

# Soil and lodgepole pine foliar responses to two fertilizer sulphur forms in the Sub-Boreal Spruce zone, central interior British Columbia

P.T. Sanborn, J. Prietzel, and R.P. Brockley

**Abstract:** Sulphur (S) fractions, total nitrogen (N) concentrations, and cumulative net S mineralization of forest floors and surface mineral soils (0–20 cm) were measured 13 years after an S-deficient lodgepole pine (*Pinus contorta* var. *latifolia* Engelm.) stand was fertilized ( $100 \text{ kg S}\cdot\text{ha}^{-1} + 400 \text{ kg N}\cdot\text{ha}^{-1}$ ) in 1990. Fertilizer treatments compared two S sources: elemental S ( $\text{S}^0$ ) and ammonium sulphate (AS). Total S and ester sulphate-S ( $\text{SO}_4\text{-S}$ ) concentrations and cumulative net S mineralization in forest floors were significantly higher in the  $\text{S}^0$  treatment than in the control and AS treatment. Except for a slight elevation of extractable  $\text{SO}_4\text{-S}$ , such differences were absent in the mineral soils. Total S and  $\text{SO}_4\text{-S}$  concentrations in current-year (2002) pine foliage from the  $\text{S}^0$  treatment were significantly higher than in foliage from the control and AS treatment, while foliar N concentrations indicated that all treatments had caused a return to N-deficiency. These results indicate that prolonged amelioration of S deficiencies in lodgepole pine stands in central interior British Columbia may be possible with single applications of  $\text{S}^0$ .

**Résumé :** Les différentes formes de soufre (S) et les concentrations d'azote (N) total, ainsi que la minéralisation nette cumulative de S, ont été mesurées dans la couverture morte et la surface du sol minéral (0–20 cm) échantillonnés 13 ans après la fertilisation ( $100 \text{ kg S}\cdot\text{ha}^{-1} + 400 \text{ kg N}\cdot\text{ha}^{-1}$ ), en 1990, d'un peuplement de pin tordu (*Pinus contorta* var. *latifolia* Engelm.) déficient en S. Les traitements de fertilisation comprenaient deux sources de S : le S élémentaire ( $\text{S}^0$ ) et le  $(\text{NH}_4)_2\text{SO}_4$  (SA). Les concentrations de S total et de S sous forme d'ester de sulfate ainsi que la minéralisation nette cumulative de S dans la couverture morte étaient significativement plus élevées dans le traitement  $\text{S}^0$  que dans les traitements témoins ou SA. À l'exception d'une faible augmentation de S sous forme de sulfate extractible, de telles différences étaient absentes dans le sol minéral. Le feuillage de pin de l'année courante (2002) dans le traitement  $\text{S}^0$  avait des concentrations de S total et de S sous forme de sulfate significativement plus élevées que dans le feuillage des traitements témoins et SA tandis que les concentrations de N foliaire indiquaient que tous les traitements étaient à nouveau déficients en N. Ces résultats indiquent qu'il est possible de produire une amélioration prolongée des déficiences en S avec une seule application de  $\text{S}^0$  dans les peuplements de pin lodgepole du centre intérieur de la Colombie-Britannique.

[Traduit par la Rédaction]

## Introduction

Forest fertilization research in the British Columbia (B.C.) interior has confirmed the existence of widespread sulphur (S) deficiencies in lodgepole pine (*Pinus contorta* var. *latifolia* Engelm.) stands, with mineral-soil S concentrations among the lowest reported for temperate- and boreal-zone forests (Brockley and Sheran 1994; Brockley 1996, 2000; Kishchuk and Brockley 2002; Brockley 2004). In operational fertilization

of lodgepole pine in the B.C. interior, a urea – ammonium sulphate blend (10% S) is commonly used, but experimental fertilization treatments have included both sulphate-S ( $\text{SO}_4\text{-S}$ ) and elemental S ( $\text{S}^0$ ) sources.  $\text{S}^0$  must undergo microbial oxidation before becoming plant-available, so soluble  $\text{SO}_4\text{-S}$  sources provide more rapid elevation of foliar S concentrations after 1 year (Brockley and Sheran 1994). In the longer term, the two S sources were equally effective in improving radial growth over 6 years (Brockley 2004).

