or stable isotopes are involved. In the case of the former, tracer studies with the radioactive isotope ^{35}S have given important insights into the biological transformations of S in soil-plant systems (e.g., Maynard et al. 1985; McLaren et al. 1985; Blair et al. 1994). However, even if there were no safety

issues, the short half-life (88 days) of ^{35}S is a major disadvantage that limits its usefulness in long-term studies.

For longer-term forest fertilization and nutrient cycling studies, the stable isotope ^{34}S is potentially much more useful. Approximately 95% of the S in the environment occurs as the stable isotope ^{32}S , while 4.22% occurs as the heavier stable isotope ^{34}S . If an artificial source of S, such as fertilizer or air pollution, differs even slightly from natural background levels in the relative amounts of these two isotopes, then it provides a distinctive fingerprint for this source. Very small differences of only a few *parts per thousand*¹ can be readily detected, even in materials that contain extremely low concentrations of S. For this reason, we can use naturally occurring S-containing materials and commercially available fertilizers in our work, providing that their isotopic signatures are sufficiently different from natural background levels in the ecosystem.

The use of stable isotope tracer methods in soil nitrogen cycling studies since the 1950s has dramatically improved understanding of the fate of N fertilizers in forestry and agriculture. A similar potential exists for the use of ^{34}S , but the high cost of artificially enriched S compounds has made field studies prohibitively expensive. With recent improvements to analytical methods, it is now more feasible to conduct field experiments using fertilizer materials with distinctive isotopic signatures as the tracer. Recent studies reviewed by Krouse et al. (1996) have shown that stable isotope techniques are suitable for studying the fate of fertilizer S added to forest soils, providing that its $\delta^{34}\text{S}$ value differs significantly from that of the soil. In the simplest case, where plant uptake occurs from two pools with different $\delta^{34}\text{S}$ values (native plant-available soil S vs. added fertilizer S), the proportion supplied from each source is easily calculated. Mayer et al. (1995) suggested that a difference of 20‰ was necessary, but another German study was able to detect fertilizer S uptake by Norway spruce when the difference was less than 10‰ relative to a plant-available native soil S fraction (Rolland et al. 1991; Giesemann et al. 1995).

Providing that no major isotopic fractionation—discrimination against the heavier isotope—occurs through interaction of fertilizer S with soil components and during biological uptake and transformations, the fate of applied fertilizer can be followed through the soil–plant system. These conditions are generally met in aerobic soils, whereas the largest fractionations, with products greatly depleted in ^{34}S , occur through dissimilatory sulphate reduction in anaerobic soils (Krouse and Tabatabai 1986).

Other transformations relevant to aerobic forest soils involve much smaller fractionations. Sulphate uptake and assimilatory sulphate reduction result in $\delta^{34}\text{S}$ values in plants and bacteria that average 1.5‰ lower than their environmental sulphate source (Trust and Fry 1992). This discrimination was used to explain $\delta^{34}\text{S}$ values in plants and forest floors that averaged 2‰ lower than atmospheric inputs (Novák et al. 2001). Adsorption of sulphate results in only a small fractionation, with Van Stempvoort et al. (1990) finding that adsorption by a Podzolic B horizon enriched the dissolved sulphate in ^{34}S by

¹ $\delta^{34}\text{S}$ is the scale used for expressing the difference in the relative abundances of ^{34}S and ^{32}S between a sample and a standard reference material, in parts per mil (thousand) (‰):

$$\delta^{34}\text{S} (\text{‰}) = \frac{R_{\text{sample}} - R_{\text{standard}}}{R_{\text{standard}}} \times 1000$$

where R is the relative number of ^{34}S and ^{32}S atoms. Positive $\delta^{34}\text{S}$ values indicate enrichment relative to the standard, while negative values indicate depletion.

only 1‰. During microbial oxidation of elemental S in soils, isotopic fractionation is minimal, with the product SO_4^{2-} enriched in the lighter ^{32}S isotope by only 1‰ (McCready and Krouse 1982), making tracer studies feasible with elemental S fertilizers.

Watershed studies (Zhang et al. 1998; Mitchell et al. 2001) have found that sulphate in surface waters tends to be slightly depleted in ^{34}S relative to atmospheric inputs, and fractionations occurring during mineralization of soil organic S are a favoured explanation. In general, mineralization discriminates against the heavier isotope, leading to inorganic SO_4 -S that is isotopically lighter than organic S (Novák et al. 2003). Differing isotopic signatures of soil organic S fractions, with values indicating ^{34}S enrichment (+3.6‰) in ester sulphates and depletion (-1‰) in C-bonded S, have been attributed to microbial mineralization and immobilization (Mayer et al. 1992). Similarly, Schoenau and Bettany (1989) suggested that ^{34}S enrichment of organic sulphates by about 3‰ relative to C-bonded S in a Luvisol forest floor indicated the higher lability of the former fraction.

Several watershed, plot-scale, and laboratory column experiments involving application of sulphates naturally enriched in ^{34}S have been documented in recent reviews (Krouse et al. 1996; Mayer and Krouse 1996; Mitchell et al. 1998). Apart from the need to use S sources with isotopic signatures that are different enough from natural background levels, these studies highlight other important considerations: magnitude of addition relative to existing soil S pools, analytical sensitivity for isotopic compositions of less abundant soil S fractions, and confounding effects of temporal variability in isotopic signatures of other S inputs (e.g., atmospheric). Even where overall S addition is small relative to existing total S pools in the soil, it may still be possible to detect sulphate fertilizer mobility and uptake by tracking $\delta^{34}\text{S}$ changes in plant tissues and plant-available soil sulphate (Pultke et al. 1997). Isotopic analysis of various soil S fractions, either by direct determination or calculation through mass and isotopic balances, can quantify the forms in which added S is retained in the soil (Mayer et al. 1993) and identify situations in which turnover of soil S is occurring despite little net retention of added S (Mitchell et al. 1998).

1.6 Pilot Studies

In designing a fertilization tracer experiment, we need to know background ^{34}S natural abundance values at candidate sites to select fertilizer materials that will have distinctively different isotopic signatures. Using stored samples collected by Kishchuk (1998) from control areas at six lodgepole pine fertilization installations in the SBS biogeoclimatic zone (Northern Interior Forest Region), we determined $\delta^{34}\text{S}$ values for total S in both forest floors and B horizons (Table 3). Based on these data, it appears that fertilizers with $\delta^{34}\text{S}$ values of +20‰ or higher would provide good leverage at most potential sites.

To determine that the stable isotope tracer methodology would work under British Columbia interior stand and soil conditions, we analyzed stored samples of foliage, forest floor, and mineral soil from a fertilization trial established by R.P. Brockley at Cluculz Creek in the Vanderhoof Forest District in 1990. Four treatments included the addition of elemental S at either 50 or 100 kg · ha⁻¹. Samples of the original elemental S fertilizer and current-year foliage taken immediately before fertilization, and after 1 and 3 years, were analyzed for $\delta^{34}\text{S}$. Note that the ^{34}S relative abundance in the fertilizer (0.8‰) was lower than in the pre-treatment foliage (Table 4), as well as in the untreated forest floor and mineral soil at this site (Table 3). As

TABLE 3 ^{34}S natural abundance in unfertilized control areas at existing lodgepole pine fertilization trials, Prince George Forest Region^a

Site	Forest floor		B horizon	
	Total S (%)	$\delta^{34}\text{S}$ (‰)	Total S (mg · kg ⁻¹)	$\delta^{34}\text{S}$ (‰)
Meadow Lake	0.09	5.0	63	-4.2
Tsus Creek	0.10	8.0	69	11.8
Gregg Creek	0.30	13.6	65	3.8
Cluculz Creek	0.08	6.4	60	4.3
Cobb Lake	0.08	6.7	60	-2.6
Bowron	0.09	7.6	67	6.4

^a Total S concentrations are from Kishchuk (1998).

TABLE 4 ^{34}S abundance in lodgepole pine current-year foliage from the Cluculz Creek fertilization installation (E.P. 886.10)

Treatment ^a	Pre-treatment (1990)	$\delta^{34}\text{S}$ (‰)	
		Year 1 (1991)	Year 3 (1993)
Control	5.4	5.6	5.4
200N+50S	5.7	4.9	4.3
200N+100S	6.4	4.7	4.6
400N+50S	6.1	4.6	3.6
400N+100S	6.4	4.6	4.6

Note: $\delta^{34}\text{S}$ for elemental S added in 1990: 0.8 (‰).

^a Treatment indicated by kg N · ha⁻¹ (as urea) and kg S · ha⁻¹ (elemental S).

the elemental S oxidized and was taken up by the trees, the foliar $\delta^{34}\text{S}$ levels gradually dropped. This detectable trend indicates that a stable isotope tracer experiment is feasible, using commonly available fertilizer materials and operationally realistic rates of S addition. This result largely reflects the very low natural concentrations of S in the soil, estimated to be only 300–400 kg · ha⁻¹ in the rooting zone.

1.7 Summary

Sulphur in forest soils in the temperate and boreal zone exists primarily in organic forms, except for some sites receiving high rates of atmospheric deposition. Sulphur availability is therefore controlled primarily by biological and biochemical processes, and to a lesser extent by sulphate adsorption and desorption. In the British Columbia interior, mineral soil S concentrations are among the lowest in the world, likely through the combined effects of parent material characteristics, blockage of natural marine inputs, repeated volatilization losses during fires, and an absence of regionally significant anthropogenic sources. Sulphur behaviour in plants is intimately linked with that of N, and the balance between organic and inorganic S forms in plant tissues is sensitive to both S and N supply. Across the northwestern United States and British Columbia, S deficiencies in managed conifer stand have been most consistently detected at inland sites, and positive responses to S (in combination with N) have been demonstrated in numerous lodgepole pine fertilization trials in the British Columbia central interior. Some of these trials have

compared various fertilizer S sources, but the long-term relative benefits of readily available sulphate-S versus more slowly released elemental S remain to be resolved. Moreover, the ultimate fate of fertilizer S in British Columbia interior soils is poorly understood—the proportions taken up by crop trees and other vegetation, and the forms in which the remainder is retained in the soil, or lost from the ecosystem, are not known. Stable isotope tracer methods have the potential to make an important contribution to understanding S fertilizer behaviour in forest soils and ecosystems. This approach, using commercially produced fertilizer materials with naturally distinctive isotopic signatures, is now technically feasible with recent advances in analytical methods. A pilot study has produced promising results at a representative lodgepole pine site in the British Columbia central interior.

2 OBJECTIVES

The preceding review of knowledge relevant to the forms, cycling, and management of S in British Columbia interior forests has identified some important gaps and uncertainties. Sulphur deficiencies and positive responses to combined N and S fertilization have been demonstrated most consistently for lodgepole pine in the central interior. The leading economic importance of this species (British Columbia Ministry of Forests 2002) warrants further research to improve the predictability of lodgepole pine fertilizer response. Looming age-class imbalances created by the current mountain pine beetle epidemic make it imperative that silviculturists have techniques available to address growth-limiting factors, as salvaged stands are regenerated.

Accordingly, this project addresses three questions:

- How rapidly does fertilizer S uptake by lodgepole pine occur, and how much is retained in the soil, and in which forms?
- Does slow-release elemental S provide greater long-term improvement in S nutrition than more readily available sulphate?
- How large are the tree growth responses to these treatments on a land area basis?

Our approach involves a combination of traditional area-based fertilization response methodology, with stable isotope tracing of S fertilizer uptake and fate in the ecosystem.

3 METHODS

3.1 Study Areas

Two study areas were selected to represent managed lodgepole pine stands across a range of climatic and soil conditions in the Sub-Boreal Spruce biogeoclimatic zone in the southern portion of the former Prince George Forest Region (now Northern Interior Forest Region). Several factors were considered in site selection; adequate stocking; absence of forest health problems; all-weather access; stand age of approximately 20 years; and, where possible, the ability to build on previous research investments.

3.1.1 Holy Cross site The Holy Cross (HC) installation is at km 138 on the Holy Cross Forest Service Road in the Vanderhoof Forest District (Figure 2) in opening 4, mapsheet 93F.004. The site is at about 920 m elevation in the Dry Cool Sub-Boreal Spruce (SBSdk) subzone, on a rolling drum-linized morainal blanket over bedrock, with local slopes ranging from level to 15% on various aspects. The nearest weather station (Fraser Lake north shore, 54° 4'N, 124° 51'W, 674 m elevation) reported a 1971–2000 mean annual temperature of 3.1°C and mean annual precipitation of 521.1 mm (63% falling as rain) (Environment Canada 2004). Based on slope position, soil properties, and understorey vegetation, this site most closely matches the 05 (Sxw–Spirea–Feathermoss) and 01 (Sxw–Spirea–Purple peavine) site series (DeLong et al. 1993). Estimated lodgepole pine site index values (SI_{50} = height at breast height age 50) are 19.5 and 18.9 m for the 01 and 05 site series, respectively (British Columbia Ministry of Forests 2003).