Research on S-fertilizer behaviour in forest soils of the temperate and boreal zones is much less developed than on its behaviour in agricultural soils, principally because S deficiencies have not been widely recognized or treated operationally. The interaction of S-containing fertilizers with a Vancouver Island forest floor was examined during a 14-day aerobic incubation; there was little retention of sulphate from the more available fertilizer forms (ammonium sulphate, thiourea, ammonium sulphate + urea) (Beaton et al. 1969). In contrast, much lower rates of sulphate leaching occurred with  $\text{S}^0$  amendments, but other possible fates of this added S were not examined. In research conducted on forest soils near natural gas processing plants in Alberta, soil properties

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have been examined in relation to S<sup>0</sup> 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 S<sup>0</sup> oxidation in forest soils have examined the roles of environmental factors, especially moisture regime, and previous S-deposition history (Lett et al. 1981a, 1981a; Nevell and Wainwright 1987). In watershed-scale experiments in the Black Forest in Germany, SO<sub>4</sub>-S applications at rates of 170 kg S·ha<sup>-1</sup> have been used, but these treatments were in addition to existing soil S reserves exceeding 1000 kg·ha<sup>-1</sup> 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). The transformations and fates of S from such treatments would not necessarily be the same if it was applied to forests with demonstrated S deficiencies, little or no anthropogenic S deposition, and natural S reserves of 500 kg·ha<sup>-1</sup> or less — conditions that occur across much of central interior B.C.

This paper examines the long-term S status of an initially S-deficient lodgepole pine stand in the central interior of B.C. where both SO<sub>4</sub>-S and S<sup>0</sup> were applied in 1990. Foliar S concentrations, soil S fractions, and S mineralization behaviour are compared in relation to fertilizer S source.

## Methods

### Location and site description

The study site (54°47'N, 123°42'W) is located at 815 m elevation 53 km southwest of Prince George, B.C., within the Stuart Dry Warm variant of the Sub-Boreal Spruce biogeoclimatic zone (SBSdw3). Site and vegetation characteristics most closely match the Pine – Black Spruce – Feathermoss (05) site series for this variant (DeLong et al. 1993). The predominant Brunisolic Gray Luvisolic soils (Soil Classification Working Group 1998) have formed on an undulating to rolling morainal deposit with loam to sandy loam textures. The site was harvested in 1973, scarified to assist natural regeneration of lodgepole pine, and thinned to a density of approximately 1500 stems/ha in 1988.

A fertilization trial (Ministry of Forests Experimental Project 886.10) with single-tree-centred circular plots (5 m radius, 0.008 ha, minimum 5 m wide untreated buffer area between plots) was established in 1990, and 11 fertilization treatments were assigned in a completely randomized design, with 15 replicates of each. For this study we sampled all plots of the unfertilized control and the two fertilization treatments with application rates of 400 kg nitrogen (N)·ha<sup>-1</sup> + 100 kg S·ha<sup>-1</sup>. In one of these treatments, S was added as ammonium sulphate (AS) (21:0:0:24 N-P-K-S). The other treatment used 100 mesh (<150 µm) S<sup>0</sup> (0:0:0:100 N-P-K-S) as the S source. Urea (46:0:0 N-P-K) was the primary N source in both treatments. All fertilizers were applied manually. Additional details of the larger experiment in which these treatments were included are given by Brockley (2000).