The dominance of lodgepole pine in this subzone reflects the disturbance history of the drier southwestern portion of the Prince George Forest Region. The opening was logged in 1982, regenerated naturally to lodgepole pine after site preparation by chain-dragging in 1982, and juvenile spaced in 1996.

Examination of road cuts and inspection pits on and near the HC installation indicated that the dominant soils are Orthic or Brunisolic Gray Luvisols, as well as their associated Gleyed subgroups. All exhibit strong morphological development of a Bt horizon, which forms a barrier to root penetration below 50–70 cm. On level or gently sloping sites, mottled horizons occur above the Bt, suggesting temporary perched water tables that likely occur during the spring snowmelt (Table 5). Soil reaction is acidic throughout the forest floor and in the A and B horizons (Table 6), but the underlying loamy parent material is calcareous, and secondary carbonates occur approximately 1 m from the soil surface. Although this area is not covered by a published

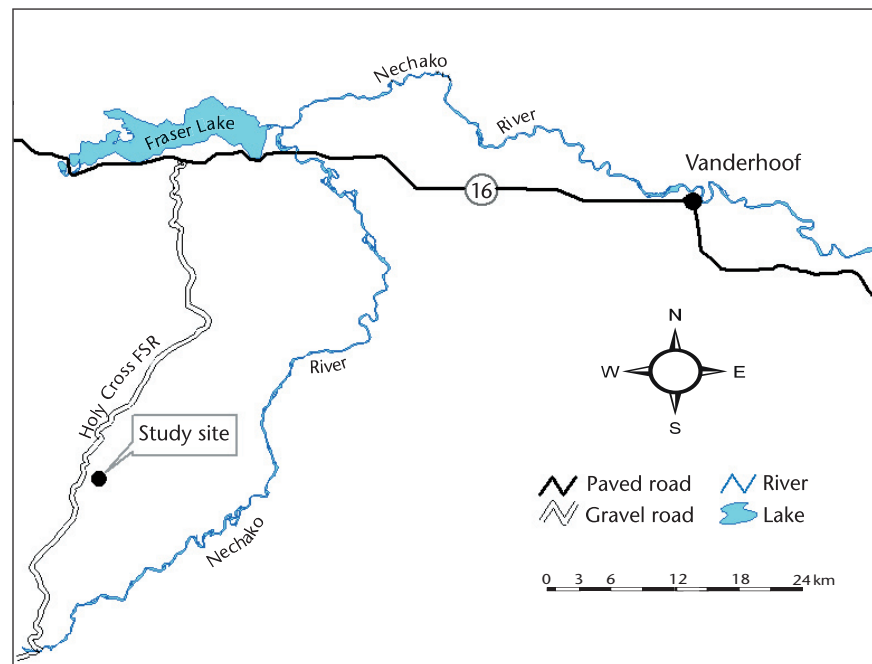


FIGURE 2 Holy Cross research site location (km 138, Holy Cross Forest Service Road, Vanderhoof Forest District).

TABLE 5 Morphological description of representative Gleyed Brunisolic Gray Luvisol, Holy Cross site, with organic horizon nomenclature according to Green et al. (1993). (Location: 53°47'23.6"N, 124°53'05.3"W, elevation; 929.6 m (± 12.7 m); 21.0 m from centre of plot 8 @ 30°)

Horizon	Depth (cm)	Description
Ln	8–7	Fresh pine needle litter; moist; single particle; loose; acerose; no roots; 1 cm thick; extremely acid.
Lv	7–6	Pine needle litter; moist; single particle; loose; acerose; no roots; 0–1 cm thick; extremely acid.
Fm	6–2	Moist; very dark grey (7.5 YR 3/1 m); weak, non-compact matted; friable; felty; abundant very fine and fine, plentiful medium and coarse roots; abundant mycelia; 3–5 cm thick; extremely acid.
Faw	5–2	Moist; dark reddish brown (5 YR 3/4 m); single particle; friable; ligneous; abundant very fine and fine, plentiful medium and coarse roots; common mycelia; 0–6 cm thick; extremely acid.
Hh	2–0	Moist; black (5 YR 2.5/1 m); moderate, fine granular; friable; greasy; abundant very fine and fine, plentiful medium and coarse roots; charcoal present; 1–3 cm thick; strongly acid.
Bm1	0–22	Dark greyish brown (10 YR 4/2 m); sandy loam; weak to moderate, medium subangular blocky; friable; few, medium and coarse, abundant very fine and fine roots; 20–25% gravel, 15–20% cobbles; gradual, wavy boundary; 15–30 cm thick; very strongly acid.
Bm2	22–40	Grayish brown (10 YR 5/2 m); sandy loam; moderate medium subangular blocky; friable; plentiful fine and medium roots; 20–25% gravel, 10–15% cobbles; clear, wavy boundary; 15–25 cm thick; very strongly acid.
Bm3	40–52	Brown (10 YR 4/3 m); loamy sand; moderate, medium subangular blocky; friable; few fine roots; 20–25% gravel, 10–15% cobbles; clear, wavy boundary; 5–15 cm thick; strongly acid.
Aeg	52–68	Dark greyish brown (10 YR 4/2 m); loamy sand; common, fine strong brown (7.5 YR 4/6 m) prominent mottles; moderate, medium platy; friable; few fine roots; 20–25% gravel, 10–15% cobbles; clear, wavy boundary; 15–22 cm thick; strongly acid.
Bt1	68–84	Brown (7.5 YR 4/2 m); sandy loam; strong, medium, and coarse subangular blocky; firm; few, fine roots; common, moderately thick clay films; 20–25% gravel, 10–15% cobbles; gradual wavy boundary; 20–30 cm thick; medium acid.
Bt2	84–105	Brown (7.5 YR 4/2 m) and light grey (10 YR 7/2 m); sandy loam; moderate, medium and coarse subangular blocky; firm; no roots; common, moderately thick clay films; 20–25% gravel, 10–15% cobbles; gradual, wavy boundary; 20–30 cm thick; medium acid.
BCk	105–125	Brown (7.5 YR 4/2 m); sandy loam; moderate, coarse subangular blocky; firm; no roots; few, moderately thick clay films; weak effervescence; 20–25% gravel, 10–15% cobbles; gradual, wavy boundary; 20–25 cm thick; neutral.
Cca	125–135+	Very dark greyish brown (10 YR 3/2 m); loam; massive; firm; no roots; moderate effervescence; discontinuous, spotted, common, medium, irregular, friable, pink (7.5 YR 7/3 m) secondary carbonates; 20–25% gravel, 10–15% cobbles; mildly alkaline.

soil survey report, the soils most closely resemble the Deserters soil association described in the adjacent Manson River–Fort Fraser area (Epp and Kenk 1983).

3.1.2 Kenneth Creek site The Kenneth Creek (KC) installation is at km 84.5 on the Beaver–Bowron Forest Service Road in the Prince George Forest District (Figure 3) in opening 9, mapsheet 93H.082. This opening includes plots established in 1993 for E.P. 886.13, and the installations for both are on a level

TABLE 6 Physical and chemical characteristics of Gleyed Brunisolic Gray Luvisol, Holy Cross site (Al_p , Fe_p = pyrophosphate-extractable Al, Fe; CEC = cation exchange capacity)

Horizon	Depth (cm)	%							
		Sand	Silt	Clay	C	N	S	Al_p	Fe_p
Ln	8–7				49.7	0.46	0.0294		
Lv	7–6				47.3	0.79	0.0505		
Fm	6–2				38.3	1.11	0.0670		
Faw	5–2				52.3	0.92	0.0672		
Hh	2–0				38.7	1.39	0.0954		
Bm1	0–22	57.9	38.5	3.6	0.7	0.05	0.0034	0.072	0.115
Bm2	22–40	58.1	36.9	5.1	0.4	0.03	0.0025	0.039	0.090
Bm3	40–52	75.7	22.1	2.3	0.3	0.02	0.0030	0.033	0.082
Aeg	52–68	77.3	21.6	1.0	0.3	0.02	0.0023	0.029	0.073
Bt1	68–84	56.9	31.5	11.6	0.1	<0.01	0.0029	0.034	0.046
Bt2	84–105	61.7	28.4	9.9	0.1	<0.01	0.0030	0.032	0.043
BCK	105–125	54.7	37.2	8.2	0.1	<0.01	0.0038	0.023	0.031
Cca	125–135+	51.4	39.0	9.6	0.3	<0.01	0.0054	0.011	0.017

Horizon	Depth (cm)	pH (H ₂ O)	pH (CaCl ₂)	Exchangeable cations (cmol (+) · kg ⁻¹)							
				Al	Ca	Fe	K	Mg	Mn	Na	CEC
Ln	8–7	4.09	3.68	2.83	9.47	0.05	2.06	6.80	2.58	0.13	23.92
Lv	7–6	4.42	4.04	1.16	24.36	0.04	2.57	5.82	2.33	0.12	36.39
Fm	6–2	4.78	4.40	0.60	42.55	0.04	1.83	5.64	2.12	0.12	52.91
Faw	5–2	4.87	4.21	0.12	44.57	0.02	1.46	5.24	0.54	0.11	52.06
Hh	2–0	5.60	5.33	0.04	122.48	0.01	1.82	7.78	0.85	0.18	133.18
Bm1	0–22	5.86	4.87	0.26	5.35	0.03	0.21	0.91	0.01	0.08	6.85
Bm2	22–40	6.09	5.00	0.13	5.02	0.02	0.11	1.12	0.01	0.10	6.52
Bm3	40–52	6.21	5.09	0.08	5.20	0.01	0.13	1.25	0.01	0.09	6.77
Aeg	52–68	6.41	5.08	0.05	5.60	0.01	0.17	1.40	0.01	0.11	7.34
Bt1	68–84	6.94	5.58	0.01	9.91	0.00	0.27	2.96	0.02	0.18	13.35
Bt2	84–105	6.48	5.93	0.01	10.28	0.01	0.15	3.05	0.02	0.17	13.69
BCK	105–125	7.96	7.23	0.00	10.06	0.00	0.19	2.25	0.00	0.14	12.66
Cca	125–135+	8.29	7.78	0.01	11.60	0.00	0.21	2.38	0.00	0.15	14.35

to slightly undulating sandy glacio-fluvial terrace north of the Bowron River at approximately 810 m elevation. Considerably wetter than HC, this site is in the Willow Wet Cool Sub-Boreal Spruce (SBSwk1) variant, adjacent to one of the westernmost outliers of the Very Wet Cool Interior Cedar–Hemlock (ICHvk2) variant. The nearest weather station (Aleza Lake, 54°7'N, 122°4'W, 625 m elevation) reported a 1953–1979 mean annual temperature of 3.0°C and mean annual precipitation of 902.8 mm (62% falling as rain) (Environment Canada 2004). The coarse-textured soil parent materials create one of the drier soil moisture regimes within this biogeoclimatic variant, with site conditions corresponding most closely to the o3 site series (Pl–Huckleberry–Velvet-leaved blueberry) (DeLong 2003). This site was logged in 1980, broadcast burned in 1982, and planted to lodgepole pine in 1983. The current site index of this lodgepole pine stand (SI_{30}) is 23 m, based on tree measurements using the criteria of Nigh (1997).