### Sampling and chemical analysis

For characterization of S fractions in horizons of the unfertilized soil at this site, a representative pedon was sampled in the untreated buffer area of the installation. Total carbon (C) and N concentrations were determined with a LECO CHN-600 analyzer. Total S concentration was deter-

mined with a LECO SC-444 analyzer and hydriodic acid (HI)-reducible S with a Johnson/Nishita apparatus (Freney 1961). Water-soluble SO<sub>4</sub>-S in organic horizons was extracted with deionized water (1:10 soil:H<sub>2</sub>O) and determined in the filtered (0.45 µm) extracts by ion chromatography (DIONEX 2020i). C-bonded S was estimated as total S less HI-reducible S. Ester SO<sub>4</sub>-S was calculated as HI-reducible S less inorganic S, which was considered equal to water-soluble SO<sub>4</sub>-S. For the mineral soil, inorganic S was assumed to be equal to NH<sub>4</sub>F-extractable SO<sub>4</sub>-S (Prietzel and Hirsch 2000). The methods used for speciation of inorganic S in the mineral soil are described in detail in Prietzel et al. (2001).

Current-year foliage was collected from the upper 1/3 of the crown of the central tree of each plot ( $n = 15$ ) in October 2002 and oven-dried (70 °C, 48 h) prior to analysis. Forest floor and surface mineral soil (0–20 cm depth) were sampled at a single point on a random bearing 2 m from each plot centre in October 2003 and stored in a field-moist condition at 4 °C. Forest-floor material was passed through a 16 mm mesh sieve and random equally weighted composites of three samples were prepared, giving five composite samples for each treatment. The mineral soils were passed through a 2 mm mesh sieve and composited similarly.

Net S mineralization was measured with an aerobic open system incubation method (Stanford and Smith (1972, as modified by Fyles and McGill 1987). The composited mineral soils and forest-floor material was incubated in duplicate for 27 weeks at 30 °C. Twenty grams (oven-dry basis) of the moist composited samples was weighed onto GF/C glass-fibre filter pads resting in the tops of 7 cm diameter plastic Buchner funnels placed on petri dishes in an incubator. At 0, 2, 4, 6, 9, 12, 15, 19, 23, and 27 weeks the samples were leached under 60 kPa suction with three 25-mL increments of 10 mmol/L KCl, followed by 25 mL of an S-free nutrient solution (2 mmol/L CaCl<sub>2</sub>, 2 mmol/L MgCl<sub>2</sub>·H<sub>2</sub>O, 5 mmol/L Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub>·H<sub>2</sub>O, 5 mmol/L KCl; Maynard et al. 1983). Except for the initial leachates, which were discarded, the leachates were made up to 100 mL with distilled water and shaken, and a portion was kept frozen until analysis of SO<sub>4</sub><sup>2-</sup> by ion chromatography after filtration (0.45 µm). Incubated samples were kept covered, and twice weekly were aerated and the moisture content was adjusted.

The remaining amounts of the composite samples were air-dried, with grinding of the forest-floor material (<2 mm), and analyzed for total C and N (LECO CHN-600 analyzer), pH (Orion 550A pH meter with 8102 BN combination electrode, using a ratio of 1:2 and 1:4 mineral soil and forest-floor material to 0.01 mol·L<sup>-1</sup> CaCl<sub>2</sub>, respectively), and S fractions: total S (LECO C analyzer), total SO<sub>4</sub>-S (HI-reducible) by the Johnson/Nishita method (Freney 1961), and extractable inorganic SO<sub>4</sub>-S (forest-floor material was extracted with 0.01 mol·L<sup>-1</sup> NH<sub>4</sub>Cl; mineral soil was extracted with 500 mg phosphorus (P)·L<sup>-1</sup> as Ca(H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub>·H<sub>2</sub>O; SO<sub>4</sub><sup>2-</sup> determined by ion chromatography). Additional S fractions were calculated as for the horizons sampled in the unfertilized pedon.

Foliar total N and S concentrations were determined with an Elementar Vario CNS analyzer. Foliar inorganic SO<sub>4</sub>-S was extracted with boiling 0.01 mol·L<sup>-1</sup> HCl, and SO<sub>4</sub><sup>2-</sup> in the extracts was determined by ion chromatography.

One-way analysis of variance (ANOVA) with SYSTAT version 10 (SPSS Inc. 2000) was used to compare soil and

**Table 1.** Selected soil properties and S-fraction concentrations from a representative Luvisolic soil at Cluculz Creek, B.C.