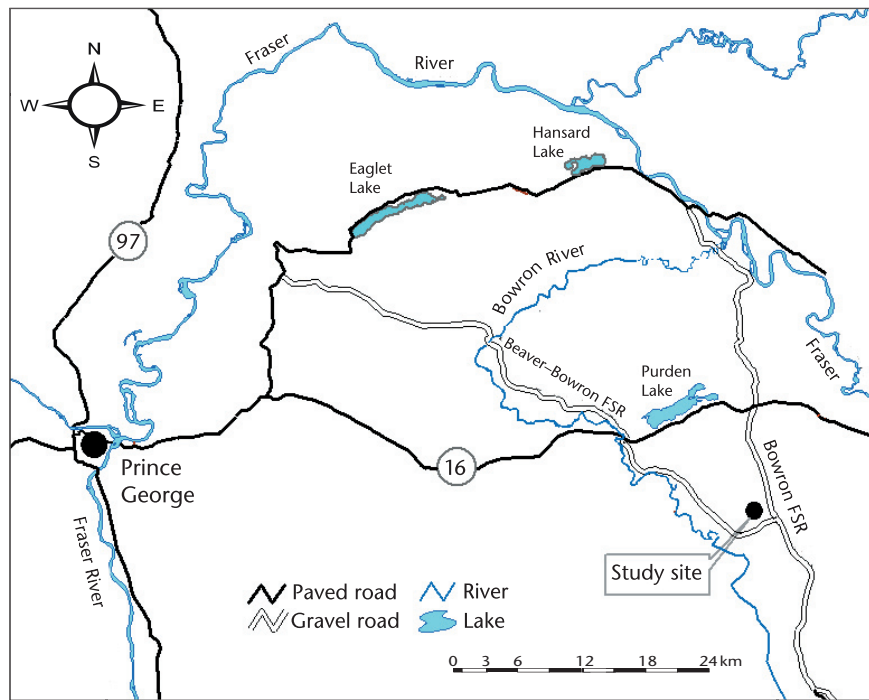


FIGURE 3 *Kenneth Creek research site location (km 84.5, Beaver–Bowron Forest Service Road, Prince George Forest District).*

The representative pedon examined in detail at this site (Tables 7 and 8) is morphologically similar to the Humo-Ferric Podzols that predominate on coarse-textured materials in the wetter SBS subzones. In this specific case, only the B-horizon thickness fails to meet the requirements of the Podzolic Order, so this pedon would be classified as an Eluviated Dystric Brunisol (Soil Classification Working Group 1998). Noteworthy characteristics are the low pH values throughout the pedon, reflecting the combination of coarse textures and a strongly leaching environment, and the low concentrations of C and organic matter–associated nutrients (N, S, P) in the mineral horizons. A modest increase in clay content in the uppermost horizons, and the apparent formation of 2:1 type clay minerals at the expense of mica and chlorite are indicators of chemical weathering (Arocena and Sanborn 1999). Soils at this site most closely match the Ptarmigan soil association described in the Barkerville soil survey report (Lord and Green 1985), although with a much lower coarse fragment content.

3.2 Plot Establishment and Experimental Design

Areas of uniform site and stand conditions were identified for plot locations at both study sites. The HC site had large areas of uneven lodgepole pine stocking, and locations for only 16 treatment plots could be identified. More uniform stand conditions at KC enabled 24 suitable plot locations to be found.

To estimate stand density, temporary sample plots were established at each selected plot location. At HC, estimated stand densities ranged from about 1800 to 2200 stems per hectare. Stand densities at KC were lower, ranging from approximately 1300 to 1700 stems per hectare. These stand density estimates were used to select suitable plot sizes for each of the study sites.

TABLE 7 Morphological description of representative Eluviated Dystric Brunisol pedon, Kenneth Creek site

Horizon	Depth (cm)	Description
L-H	2-0	Litter and semi-decomposed organic material; abrupt, wavy boundary; extremely acid.
Ae	0-5	Light brownish grey (10YR 6/2 m); sandy loam; 0% coarse fragments; weak fine platy; very friable; plentiful fine and medium horizontal roots; abrupt wavy boundary; 3-8 cm thick; extremely acid.
Bf	5-12	Strong brown (7.5YR 4/6 m); loamy sand; 0% coarse fragments; massive; friable; plentiful fine and medium, horizontal roots; gradual wavy boundary; 4-13 cm thick; extremely acid.
Bfj	12-27	Brown (10YR 4/3 m); sand; 0% coarse fragments; massive; friable; plentiful fine and medium, few coarse, horizontal roots; gradual wavy boundary; 9-15 cm thick; very strongly acid.
Bm	27-60	Olive brown (2.5Y 4/4 m); sand; 0% coarse fragments; single grain; very friable; few fine and medium oblique roots; diffuse wavy boundary; 30-40 cm thick; very strongly acid.
BC	60-100	Greyish-brown (2.5Y 5/2 m); sand; 0% coarse fragments; single grain; very friable; no roots; diffuse wavy boundary; 40-50 cm thick; strongly acid.
C	100-125+	Dark greyish brown (2.5Y 4/2 m); sand; 0% coarse fragments; single grain; very friable; no roots; very strongly acid.

TABLE 8 Physical and chemical characteristics of Eluviated Dystric Brunisol, Kenneth Creek site (Al_p , Fe_p = pyrophosphate-extractable Al, Fe; CEC = cation exchange capacity)

Horizon	Depth (cm)	%								Avail. P ($mg \cdot kg^{-1}$)
		Sand	Silt	Clay	Al_p	Fe_p	C	N	S	
L-H	2-0	n.d.	n.d.	n.d.	n.d.	n.d.	32.07	0.64	0.1128	32.7
Ae	0-5	73.0	23.8	3.2	0.037	0.033	0.61	0.03	0.0043	3.5
Bf	5-12	81.1	13.1	5.7	0.384	0.421	0.89	0.07	0.0076	51.5
Bfj	12-27	91.2	7.0	1.8	0.288	0.128	0.39	0.05	0.0047	58.1
Bm	27-60	92.8	5.6	1.5	0.098	0.068	0.45	0.06	0.0039	32.9
BC	60-100	96.3	3.4	0.3	0.045	0.046	0.14	0.03	0.0044	12.8
C	100-125+	97.3	2.1	0.6	0.086	0.073	0.13	0.01	0.0044	17.9

Horizon	Depth (cm)	pH (H_2O)	pH ($CaCl_2$)	Exchangeable cations ($cmol (+) \cdot kg^{-1}$)							
				Al	Ca	Fe	K	Mg	Mn	Na	CEC
L-H	2-0	4.23	3.58	2.48	7.84	0.39	0.64	1.15	0.71	0.55	13.75
Ae	0-5	4.20	3.44	1.87	1.09	0.12	0.03	0.33	0.02	0.05	3.52
Bf	5-12	4.84	4.35	0.52	0.28	0.04	0.03	0.05	0.02	0.10	1.05
Bfj	12-27	5.49	5.01	0.04	0.21	0.01	0.01	0.02	0.01	0.10	0.39
Bm	27-60	5.67	4.95	0.03	0.46	0.00	0.02	0.03	0.01	0.09	0.64
BC	60-100	5.98	5.11	0.02	0.71	0.00	0.03	0.09	0.00	0.05	0.90
C	100-125+	5.82	4.99	0.03	0.39	0.00	0.02	0.05	0.00	0.04	0.53

At HC, each circular treatment plot consisted of a 7-m radius measurement area (0.015 ha) surrounded by a 3-m buffer. The selected measurement area was large enough to contain 25 suitable, well-spaced dominant or co-dominant lodgepole pine “crop” trees, representing a stand density of 1600 stems per hectare. Assessment plots at KC were 8.5 m in radius (0.023 ha) surrounded by a 3-m buffer, ensuring 25 “crop” trees at a stand density of 1100 stems per hectare.

The outer treatment plot perimeters and inner measurement plot perimeters were marked at 45° intervals with wooden survey stakes. A total of 25 suitable lodgepole pine trees were selected within each measurement area. Crop tree selection was based on factors such as tree health, size, and spacing. A painted band was permanently marked on the bole of each “crop” tree at a point 1.30 m above the ground. A numbered tree tag was affixed with a cable tie approximately 25 cm above the painted band. Tree numbering ranges for each plot are given in Tables 10 and 11. Using about the same inter-tree spacing as in the central assessment plot, suitable well-spaced trees were also selected within the buffer area of each plot. All “extra” trees within each treatment plot were marked for removal following plot establishment.

Four treatments with four replicates were established at both sites, with two additional treatments at KC. At both sites at least 10 m of untreated and unthinned stand separated adjacent plots. The plot layouts are shown in Figures 4 and 5, and the location coordinates for the tie-points in Table 9.

All S additions were at 100 kg S · ha⁻¹, and N (as urea) at 300 kg N · ha⁻¹, rates that are typical of operational and research fertilization treatments in the central interior. As a minimum, we wanted to compare the behaviour of S in two forms (relatively soluble sulphate vs. elemental S) when added with N. Pre-testing of more than 30 commercial S fertilizer materials from across North America identified three promising candidate tracer materials:

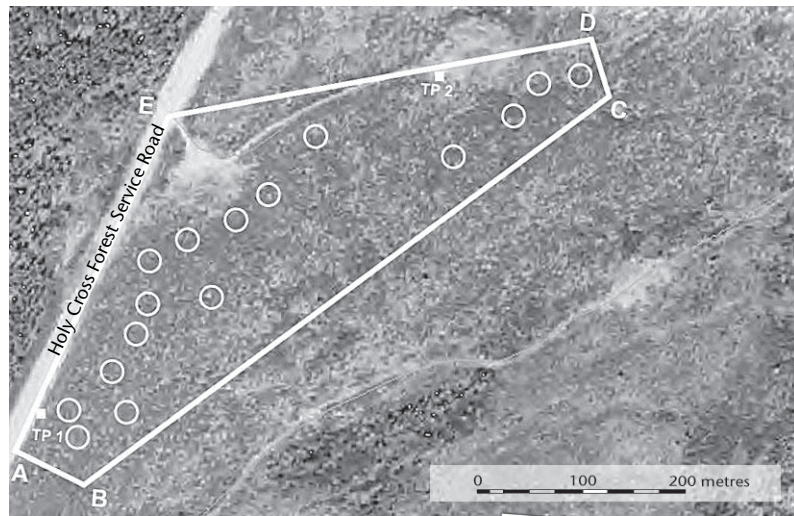
- K₂SO₄ (IMC Kalium, Evergro Fertilizers, Delta, British Columbia), δ³⁴S = 17.5‰
- “Shell elemental S” (Shell Canada, Cremona, Alta.), δ³⁴S = 22.6‰
- “Agrimax elemental S”: Sulfur 95 – commercial elemental S fertilizer (Agrimax Corporation, Calgary, Alta.), δ³⁴S = 19.3‰

Based on isotopic analysis of preliminary samples of pine foliage, these fertilizers differ from background δ³⁴S values at both sites by > 10‰, making them suitable tracers at the planned addition rates.

The treatments at both sites compare either sulphate-S or elemental S (added in combination with N), with N-only and unfertilized controls.

TABLE 9 Location coordinates for installation tie-points: Holy Cross and Kenneth Creek sites

Site	Tie-point	Latitude	Longitude
Holy Cross	1	53°47'19.6"	124°53'13.8"
	2	53°47'30.7"	124°52'54.8"
Kenneth Creek	1	53°49'21.6"	121°47'02.2"
	2	53°49'15.4"	121°46'58.3"
	3	53°49'03.6"	121°47'07.2"



TP 1 to Plot 1	42 m @ 128°
Plot 1 to Plot 2	27 m @ 342°
Plot 2 to Plot 3	52 m @ 93°
Plot 3 to Plot 4	38 m @ 345°
Plot 4 to Plot 5	40 m @ 33°
Plot 5 to Plot 6	30 m @ 20°
Plot 6 to Plot 7	41 m @ 2°
Plot 7 to Plot 8	68 m @ 124°
Plot 8 to Plot 9	59 m @ 340°
Plot 9 to Plot 10	49 m @ 70°
Plot 10 to Plot 11	39 m @ 50°
Plot 11 to Plot 12	70 m @ 41°
Plot 12 to Plot 13	127 m @ 101°
Plot 13 to Plot 14	69 m @ 56°
Plot 14 to Plot 15	37 m @ 40°
Plot 15 to Plot 16	37 m @ 81°
Plot 13 to TP 2	98 m @ 349°

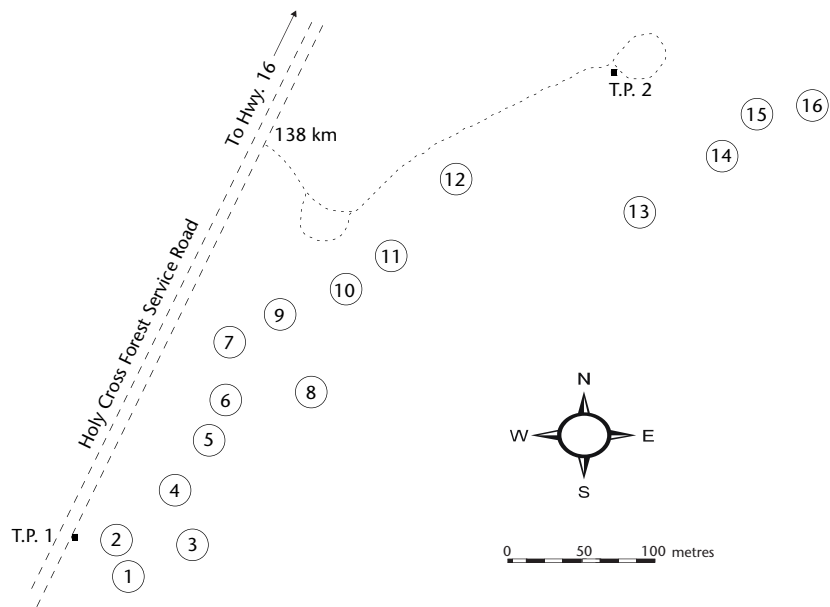
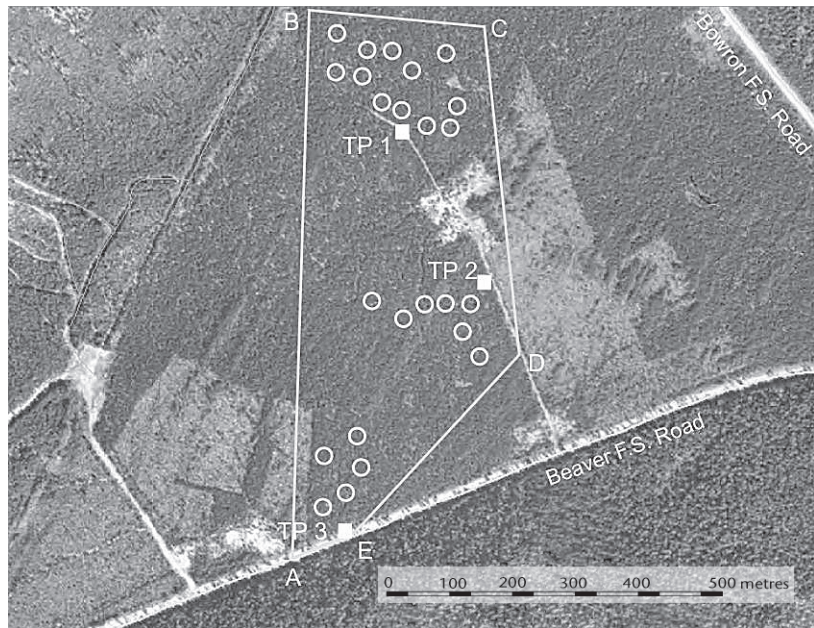


FIGURE 4 Plot layout map, Holy Cross installation.

N-only and N + elemental S treatments included sufficient KCl to remove any confounding effect of K in K_2SO_4 . Treatments 1–4 were used at both sites, with two additional treatments (5 and 6) included in the extra plots at KC:

1. Control
2. N + KCl
3. N + K_2SO_4
4. N + Shell elemental S + KCl
5. N + Agrimax elemental S + KCl
6. Agrimax elemental S

Each site comprised a single factor experiment with a randomized complete block design. The initial diameter at breast height (dbh) measurement data (Section 3.3.1) were used to assign plots to one of four blocks before treatment allocation. The 16 plots at HC were ranked in order of largest to



TP 1 to Plot 1	38.0 m @ 10°
Plot 1 to Plot 2	31.5 m @ 313°
Plot 2 to Plot 3	47.5 m @ 343°
Plot 3 to Plot 4	37.5 m @ 300°
Plot 4 to Plot 5	56.0 m @ 22°
Plot 5 to Plot 6	51.5 m @ 140°
Plot 6 to Plot 7	37.0 m @ 112°
Plot 7 to Plot 8	40.5 m @ 156°
Plot 8 to Plot 9	56.5 m @ 84°
Plot 9 to Plot 10	80.5 m @ 188°
Plot 10 to Plot 11	33.3 m @ 225°
Plot 11 to Plot 12	32.3 m @ 296°
Plot 12 to TP 1	37.6 m @ 264°
TP 2 to Plot 13	46.0 m @ 229°
Plot 13 to Plot 14	80.0 m @ 191°
Plot 14 to Plot 15	44.0 m @ 347°
Plot 15 to Plot 16	49.0 m @ 349°
Plot 16 to Plot 17	31.0 m @ 287°
Plot 17 to Plot 18	38.0 m @ 255°
Plot 18 to Plot 19	53.5 m @ 320°
TP 3 to Plot 20	59.5 m @ 326°
Plot 20 to Plot 21	39.5 m @ 80°
Plot 21 to Plot 22	44.0 m @ 52°
Plot 22 to Plot 23	47.0 m @ 13°
Plot 23 to Plot 24	57.0 m @ 260°

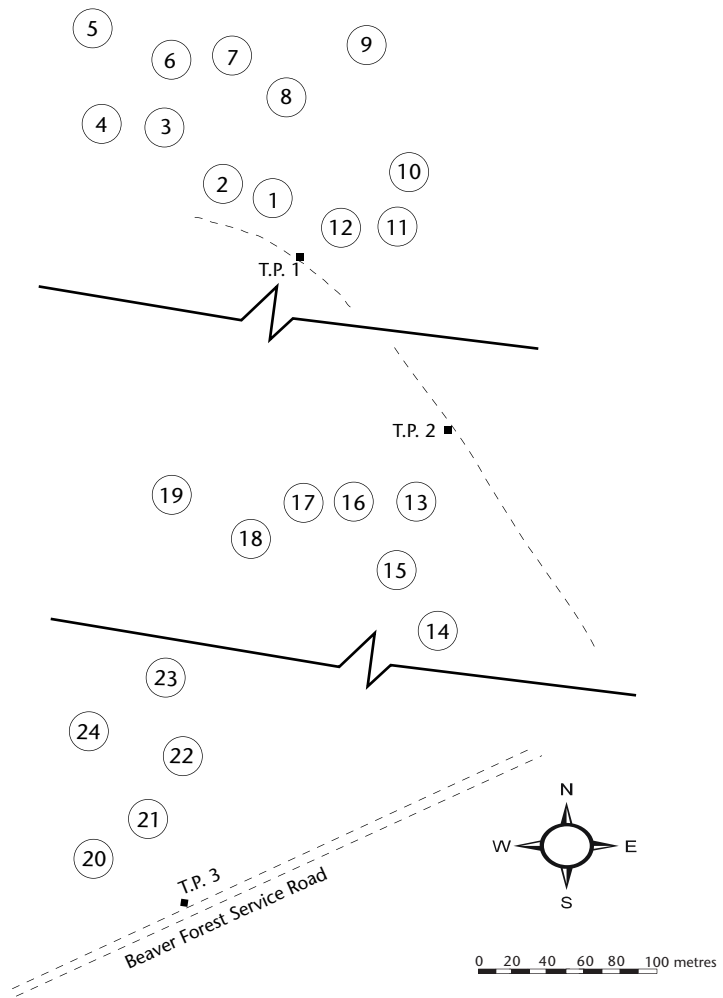


FIGURE 5 Plot layout map, Kenneth Creek installation.

TABLE 10 *Treatment allocation and crop tree numbering sequence at Holy Cross installation*

Plot	Treatment	Tree numbering range
1	N + KCl	501–525
2	N + KCl	526–550
3	N + K ₂ SO ₄	551–575
4	Control	576–600
5	N + K ₂ SO ₄	601–625
6	Control	626–650
7	N + S ⁰ + KCl	651–675
8	Control	676–700
9	Control	701–725
10	N + KCl	726–750
11	N + S ⁰ + KCl	751–775
12	N + K ₂ SO ₄	776–800
13	N + S ⁰ + KCl	801–825
14	N + K ₂ SO ₄	826–850
15	N + KCl	851–875
16	N + S ⁰ + KCl	876–900

Note: S⁰ = elemental S; K₂SO₄ average δ³⁴S = +17.7‰

TABLE 11 *Treatment allocation and crop tree numbering sequence at Kenneth Creek installation*

Plot	Treatment	Tree numbering range
1	N + Agrimax + KCl	1301–1325
2	Agrimax only	1326–1350
3	N + Agrimax + KCl	1351–1375
4	N + S ⁰ + KCl	1376–1400
5	Control	1401–1425
6	N + K ₂ SO ₄ (<i>18.3‰</i>)	1426–1450
7	N + S ⁰ + KCl	1451–1475
8	N + KCl	1476–1500
9	N + K ₂ SO ₄ (<i>17.3‰</i>)	1501–1525
10	Agrimax only	1526–1550
11	Control	1551–1575
12	N + KCl	1576–1600
13	N + Agrimax + KCl	1601–1625
14	Control	1626–1650
15	N + S ⁰ + KCl	1651–1675
16	Agrimax only	1676–1700
17	N + Agrimax + KCl	1701–1725
18	N + KCl	1726–1750
19	N + S ⁰ + KCl	1751–1775
20	N + K ₂ SO ₄ (<i>17.7‰</i>)	1776–1800
21	Agrimax only	1801–1825
22	N + KCl	1826–1850
23	Control	1851–1875
24	N + K ₂ SO ₄ (<i>17.7‰</i>)	1876–1900

Note: S⁰ = elemental S; K₂SO₄ δ³⁴S values differed slightly between plots as noted in italics next to treatment name.

smallest mean dbh and then grouped into four blocks, with the four plots within each block having similar mean dbh. Each of the four treatments was then randomly assigned to one of the plots within each block (Table 10). The same procedure was used at KC, with each of the six treatments randomly assigned to one of the six plots within each block (Table 11).

Fertilizers were pre-weighed and bagged into amounts needed for one-eighth segments of each plot, and applied by hand during the first 2 weeks of November 2002. Unusually mild weather allowed application under snow-free conditions at KC and on a melting 2 cm thick snow layer at HC. The Shell elemental S was applied as raw hemispherical pellets, about 5 mm in diameter and 2 mm thick. It would have been preferable to grind this material before application to accelerate the rate of oxidation. The $\delta^{34}\text{S}$ values of K_2SO_4 among the individual 25-kg bags from the supplier varied, so the bags used on each plot were noted.

3.3 Measurements

3.3.1 Tree growth The dbh, total height, and height to the base of the live crown of all 25 tagged trees within the central assessment area of each treatment plot were measured in October 2002 at both study sites. Trees within the treated buffer of each plot were not measured. Diameters were measured with a steel diameter tape at the top of a painted band located approximately 1.30 m above the ground. Tree heights were measured with a Forestor Vertex[®] hypsometer (Forestor Instrument AB, Taby, Sweden). A telescoping height pole was used to measure the height to the base of the live crown of each tagged tree. The base of the live crown was defined as the lowest live branch that forms part of the uninterrupted live crown. Standard damage and form codes developed by the Ministry of Forests were used to describe the condition of each tagged tree.

3.3.2 Soil sampling The soil sampling design was influenced by previous studies of soil variability at both the KC installation of E.P. 886.13 and other lodgepole pine sites in the British Columbia central interior (Sanborn et al. 2001), as well as by cost considerations due to the S stable isotope methodology used in this study. Pre-treatment sampling was carried out at 15 random points in each treatment plot. If a sampling point landed on a stump, boulder, or bedrock outcrop, the samples were collected from the nearest location that was physically possible to sample.

Three horizons or depths were sampled at both installations: litter (consisting mostly of pine needle litter on the forest floor surface), FH (partially decomposed and humified forest floor organic materials sampled down to the contact with the mineral soil), and the uppermost 20 cm of the mineral soil (sampled with a stony soil auger). Mineral soils were sampled by depth intervals rather than by horizon. Although this approach combined horizons with differing chemical properties, particularly in the 0–20 cm depth interval at the KC site, the scale of the sampling effort required that several individuals be involved, which would have posed difficulties if consistent recognition of horizon boundaries had been required.

Humus forms at both sites were either mor or moder (Green et al. 1993), with abrupt transitions to the underlying mineral soil, enabling consistent identification of the lower boundary of the forest floor in most cases. Woody materials were included in the FH samples if the material could be crushed or broken between the fingers in its field-moist condition. This restriction largely excluded slash from the 1996 thinning at the HC site. At the KC site,

the absence of coarse fragments allowed soil sampling to greater depth, and the 20–40 cm depth of the mineral soil was also sampled. All samples were stored at 4°C until air-dried. Litter and FH samples were individually ground with a hammermill, then a single equally weighted composite of each sample type was prepared for each plot. Mineral soils were sieved (2 mm) and composited in similar fashion.

3.3.3 Foliar sampling Current-year pine foliage was collected from one lateral branch in the upper third of the live canopy of 10 dominant or codominant trees from each plot. These trees were flagged for future resampling. Samples were frozen following field collection, and then oven-dried at 70°C for 20 h before analysis. One composite sample consisting of equal amounts of foliage from each of the 10 trees per treatment plot was prepared for chemical analysis, and ground in an electric coffee grinder.

3.3.4 Chemical and physical analysis methods The Ministry of Forests analytical chemistry laboratory performed the following analyses:

- 1) for litter, FH, and mineral soils:
 - total C and N (also for foliage) (LECO CHN-600 Elemental Analyzer),
 - total S (also for foliage) (LECO SC-132 S Analyzer),
 - mineralizable N (anaerobic incubation; Powers 1980),
 - extractable P (Bray P₁ method; Kalra and Maynard 1991),
 - extractable inorganic sulphate-S (foliage, litter, and FH extracted with 0.01 M NH₄Cl; mineral soils with 500 mg P·L⁻¹ as Ca(H₂PO₄)₂·H₂O; SO₄²⁻ determined by ion chromatography),
 - pH (Fisher Accumet pH Meter (model 915) with a Broadley-James E-9405-EC1-A03BC soil probe pH electrode, using soil/water ratios of 1:1 for mineral soils and 1:2 for forest floors, and a soil/0.01 M CaCl₂ ratio of 1:2 for both mineral soils and forest floors, and
 - CEC (BaCl₂ method; Hendershot and Duquette 1986), with exchangeable cations (Ca, Mg, K, Na, Fe, Al, Mn) determined by inductively coupled argon plasma-atomic emission spectroscopy (ICAP).
- 2) for foliage, litter, and FH samples:
 - total elemental concentrations (Al, B, Ca, Cu, Fe, K, Mn, Mg, P, S, Zn) were determined by microwave digestion in HNO₃-HCl-H₂O₂ (Kalra and Maynard 1991) and ICAP.
- 3) for the mineral soil horizons of representative pedons:
 - Fe and Al were extracted with Na-pyrophosphate (McKeague 1967) and determined by ICAP.

All data were reported on an oven-dry basis.

The two methods (combustion vs. wet digestion-ICAP) used for determining total S in the litter and FH samples yielded somewhat different mean values, and the direction of difference was not consistent for both sites. The performance of these methods has not been compared systematically for forest floor horizons, but Brockley (2001b) noted that for foliar samples the wet digestion-ICAP method tends to give slightly lower values. Pending more detailed comparisons, total S determinations by both methods are reported for litter and FH samples.

Particle-size analysis of all mineral soil composites and the horizon samples from the KC site was performed with the pipette method by Soilcon Laboratories (Richmond, British Columbia), with Fe-removal pre-treatment of all samples. Pipette analysis of the horizon samples from the HC site was performed in the University of Northern British Columbia soil science laboratory, with calcareous horizons pre-treated with HCl to remove carbonates.

Detailed S fractionations were performed on horizon samples from the representative pedon sampled at each site. The KC Brunisol pedon was analyzed at Technische Universität München. Total S was determined using a LECO SC-444 analyzer. HI-reducible S was analyzed with a Johnson/Nishita apparatus (Freney 1961). Water-soluble SO_4 -S was quantified by shaking 5 g sieved (forest floor) or ground (litter) sample material with 50 mL deionized water for 18 h in an end-over-end shaker. The solution was filtered by membrane filtration (cellulose acetate filter 0.45 μm pore size; Schleicher and Schuell Comp., Dassel, Germany). Subsequently, the SO_4^{2-} concentration in the filtrates was determined with an ion chromatograph (DIONEX 2020i; Sunnyvale, California, U.S.A.). From the analytically determined S fractions, the concentrations of various S forms in the forest floor samples were calculated as follows. C-bonded S was estimated as total S less HI-reducible S, ester sulphate-S was calculated as HI-reducible S less inorganic S, which was considered equal to water-soluble SO_4 -S. For the mineral soil, inorganic S was assumed to be equal to NH_4F -extractable SO_4 -S (Prietzl and Hirsch 2000). The methods used for speciation of inorganic S in the mineral soil are described in detail in Prietzl et al. (2001).

A similar fractionation scheme was used for the horizon samples from the representative HC pedon, with total S and extractable inorganic SO_4 -S determined at the Ministry of Forests laboratory (as described above), and HI-reducible S by the Johnson/Nishita method at Pacific Soil Analysis Inc. (Richmond, British Columbia). C-bonded S was calculated as the difference between total S and HI-reducible S, and ester sulphate-S as the difference between HI-reducible S and extractable inorganic sulphate-S. (For the mineral soil horizons, extractable inorganic sulphate-S would include both adsorbed and soluble sulphate-S.)

3.3.5 Stable isotope analysis methods HI-reducible S, comprising organic sulfate and inorganic sulfate, was analyzed on fine-ground foliage, litter, forest floor, and (unground) mineral soil samples using the method of Freney (1961).

Sulphur isotope ratios, expressed as $\delta^{34}\text{S}$ values, were determined for total S and total sulphate (i.e., HI-reducible S) on foliage and soil samples. Isotope ratios for LiCl-extractable inorganic sulphate were determined separately only for the mineral soil, as organic sulphate is absent from pine foliage (total sulphate = inorganic sulphate), and extraction techniques do not clearly separate inorganic from organic sulphate in forest floor materials.

Stable S isotope ratios of total S, inorganic sulfate, and HI-reducible S in foliage and soil material were determined as follows:

- Total S in soils was converted to SO_4^{2-} by digestion in a HNO_3 - Br_2 mixture (Zhabina and Volkov 1978; Krouse and Tabatabai 1986). Undissolved matter was removed by filtration, and SO_4^{2-} was precipitated as BaSO_4 by addition of 50 mL 0.25 M BaCl_2 solution. Total S in pine needles was converted to SO_4^{2-} by the Parr bomb technique (Siegfriedt et al. 1951). The SO_4^{2-} was subsequently precipitated as BaSO_4 as described above.

- Inorganic sulfate was extracted from mineral soil with a 0.1 M LiCl solution in an overhead shaker (10 rpm) using a 10:1 solution/soil ratio and an extraction time of 24 h. Subsequently, the sulfate-containing extraction solution was filtered and evaporated to less than 200 mL, and BaSO₄ was precipitated as described previously.
- HI-reducible S was extracted from foliage and soil samples using the Johnson-Nishita reduction mixture, which comprises HI-HCOOH-H₃PO₂ in a 4:2:1 ratio (Johnson and Nishita 1952). The Johnson-Nishita (or HI) reduction mixture converts both inorganic and organic sulfate to H₂S (Freney 1961), a fraction that is often referred to as total sulphate or HI-reducible S (S_{HI-red}). Foliage and soil samples were reacted in a closed and deoxygenated reaction flask with the reduction mixture at 117°C to evolve H₂S. The H₂S was then swept with a N₂ carrier gas through a washing solution into a chemical trap to form CdS, which was subsequently converted to Ag₂S by titration with AgNO₃ solution.
- Sulphur isotope ratios of the produced BaSO₄ or Ag₂S precipitates were determined by isotope ratio monitoring elemental analyzer mass spectrometry using a Carlo Erba 1500 connected to a VG Prism (Giesemann et al. 1994). Sulphur isotope ratios are reported on the usual δ-scale in parts per thousand deviation relative to the internationally accepted standard CDT (troilite from the Cañon Diablo meteorite).

3.4 Sampling and Measurement Schedule

Pre-treatment litter, FH horizons, and mineral soils at both sites were sampled in July–September 2001. Pre-treatment foliage was sampled immediately before fertilizer application in October 2002. Post-treatment current-year pine foliage, litter, FH horizons, and mineral soils were sampled in fall 2003 and 2004, and tree measurements were repeated in fall 2004. Repetition of the 2004 (year 2) sampling and measurements is planned for 2007 (year 5), with annual foliar sampling in the intervening years. Beyond year 5, further sampling and measurements will depend on the duration of tree growth responses and the degree of attenuation of the isotopic signatures in the fertilized treatments.

3.5 Data Analysis

Treatment effects on lodgepole pine growth and on soil and foliar variables will be examined by one-way analysis of variance (ANOVA), using the general linear model procedure (SAS Institute Inc. 1989). Mensurational data may be subjected to covariance analysis, using appropriate initial measurements as covariates. Three *a priori* questions will be tested using single degree-of-freedom contrasts:

1. Is control different from fertilized treatments?
2. Is N alone different from N+S?
3. Is N+S (sulphate-S) different from N+S (S^o)?

4 PRE-TREATMENT RESULTS

4.1 Tree Measurements

Although harvest and regeneration dates of these sites differ only slightly, the substantially greater dbh and height of crop trees at KC than at HC is consistent with the higher productivity of the moister SBSwk1 variant (British Columbia Ministry of Forests 2003) (Table 12).

TABLE 12 *Pre-treatment lodgepole pine crop tree measurements by treatment and installation: October 2002^a*

Holy Cross

Treatment ^b		dbh (cm)	Height (m)	Ht. live crown (m)
1	Mean	9.39	7.37	1.69
	SD	1.79	0.91	0.40
2	Mean	9.39	7.51	1.49
	SD	1.90	0.92	0.45
3	Mean	9.36	7.44	1.57
	SD	1.64	0.87	0.45
4	Mean	9.36	7.38	1.59
	SD	1.92	0.94	0.35
All	Mean	9.37	7.42	1.58
	SD	1.81	0.91	0.42

Kenneth Creek

Treatment ^b		dbh (cm)	Height (m)	Ht. live crown (m)
1	Mean	14.61	10.88	3.25
	SD	2.05	0.91	0.73
2	Mean	14.49	10.85	2.84
	SD	2.00	1.01	0.76
3	Mean	14.53	10.85	2.99
	SD	1.57	0.92	0.70
4	Mean	14.55	10.96	2.95
	SD	1.76	0.90	0.76
5	Mean	14.57	11.05	3.08
	SD	1.66	0.92	0.82
6	Mean	14.79	10.86	3.11
	SD	2.13	1.05	0.66
All	Mean	14.59	10.91	3.04
	SD	1.87	0.95	0.75

a n = 4 (four plots per treatment * one composite sample per plot).

b 1= control, 2 = N + KCl, 3 = N + K₂SO₄, 4 = N + S^o+ KCl, 5 = N + Agrimax + KCl, 6 = Agrimax only.

4.2 Soil and Forest Floor Properties

Surface soil textures at HC are finer than at KC (loam vs. sandy loam) (Table 13) while the coarse fragment content, estimated visually at 20–30% at HC, contrasts strongly with the largely gravel-free materials at KC.

Pre-treatment chemical analyses of composite litter, FH horizon, and mineral soils revealed obvious differences between the two installations (Table 14). In general, the HC site is more nutrient-rich, with higher concentrations of both total and mineralizable N, total S, and exchangeable base cations (Ca, Mg, K) in most of the horizons or depth intervals. Total elemental concentrations for most macro- and micronutrients are also higher in the HC litter and FH horizon samples. Cation exchange capacity is also higher at the HC site, reflecting the higher clay and C concentrations in the mineral soil, and perhaps a higher degree of humification of the forest floors, as suggested by narrower C/N ratios. Exchangeable Al concentrations are higher in the KC forest floor and mineral soil, consistent with the more acidic conditions. Although total S concentrations are higher at the HC site in the FH and 0–20 cm mineral soil, extractable sulphate is consistently higher at the KC site.

Total S concentrations in the forest floors of the HC and KC soil profiles are similar to those reported for other central interior sites, while the values for most of the mineral soil horizons (20–50 mg · kg⁻¹) are at the low end of the range when compared both regionally and globally with other temperate and boreal forest soils (Tables 1, 15, 16) (Mitchell et al. 1992; Kishchuk 1998). Total S is almost entirely organic, with the ester sulphates equalling or exceeding C-bonded S in most of the mineral soil horizons at KC, but comprising most of the organic S in the A and B horizons at HC. Some mineral soil horizons at KC have a much higher proportion of total S occurring as inorganic sulphate-S than at HC: 30% and 20% of total S, in the Bf and Bfj, respectively. The inorganic S is mainly adsorbed SO₄²⁻, and to a minor extent also precipitated SO₄²⁻. (Note that analytical procedures differed for the two profiles, so that inorganic sulphate-S determined for the HC profile included both adsorbed and soluble sulphate-S, which were extracted separately for the KC profile samples.) The accumulation of organic and inorganic S in the KC Bf horizon is enhanced by the enrichment of amorphous and crystalline Al and Fe oxyhydroxides through podzolization (Arocena and Sanborn 1999). These pedogenic minerals are able to adsorb soluble organic matter and inorganic SO₄²⁻ at pH values < 6.

TABLE 13 Particle-size analyses of mineral soil composites

Site	Depth (cm)		% Sand	% Silt	% Clay
Holy Cross (n = 16)	0–20	Mean	49.48	39.21	11.32
		Std. Dev.	3.20	2.84	1.15
Kenneth Creek (n = 24)	0–20	Mean	70.15	22.83	7.02
		Std. Dev.	3.79	3.45	0.59
	20–40	Mean	81.44	13.77	4.79
		Std. Dev.	6.69	5.80	0.94

TABLE 14 Pre-treatment (2001) chemical properties of litter, FH horizons, and mineral soils at Holy Cross and Kenneth Creek installations: means and standard deviations^a

Holy Cross

Horizon/ Depth	Treatment ^b		Exchangeable cations [cmol(+) · kg ⁻¹]								
			CEC	Al	Ca	Fe	K	Mg	Mn	Na	
Litter	1	Mean	37.79	0.530	24.22	0.005	2.80	8.16	2.053	0.032	
		SD	4.73	0.209	2.63	0.003	0.59	1.44	0.302	0.015	
	2	Mean	41.49	0.433	27.49	0.007	2.77	8.44	2.301	0.048	
		SD	2.06	0.070	0.99	0.004	0.30	0.67	0.438	0.018	
	3	Mean	39.70	0.583	25.23	0.004	2.93	8.60	2.311	0.048	
		SD	3.16	0.220	2.74	0.001	0.23	0.92	0.399	0.032	
	4	Mean	44.87	0.349	28.93	0.003	4.09	9.38	2.072	0.042	
		SD	3.84	0.041	2.89	0.001	1.20	1.17	0.492	0.011	
	All	Mean	40.96	0.473	26.47	0.005	3.15	8.65	2.184	0.042	
		SD	4.19	0.168	2.90	0.003	0.84	1.08	0.391	0.018	
	FH	1	Mean	44.08	0.463	34.52	0.019	1.92	5.37	1.724	0.062
			SD	5.96	0.226	5.01	0.007	0.29	0.92	0.197	0.020
2		Mean	42.45	0.460	33.13	0.020	1.83	5.27	1.669	0.076	
		SD	6.01	0.145	6.13	0.005	0.27	0.34	0.211	0.009	
3		Mean	44.02	0.545	34.27	0.022	1.94	5.60	1.573	0.067	
		SD	2.82	0.237	3.19	0.012	0.10	0.48	0.232	0.034	
4		Mean	48.33	0.313	37.29	0.013	2.36	6.45	1.836	0.066	
		SD	7.33	0.082	6.71	0.002	0.16	0.62	0.278	0.013	
All		Mean	44.72	0.445	34.80	0.018	2.01	5.67	1.700	0.068	
		SD	5.63	0.186	5.11	0.008	0.29	0.74	0.229	0.020	
0–20 cm		1	Mean	8.54	0.735	6.07	0.065	0.31	1.22	0.087	0.041
			SD	0.97	0.236	0.68	0.043	0.06	0.09	0.031	0.008
	2	Mean	8.40	0.685	5.98	0.047	0.30	1.21	0.131	0.044	
		SD	0.87	0.310	1.01	0.032	0.03	0.24	0.064	0.017	
	3	Mean	8.56	0.612	6.12	0.046	0.32	1.34	0.091	0.048	
		SD	0.29	0.208	0.32	0.029	0.06	0.18	0.020	0.008	
	4	Mean	9.63	0.608	7.13	0.035	0.39	1.25	0.177	0.035	
		SD	2.36	0.243	2.33	0.019	0.07	0.25	0.072	0.014	
	All	Mean	8.78	0.660	6.32	0.048	0.33	1.25	0.121	0.042	
		SD	1.32	0.232	1.28	0.031	0.06	0.19	0.060	0.012	

Note: Min N = mineralizable N; Avail P = available P (Bray P1); SO₄-S = phosphate-extractable inorganic sulphate-S; S-Combust = S determined by LECO S analyzer; ICP = inductively coupled argon plasma.

a n = 4 (four plots per treatment * one composite sample per plot).

b 1 = control, 2 = N + KCl, 3 = N + K₂SO₄, 4 = N + S^o + KCl, 5 = N + Agrimax + KCl, 6 = Agrimax only.

TABLE 14 *Continued*

Holy Cross

Horizon/ Depth	Treatment		mg · kg ⁻¹			Total (%)			pH (CaCl ₂)	pH (H ₂ O)
			Min N	Avail P	SO ₄ -S	C	N	S-Combust		
Litter	1	Mean	525.3	76.8	12.1	53.38	1.294	0.0676	4.11	4.35
		SD	181.0	14.9	2.6	0.96	0.165	0.0095	0.13	0.11
	2	Mean	340.1	67.8	13.4	52.94	1.258	0.0642	4.18	4.43
		SD	181.1	10.3	1.9	1.15	0.160	0.0152	0.09	0.08
	3	Mean	521.6	74.9	11.2	53.62	1.395	0.0781	4.12	4.36
		SD	208.6	13.4	1.8	0.53	0.081	0.0008	0.09	0.11
	4	Mean	783.4	110.5	12.6	53.23	1.353	0.0728	4.30	4.51
		SD	36.9	29.5	3.7	0.68	0.091	0.0097	0.08	0.07
	All	Mean	542.6	82.5	12.3	53.29	1.325	0.0706	4.18	4.41
		SD	220.6	23.8	2.5	0.81	0.128	0.0106	0.12	0.11
FH	1	Mean	325.8	70.3	9.2	42.38	1.174	0.1003	4.27	4.60
		SD	81.7	15.4	3.9	2.71	0.144	0.0099	0.19	0.13
	2	Mean	248.2	75.2	13.5	40.46	1.089	0.0934	4.29	4.62
		SD	87.3	19.8	5.8	3.15	0.107	0.0053	0.12	0.15
	3	Mean	316.5	66.3	11.9	40.46	1.134	0.0945	4.33	4.66
		SD	69.5	10.6	2.6	4.96	0.046	0.0070	0.16	0.16
	4	Mean	448.5	81.9	12.1	40.63	1.281	0.1057	4.48	4.76
		SD	74.4	23.4	3.1	3.78	0.123	0.0102	0.13	0.15
	All	Mean	334.7	73.4	11.7	40.98	1.169	0.0984	4.34	4.66
		SD	102.4	17.2	3.9	3.46	0.123	0.0090	0.16	0.14
0–20 cm	1	Mean	13.8	151.8	1.0	2.00	0.103	0.0061	4.66	4.99
		SD	3.1	71.0	0.4	0.57	0.021	0.0025	0.05	0.16
	2	Mean	14.8	149.1	1.3	2.15	0.103	0.0066	4.66	4.99
		SD	0.9	74.3	0.4	0.42	0.014	0.0019	0.09	0.20
	3	Mean	12.5	137.9	1.0	1.90	0.098	0.0054	4.72	5.03
		SD	2.4	74.0	0.3	0.26	0.016	0.0009	0.08	0.15
	4	Mean	20.5	213.3	1.5	2.94	0.129	0.0079	4.75	4.95
		SD	9.0	94.0	0.6	1.03	0.029	0.0020	0.14	0.18
	All	Mean	15.4	163.0	1.2	2.25	0.108	0.0065	4.70	4.99
		SD	5.4	76.8	0.4	0.71	0.022	0.0020	0.09	0.16

TABLE 14 *Continued*

Holy Cross

Horizon/ Depth	Treatment		Total (Microwave digest - ICAP)										
			mg · kg ⁻¹						%				
			Al	B	Cu	Fe	Mn	Zn	Ca	K	Mg	P	S
Litter	1	Mean	988.5	5.5	4.9	676.6	725.1	58.0	0.871	0.136	0.096	0.115	0.079
		SD	203.4	1.1	0.5	236.0	71.8	4.5	0.101	0.020	0.009	0.017	0.008
	2	Mean	1220.2	6.2	5.6	996.0	819.8	61.8	0.926	0.135	0.098	0.120	0.082
		SD	290.4	0.3	0.4	518.2	163.0	6.0	0.061	0.012	0.012	0.003	0.009
	3	Mean	966.4	6.4	5.1	564.0	779.9	58.8	0.900	0.137	0.102	0.116	0.084
		SD	41.8	0.7	0.4	110.3	162.5	4.0	0.068	0.010	0.005	0.012	0.004
	4	Mean	914.5	7.0	5.3	563.7	729.8	58.0	1.006	0.174	0.106	0.124	0.085
		SD	91.5	1.3	0.5	126.0	174.0	4.9	0.035	0.033	0.003	0.013	0.007
	All	Mean	1022.4	6.3	5.2	700.1	763.7	59.1	0.926	0.145	0.100	0.119	0.082
		SD	204.6	1.0	0.5	322.3	138.9	4.7	0.081	0.025	0.008	0.012	0.007
FH	1	Mean	5561.4	4.5	8.4	7042.0	1032.6	63.3	0.869	0.161	0.095	0.145	0.082
		SD	563.8	0.6	0.6	1050.0	116.9	4.9	0.088	0.019	0.012	0.019	0.009
	2	Mean	6237.1	5.4	9.0	7954.8	1104.5	65.5	0.856	0.179	0.090	0.154	0.075
		SD	589.2	0.8	0.5	1470.0	256.6	6.9	0.136	0.010	0.012	0.013	0.009
	3	Mean	6605.6	5.4	9.1	8237.7	1069.6	64.4	0.877	0.175	0.100	0.163	0.077
		SD	1032.5	0.6	0.4	1866.0	166.6	10.3	0.072	0.016	0.013	0.020	0.003
	4	Mean	4839.3	6.1	8.4	6356.2	1073.2	69.1	0.932	0.180	0.111	0.161	0.085
		SD	2639.4	0.5	2.1	4050.8	276.5	4.2	0.053	0.036	0.012	0.039	0.007
	All	Mean	5810.9	5.3	8.7	7397.7	1070.0	65.6	0.884	0.174	0.099	0.156	0.080
		SD	1491.5	0.8	1.1	2285.8	193.5	6.6	0.088	0.022	0.014	0.024	0.008

Kenneth Creek

Horizon/ Depth	Treatment		Exchangeable cations [cmol(+) · kg ⁻¹]							
			CEC	Al	Ca	Fe	K	Mg	Mn	Na
Litter	1	Mean	31.65	2.793	15.22	0.011	2.63	6.81	4.110	0.080
		SD	1.11	0.324	1.23	0.001	0.28	0.18	0.053	0.020
	2	Mean	32.77	2.556	15.99	0.011	2.80	7.46	3.854	0.090
		SD	1.78	0.375	1.59	0.003	0.46	0.53	0.254	0.010
	3	Mean	36.90	2.303	17.88	0.009	3.45	9.07	4.089	0.092
		SD	6.70	1.098	4.69	0.002	1.47	2.45	0.931	0.013
	4	Mean	34.72	2.414	17.26	0.010	3.27	8.01	3.667	0.097
		SD	8.26	0.786	5.19	0.002	1.18	2.41	0.213	0.014
	5	Mean	29.32	2.145	14.27	0.011	2.60	6.46	3.740	0.091
		SD	5.94	0.228	3.52	0.003	0.68	1.76	0.315	0.017
	6	Mean	34.31	2.747	16.88	0.011	2.94	7.78	3.854	0.104
		SD	7.11	0.717	4.95	0.002	0.64	1.78	0.204	0.022
	All	Mean	33.28	2.493	16.25	0.010	2.95	7.60	3.886	0.092
		SD	5.71	0.632	3.65	0.002	0.85	1.77	0.418	0.016

TABLE 14 *Continued*

Kenneth Creek

Horizon/ Depth	Treatment		Exchangeable cations [cmol(+) · kg ⁻¹]							
			CEC	Al	Ca	Fe	K	Mg	Mn	Na
FH	1	Mean	24.61	1.782	16.41	0.067	1.70	2.37	2.198	0.083
		SD	2.05	0.255	1.62	0.029	0.36	0.39	0.312	0.017
	2	Mean	25.06	1.486	17.27	0.082	1.86	2.57	1.722	0.074
		SD	3.71	0.264	3.33	0.026	0.19	0.41	0.382	0.011
	3	Mean	28.15	1.481	19.71	0.071	1.71	3.21	1.887	0.079
		SD	8.04	0.878	7.09	0.054	0.32	1.55	0.214	0.015
	4	Mean	26.07	1.512	17.86	0.073	1.76	3.04	1.756	0.070
		SD	4.46	0.565	3.80	0.039	0.33	0.81	0.360	0.009
	5	Mean	23.24	1.746	15.33	0.094	1.69	2.52	1.763	0.100
		SD	4.02	0.328	3.57	0.040	0.28	0.53	0.334	0.013
	6	Mean	28.46	1.414	19.77	0.054	1.97	3.19	1.988	0.082
		SD	5.44	0.327	4.56	0.029	0.46	0.87	0.468	0.015
	All	Mean	25.93	1.570	17.72	0.073	1.78	2.82	1.885	0.081
		SD	4.80	0.456	4.17	0.035	0.31	0.83	0.356	0.015
0–20 cm	1	Mean	2.50	1.273	0.96	0.014	0.04	0.16	0.035	0.019
		SD	0.44	0.194	0.32	0.003	0.01	0.04	0.012	0.010
	2	Mean	2.93	1.506	1.14	0.026	0.04	0.17	0.034	0.025
		SD	0.13	0.540	0.43	0.006	0.01	0.04	0.014	0.006
	3	Mean	3.23	1.555	1.31	0.013	0.04	0.24	0.045	0.019
		SD	0.44	0.249	0.53	0.003	0.01	0.08	0.013	0.007
	4	Mean	2.89	1.601	0.97	0.017	0.05	0.19	0.037	0.023
		SD	0.34	0.460	0.23	0.008	0.01	0.03	0.015	0.003
	5	Mean	2.84	1.762	0.81	0.022	0.04	0.15	0.035	0.026
		SD	0.40	0.599	0.34	0.008	0.01	0.04	0.017	0.004
	6	Mean	2.91	1.654	0.95	0.023	0.04	0.18	0.035	0.025
		SD	0.33	0.535	0.28	0.012	0.00	0.05	0.014	0.002
	All	Mean	2.88	1.558	1.02	0.019	0.04	0.18	0.037	0.023
		SD	0.39	0.432	0.36	0.008	0.01	0.05	0.013	0.006
20–40 cm	1	Mean	0.54	0.115	0.33	0.002	0.02	0.06	0.008	0.004
		SD	0.15	0.047	0.11	0.003	0.01	0.02	0.004	0.004
	2	Mean	0.51	0.079	0.35	0.000	0.02	0.05	0.007	0.005
		SD	0.09	0.035	0.07	0.000	0.00	0.02	0.004	0.002
	3	Mean	0.85	0.264	0.47	0.008	0.02	0.08	0.009	0.003
		SD	0.38	0.196	0.19	0.008	0.00	0.02	0.005	0.003
	4	Mean	0.65	0.134	0.41	0.002	0.02	0.07	0.007	0.007
		SD	0.22	0.039	0.17	0.002	0.01	0.03	0.004	0.006
	5	Mean	0.40	0.108	0.23	0.003	0.01	0.04	0.007	0.007
		SD	0.11	0.068	0.04	0.003	0.01	0.01	0.004	0.004
	6	Mean	0.63	0.178	0.35	0.005	0.02	0.06	0.006	0.009
		SD	0.26	0.124	0.13	0.003	0.00	0.02	0.003	0.002
	All	Mean	0.60	0.146	0.36	0.003	0.02	0.06	0.007	0.006
		SD	0.24	0.110	0.14	0.004	0.01	0.02	0.004	0.004

TABLE 14 *Continued*

Kenneth Creek (KC)

Horizon/ Depth	Treatment		$\text{mg} \cdot \text{kg}^{-1}$			Total (%)			pH (CaCl_2)	pH (H_2O)
			Min N	Avail P	$\text{SO}_4\text{-S}$	C	N	S-Combust		
Litter	1	Mean	124.4	104.7	15.7	53.25	0.708	0.0580	3.76	3.95
		SD	10.9	13.3	7.6	1.07	0.045	0.0073	0.06	0.02
	2	Mean	106.1	133.4	19.1	53.80	0.745	0.0694	3.81	4.05
		SD	23.7	41.3	6.7	1.07	0.033	0.0059	0.03	0.05
	3	Mean	115.7	165.1	25.7	53.37	0.753	0.0671	3.93	4.14
		SD	32.4	106.3	13.3	1.20	0.062	0.0069	0.21	0.18
	4	Mean	128.4	169.8	24.1	52.94	0.771	0.0676	3.89	4.10
		SD	28.7	92.3	19.9	1.23	0.049	0.0035	0.25	0.23
	5	Mean	137.7	106.0	15.1	54.03	0.729	0.0655	3.72	3.96
		SD	51.2	27.3	3.6	0.77	0.009	0.0018	0.24	0.20
	6	Mean	123.9	149.4	17.5	54.01	0.728	0.0643	3.84	4.04
		SD	20.2	17.8	5.1	1.15	0.039	0.0054	0.20	0.19
	All	Mean	122.7	138.1	19.5	53.56	0.739	0.0653	3.82	4.04
		SD	28.8	60.6	10.5	1.05	0.043	0.0061	0.18	0.16
FH	1	Mean	163.1	52.9	19.9	41.35	0.943	0.0678	3.78	4.04
		SD	50.0	4.5	4.5	4.65	0.066	0.0170	0.16	0.16
	2	Mean	159.6	61.2	19.0	39.03	0.980	0.0700	3.83	4.09
		SD	46.8	6.5	3.1	1.21	0.122	0.0158	0.18	0.14
	3	Mean	174.7	59.9	18.0	39.95	0.934	0.0710	3.98	4.24
		SD	48.6	26.8	4.5	6.53	0.070	0.0188	0.46	0.39
	4	Mean	167.0	60.5	18.1	40.16	0.968	0.0716	3.87	4.16
		SD	39.8	18.4	4.8	5.39	0.092	0.0118	0.26	0.21
	5	Mean	165.8	54.1	15.3	42.50	0.999	0.0734	3.65	3.96
		SD	64.0	7.3	2.9	6.50	0.071	0.0148	0.29	0.24
	6	Mean	182.2	62.7	18.7	42.10	1.034	0.0747	3.92	4.17
		SD	21.7	17.8	5.5	5.41	0.022	0.0162	0.29	0.15
	All	Mean	168.7	58.5	18.2	40.85	0.976	0.0714	3.84	4.11
		SD	42.2	14.4	4.1	4.83	0.078	0.0142	0.28	0.23
0–20 cm	1	Mean	6.6	62.4	7.5	1.08	0.053	0.0048	4.55	5.10
		SD	1.5	18.9	3.2	0.17	0.006	0.0006	0.14	0.19
	2	Mean	8.7	47.8	6.9	1.19	0.062	0.0052	4.54	5.04
		SD	0.7	4.2	1.7	0.14	0.006	0.0008	0.26	0.25
	3	Mean	7.7	67.9	4.2	1.09	0.056	0.0040	4.47	5.01
		SD	2.3	5.5	1.4	0.18	0.011	0.0014	0.11	0.14
	4	Mean	7.9	49.8	6.3	1.23	0.059	0.0051	4.47	4.98
		SD	1.1	7.0	2.2	0.15	0.007	0.0010	0.17	0.22
	5	Mean	8.3	37.3	9.3	1.14	0.057	0.0053	4.41	4.89
		SD	1.4	1.8	1.7	0.10	0.003	0.0009	0.20	0.24
	6	Mean	8.0	43.3	7.5	1.14	0.057	0.0052	4.47	5.00
		SD	1.8	7.9	2.5	0.16	0.006	0.0012	0.17	0.21
	All	Mean	7.9	51.4	6.9	1.14	0.057	0.0049	4.48	5.00
		SD	1.5	13.6	2.5	0.15	0.007	0.0010	0.17	0.20

TABLE 14 *Concluded*

Kenneth Creek														
Horizon/ Depth	Treatment		mg · kg ⁻¹			Total (%)			pH (CaCl ₂)	pH (H ₂ O)				
			Min N	Avail P	SO ₄ -S	C	N	S-Combust						
20–40 cm	1	Mean	3.9	58.1	6.7	0.43	0.036	0.0035	5.22	5.62				
		SD	0.4	6.2	2.3	0.02	0.002	0.0009	0.13	0.21				
	2	Mean	4.2	51.5	7.9	0.46	0.039	0.0030	5.30	5.64				
		SD	0.4	3.3	1.7	0.07	0.003	0.0005	0.13	0.19				
	3	Mean	4.7	55.1	7.2	0.53	0.041	0.0039	5.09	5.53				
		SD	0.5	3.9	3.7	0.03	0.003	0.0007	0.24	0.25				
	4	Mean	4.0	44.7	10.0	0.50	0.041	0.0040	5.18	5.51				
		SD	0.2	6.0	7.0	0.04	0.005	0.0007	0.08	0.13				
	5	Mean	4.0	44.2	8.3	0.44	0.039	0.0033	5.21	5.53				
		SD	0.4	4.4	1.6	0.05	0.002	0.0004	0.18	0.23				
	6	Mean	4.2	44.8	10.1	0.48	0.040	0.0034	5.18	5.58				
		SD	0.4	8.7	3.2	0.06	0.003	0.0003	0.20	0.21				
	All	Mean	4.2	49.7	8.3	0.47	0.039	0.0035	5.19	5.57				
		SD	0.4	7.6	3.6	0.06	0.003	0.0006	0.16	0.19				
	Total (Microwave digest - ICAP)													
	Horizon/ Depth	Treatment		mg · kg ⁻¹						%				
				Al	B	Cu	Fe	Mn	Zn	Ca	K	Mg	P	S
	Litter	1	Mean	1001.6	7.6	2.6	262.9	1189.	57.3	0.726	0.108	0.061	0.086	0.050
SD			83.1	0.8	0.3	115.5	42.	2.5	0.052	0.011	0.001	0.005	0.002	
2		Mean	982.9	7.9	2.5	249.1	1140.	54.7	0.771	0.117	0.062	0.095	0.051	
		SD	85.1	0.8	0.1	96.4	68.	5.7	0.027	0.018	0.007	0.007	0.002	
3		Mean	968.5	9.5	2.5	269.0	1202.	60.5	0.822	0.139	0.070	0.112	0.052	
		SD	153.9	2.1	0.2	75.4	237.	2.6	0.116	0.055	0.015	0.028	0.004	
4		Mean	1031.7	8.0	2.8	300.3	1114.	59.8	0.806	0.142	0.071	0.106	0.056	
		SD	210.7	1.5	0.4	263.8	67.	12.7	0.186	0.052	0.013	0.035	0.005	
5		Mean	867.1	6.9	2.5	207.4	1136.	54.3	0.723	0.111	0.059	0.084	0.051	
		SD	79.5	1.4	0.5	53.3	93.	19.3	0.185	0.031	0.006	0.024	0.002	
6		Mean	1042.9	8.1	2.5	202.9	1134.	51.3	0.771	0.120	0.065	0.097	0.052	
		SD	156.7	1.8	0.3	105.6	39.	6.0	0.147	0.024	0.005	0.021	0.003	
All		Mean	982.4	8.0	2.6	248.6	1152.	56.3	0.770	0.123	0.065	0.096	0.052	
		SD	135.0	1.5	0.3	125.8	105.	9.6	0.124	0.035	0.009	0.022	0.003	
FH		1	Mean	4600.9	4.8	8.1	4995.8	1211.	63.7	0.497	0.120	0.086	0.104	0.065
			SD	881.4	0.5	0.7	1499.0	391.	7.0	0.068	0.009	0.002	0.021	0.005
		2	Mean	4773.8	4.9	8.0	4952.2	887.	59.1	0.500	0.133	0.089	0.107	0.065
			SD	622.0	0.4	0.9	601.5	280.	12.7	0.093	0.020	0.008	0.023	0.006
	3	Mean	4821.9	5.0	8.1	5153.6	1246.	64.8	0.582	0.131	0.089	0.120	0.068	
		SD	989.5	1.3	1.3	1275.1	487.	11.5	0.173	0.020	0.010	0.036	0.008	
	4	Mean	5129.0	5.1	8.1	5211.3	915.	55.6	0.490	0.140	0.086	0.113	0.066	
		SD	915.7	0.9	1.5	1524.2	264.	8.3	0.094	0.021	0.012	0.025	0.002	
	5	Mean	4115.7	4.3	7.1	4412.1	802.	50.7	0.436	0.114	0.081	0.091	0.065	
		SD	1200.1	1.3	1.3	1960.9	173.	12.1	0.107	0.028	0.009	0.035	0.007	
	6	Mean	4687.0	5.5	7.6	4749.3	1097.	60.7	0.540	0.138	0.094	0.110	0.072	
		SD	1262.8	1.3	1.2	1582.6	287.	10.1	0.123	0.034	0.009	0.036	0.005	
	All	Mean	4688.1	4.9	7.8	4912.4	1026.	59.1	0.507	0.129	0.087	0.107	0.067	
		SD	938.7	1.0	1.1	1326.2	338.	10.5	0.111	0.023	0.009	0.028	0.006	

TABLE 15 Sulphur fractions of Gleyed Brunisolic Gray Luvisol, Holy Cross installation

Horizon	Depth (cm)	mg · kg ⁻¹			
		Total S	C-S ^a	Ester SO ₄ -S	Inorganic SO ₄ -S
Ln	8–7	294	264	28	1.7
Lv	7–6	505	440	59	6.5
Fm	6–2	670	520	139	10.9
Faw	5–2	672	551	117	3.8
Hh	2–0	954	681	267	6.5
Bm1	0–22	34	10	23	1.1
Bm2	22–40	25	1	23	0.8
Bm3	40–52	30	0	31	0.8
Aeg	52–68	23	4	18	0.8
Bt1	68–84	29	0	33	0.6
Bt2	84–105	30	2	28	0.5
BCk	105–125	38	12	25	0.6
Cca	125–135+	54	24	29	0.8

a C-S = carbon-bonded S.

TABLE 16 Sulphur fractions of Eluviated Dystric Brunisol, Kenneth Creek installation

Horizon	Depth (cm)	mg · kg ⁻¹						
		Total S	Org. S	C-S ^a	Ester SO ₄ -S	Inorg. S	Adsorbed SO ₄ -S	Soluble SO ₄ -S
LF	3–1	1180	1110	956	154	70	0	70
H	1–0	1076	1028	798	230	48	0	48
Ae	0–5	43	39	25	14	4	0	2
Bf	5–12	76	54	35	19	22	13	1
Bfj	12–27	47	38	17	21	9	5	1
Bm	27–60	39	36	16	20	3	0	1
BC	60–100	44	41	26	15	3	0	1
C	100–125+	44	42	18	24	2	0	1

a C-S = carbon-bonded S.

4.3 Lodgepole Pine Foliar Analysis

The pre-treatment foliar nutrient concentrations and nutrient ratios at HC and KC are summarized in Tables 17 and 18, respectively. Pre-treatment mean foliar N and S levels at HC and KC indicated slight to moderate N deficiency and moderate to severe S deficiency (Ballard and Carter 1986; Brockley 2001b). However, foliar analytical results for N and S depend highly on the methodology used for extraction and determination. Foliar nutrient interpretative criteria for lodgepole pine have largely been developed from wet digestion (e.g., Kjeldahl) and dry combustion (e.g., LECO) methodologies for N and S, respectively (Brockley 2001b). These methods typically yield lower N values and higher S values than the dry combustion (for N) and wet digestion (for S) methods used by the Ministry of Forests laboratory (Brockley 2000, 2001a). A recently developed Web-based foliar nutrient diagnosis system uses functions developed from comparisons of wet and dry analytical methodologies to “normalize” values for diagnostic purposes (Thomson et al. 2001). Normalized N and S levels for HC and KC indicate moderate to severe N deficiencies and slight to moderate S deficiencies. Normalized foliar N/S ratios indicate no actual S deficiency but the possibility of S deficiency induced by N fertilization at both sites (Ballard and Carter 1986; Brockley 2001b). Pre-treatment foliar SO_4 was less than $60 \text{ mg} \cdot \text{kg}^{-1}$ at HC, indicating actual S deficiency or the likelihood of induced deficiency following N fertilization (Brockley 2001b). At KC, foliar SO_4 levels indicate no actual or inducible S deficiency. Pre-treatment foliar SO_4 is likely better than total S or N/S ratio as an indicator of lodgepole pine S status (Brockley 2000).

Pre-treatment foliar B levels at HC and KC are below a threshold value of $12 \text{ mg} \cdot \text{kg}^{-1}$, below which N fertilization may suppress height growth and (or) provoke development of acute B deficiency symptoms (Brockley 2003). These values are at the lower end of those typically reported for lodgepole pine in the British Columbia interior (Brockley 2001a).

At both sites, actual P and K concentrations indicate possible slight P and K deficiencies (Ballard and Carter 1986). However, revised interpretative criteria for lodgepole pine indicate sufficiency (Brockley 2001b). Also, “normalized” N/P and N/K ratios indicate favourable foliar nutrient balance.

Foliar Cu levels at HC and the Cu and Fe levels at KC indicate possible deficiencies (Ballard and Carter 1986; Brockley 2001b). However, Cu and Fe deficiencies in lodgepole pine, and growth responses following Cu and Fe fertilization, are poorly documented. Low Cu and Fe levels are common in lodgepole pine in the British Columbia interior, and do not appear to negatively affect stand performance and (or) growth response following N fertilization (Brockley 1996).

Foliar Ca, Mg, Zn, and Mn levels are adequate, according to available interpretative criteria (Ballard and Carter 1986; Brockley 2001b).

TABLE 17 Nutrient concentrations in current-year foliage by installation and treatment: October 2002^a

Treatment ^b		%						mg · kg ⁻¹						
		N	P	Ca	Mg	K	S ^c	SO ₄ -S	Cu	Zn	Fe	Mn	B	Al
Holy Cross														
1	Mean	1.23	0.13	0.14	0.102	0.51	0.069	46	2.8	44	58	298	8.3	452
	SD	0.02	0.01	0.01	0.003	0.02	0.005	7	0.7	3	11	17	0.5	37
2	Mean	1.24	0.14	0.15	0.097	0.51	0.074	57	2.7	45	56	310	9.5	469
	SD	0.11	0.01	0.02	0.005	0.03	0.002	5	0.1	5	29	28	2.8	59
3	Mean	1.24	0.13	0.14	0.101	0.50	0.071	57	2.3	46	52	285	8.1	397
	SD	0.08	0.01	0.01	0.004	0.02	0.004	21	0.2	3	18	31	2.1	41
4	Mean	1.28	0.14	0.15	0.097	0.51	0.075	63	2.5	44	43	308	8.9	411
	SD	0.11	0.01	0.01	0.004	0.06	0.006	37	0.2	1	16	72	3.5	9
All	Mean	1.25	0.13	0.15	0.099	0.51	0.072	56	2.6	45	52	300	8.7	432
	SD	0.08	0.01	0.01	0.004	0.03	0.005	21	0.4	3	19	39	2.3	47
Kenneth Creek														
1	Mean	1.15	0.12	0.14	0.086	0.41	0.070	90	2.5	40	27	384	7.3	478
	SD	0.02	0.01	0.01	0.005	0.02	0.002	12	0.0	3	1	39	3.0	44
2	Mean	1.11	0.12	0.15	0.092	0.41	0.070	88	2.5	40	28	380	8.4	522
	SD	0.02	0.00	0.01	0.004	0.01	0.003	13	0.1	4	2	59	2.3	34
3	Mean	1.12	0.12	0.15	0.090	0.44	0.069	74	2.3	40	29	384	8.9	486
	SD	0.03	0.01	0.01	0.007	0.05	0.002	11	0.1	3	4	61	1.8	75
4	Mean	1.11	0.12	0.14	0.090	0.41	0.066	87	2.3	38	26	363	7.6	529
	SD	0.02	0.00	0.01	0.002	0.02	0.002	12	0.2	3	1	18	1.1	32
5	Mean	1.12	0.12	0.14	0.090	0.41	0.068	94	2.4	38	28	333	5.6	476
	SD	0.06	0.01	0.01	0.004	0.04	0.005	13	0.2	2	2	28	1.3	46
6	Mean	1.14	0.12	0.16	0.092	0.40	0.070	94	2.4	39	28	388	7.4	537
	SD	0.09	0.01	0.02	0.001	0.01	0.003	7	0.1	1	1	16	1.6	83
All	Mean	1.12	0.12	0.15	0.090	0.41	0.069	88	2.4	39	28	372	7.5	505
	SD	0.04	0.01	0.01	0.004	0.03	0.003	12	0.1	3	2	41	2.0	56

a n = 4 (four plots per treatment * one composite sample per plot).

b 1 = control, 2 = N + KCl, 3 = N + K₂SO₄, 4 = N + So + KCl, 5 = N + Agrimax + KCl, 6 = Agrimax only.

c Total S values are for the wet digestion-ICAP method.

TABLE 18 *Nutrient concentration ratios in current-year foliage by installation and treatment: October 2002^a*

Treatment ^b		N/P	N/K	N/Ca	N/Mg	N/S
Holy Cross						
1	Mean	9.2	2.4	8.7	12.1	17.9
	SD	0.5	0.1	0.6	0.4	1.4
2	Mean	9.1	2.4	8.2	12.8	16.8
	SD	0.7	0.2	0.6	1.3	1.4
3	Mean	9.3	2.5	8.8	12.3	17.6
	SD	0.3	0.2	1.3	1.1	1.7
4	Mean	9.4	2.5	8.6	13.2	17.1
	SD	0.6	0.2	0.9	1.3	1.4
All	Mean	9.2	2.5	8.6	12.6	17.3
	SD	0.5	0.2	0.9	1.1	1.4
Kenneth Creek						
1	Mean	9.5	2.8	8.1	13.4	16.4
	SD	0.5	0.1	0.4	0.5	0.3
2	Mean	9.4	2.7	7.4	12.1	15.9
	SD	0.3	0.1	0.6	0.4	0.8
3	Mean	9.2	2.6	7.7	12.5	16.2
	SD	0.3	0.3	0.7	0.9	0.6
4	Mean	9.6	2.7	7.7	12.4	16.8
	SD	0.3	0.1	0.4	0.3	0.6
5	Mean	9.4	2.8	7.9	12.5	16.5
	SD	0.4	0.1	0.7	0.4	0.6
6	Mean	9.6	2.9	7.1	12.3	16.3
	SD	0.5	0.3	0.8	0.9	0.8
All	Mean	9.5	2.7	7.7	12.5	16.4
	SD	0.4	0.2	0.7	0.7	0.6

a n = 4 (four plots per treatment * one composite sample per plot).

b 1 = control, 2 = N + KCl, 3 = N + K₂SO₄, 4 = N + So + KCl, 5 = N + Agrimax + KCl, 6 = Agrimax only.

**4.4 Sulphur
Stable Isotope
Analyses: Soils and
Foliage**

At HC, the isotope ratios of total S in foliage before fertilization were on average +5.2‰ and those of soil samples ranged between +4.9 and +4.4‰ (Table 19). At KC, the isotope ratios of total S averaged +8.2‰ in the foliage, and ranged between +8.0 and +6.6‰ in the various soil horizons. The standard deviations of average $\delta^{34}\text{S}$ values for total S determined in foliage and soils from the 16 different plots in HC and the 24 different plots in KC were typically ± 0.5 or less. This is only marginally higher than the analytical uncertainty and indicates that the various plots at both sites are isotopically similar. All $\delta^{34}\text{S}$ values for total S ranged between +4.4 and +8.2‰, significantly lower than those of the selected fertilizers, enabling tracing of the fate of the applied S with isotope techniques.

TABLE 19 *Stable Sulphur isotope data summary (2001 pre-treatment samples): means and standard deviations for $\delta^{34}\text{S}$ (‰) in total Sulphur, total sulphate-S (i.e., HI-reducible S), and inorganic sulphate-S*

Site	Sample type	$\delta^{34}\text{S}$ (‰)					
		Total S		Total $\text{SO}_4\text{-S}$		Inorganic $\text{SO}_4\text{-S}$	
		Mean	SD	Mean	SD	Mean	SD
Holy Cross (n = 16)	Foliage	5.2	0.7	8.7	1.0	n.d.	
	Litter	4.5	0.4	5.4	1.2	n.d.	
	FH	4.9	0.5	3.4	1.0	n.d.	
	Mineral soil (0–20 cm)	4.4	0.7	3.3	0.4	2.9	0.7
Kenneth Creek (n = 24)	Foliage	8.2	0.3	11.6	0.8	n.d.	
	Litter	7.2	0.4	7.2	1.7	n.d.	
	FH	8.0	0.3	5.4	1.7	n.d.	
	Mineral soil (0–20 cm)	7.3	0.7	4.1	1.7	3.9	0.7
	Mineral soil (20–40 cm)	6.6	0.5	3.9	1.5	3.9	1.1

Note: n.d. = not determined

For samples collected in 2001, S isotope ratios of inorganic sulphate were obtained only for mineral soil horizons, and ranged between 2.9 and 3.9‰. The S isotope ratios of total sulphate were similar to those of inorganic sulphate in the mineral soils. $\delta^{34}\text{S}$ values of total sulphate were highest in the pine foliage (8.7 and 11.6‰ for HC and KC, respectively) and progressively decreased in the litter (5.4 and 7.2‰, respectively) and forest floor (3.4 and 5.4‰, respectively). These patterns are consistent with known small isotope effects during S transformation processes in forest stands and soils.

The standard deviation of average $\delta^{34}\text{S}$ values for inorganic sulphate and total sulphate obtained from the 16 plots at HC and 24 plots at KC was generally less than 1.7‰. The strong reproducibility of $\delta^{34}\text{S}$ values for total and inorganic sulphate for samples from the respective plots at HC and KC provides an excellent basis for tracing the fate of the applied fertilizers with their distinct S isotope ratios.

5 SUMMARY AND CONCLUSIONS

1. The KC site, in the SBSwk₁, has a higher site index for lodgepole pine than the HC site, in the SBSdk. This finding is consistent with the moister climate of the KC site. Although the sandier soils at KC would have a lower water-holding capacity than the medium-textured soils at HC, the approximately two-fold higher annual precipitation at the KC site apparently offsets this potential site limitation. In contrast, concentrations of total and (or) available soil macronutrients (N, S, P, Ca, Mg, K) are usually higher at the HC site.
2. The Luvisolic and Brunisolic/Podzolic soils at the HC and KC sites, respectively, exhibit total S concentrations in organic and mineral horizons that are typical of forest soils in the British Columbia central interior, with the mineral soil S concentrations among the lowest found in the temperate and boreal zones. Organic S forms predominate in both organic and mineral horizons.
3. Current-year foliar nutrient concentrations and ratios indicate a more adequate S status at KC. Boron concentrations are low enough at both sites that deficiencies may be induced by N fertilization. With the exception of N, all other macro- and micronutrients appear adequate at both sites, according to current interpretive criteria.
4. Background stable isotope ratios for total S in current-year pine foliage and soils, expressed as $\delta^{34}\text{S}$, ranged between +4 and +9‰. These values differ sufficiently from the fertilizer S (+17.5 to 22.6‰) to make a tracer experiment feasible, at realistic rates of S addition (100 kg S · ha⁻¹).

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