Soil horizon	Depth (cm)	Total C (g·kg <sup>-1</sup> )	Total N (g·kg <sup>-1</sup> )	Total S (mg·kg <sup>-1</sup> )	C-bonded S (mg·kg <sup>-1</sup> )	Ester SO <sub>4</sub> -S (mg·kg <sup>-1</sup> )	Adsorbed SO <sub>4</sub> -S (mg·kg <sup>-1</sup> )	Water-soluble SO <sub>4</sub> -S (mg·kg <sup>-1</sup> )
F	2–1	458	11.5	1030	864	160	0	6
H	1–0	377	8.6	822	662	149	0	11
Ahe	0–10	64	0.4	43	17	22	0	2
Bm1	10–21	30	0.3	38	17	19	0	1
Bm2	21–33	20	0.2	30	5	23	0	1
Bgj	33–53	14	0.1	26	9	14	1	2
Btg	53–76	18	0.1	25	10	14	0	1
BC	76–100+	10	0.1	25	0	25	0	1

**Table 2.** ANOVA summary table of nutrient concentrations in current-year lodgepole pine foliage showing variance ratios (*F*), *p* values, and mean square errors.

Source of variation	df	<i>F</i>	<i>p</i> > <i>F</i>
Total N			
Treatment	2	0.736	0.485
Mean square error	41		1.073
Total S			
Treatment	2	5.054	0.011
Mean square error	41		0.010
Inorganic SO <sub>4</sub> -S			
Treatment	2	13.192	0.000
Mean square error	41		790.941

foliar properties and cumulative S mineralization between treatments. A transformation (square root of the inverse) was used to improve the normality of the residuals for total N, total S, C-bonded S, ester SO<sub>4</sub>-S, and inorganic SO<sub>4</sub>-S concentrations, cumulative net S mineralization (forest floor), and total C concentration (mineral soils).

## Results

Total S concentrations were highest in the forest floors and decreased with depth in the mineral-soil horizons (43–25 mg·kg<sup>-1</sup>), though not to the same degree as total C and N concentrations (Table 1). On average, organic S accounted for 95% or more of total S, with C-bonded S predominating in organic horizons and ester sulphates in mineral-soil horizons. Adsorbed and water-soluble sulphate concentrations were at or below 2 mg·kg<sup>-1</sup> in the mineral-soil horizons. C:S ratios were above 500 in the upper 50 cm of the soil profile.

Current-year foliar N concentrations showed no treatment effect, unlike total S and inorganic SO<sub>4</sub>-S concentrations, which were both highest in the S<sup>0</sup> treatment (Tables 2 and 3). In the forest floors sampled in 2003, effects of the selected 1990 treatments were significant for total S, C-bonded S, ester SO<sub>4</sub>-S, and inorganic SO<sub>4</sub>-S, with the highest concentrations in the S<sup>0</sup> treatment and no significant difference between the AS treatment and the control (Tables 3 and 4). Total N concentrations showed no treatment effects in either the forest floor or the mineral soil. Although both forest-floor and mineral-soil pH values were lowest in the S<sup>0</sup> treatment, these differences were not significant (Tables 3 and 4). In the mineral soils (0–20 cm depth), significant treatment effects occurred only for inorganic SO<sub>4</sub>-S, which was high-

est in the S<sup>0</sup> treatment, though with very low absolute values that averaged only 2.1 mg·kg<sup>-1</sup>. Net aerobic S mineralization paralleled the patterns for S fractions, with cumulative mineralization at 27 weeks showing a significant treatment effect only in the forest floor, with highest rates occurring in the S<sup>0</sup> treatment and no significant difference between the AS treatment and the control (Tables 3 and 4, Fig. 1).

## Discussion

Earlier analyses of horizons from three pedons in the unfertilized portion of this site (Kishchuk 1998) also showed low absolute concentrations of total S and S fractions in the mineral soils, consistent with the broad regional pattern in the B.C. interior. Total S concentrations in the A and B horizons of our pedon (25–43 mg·kg<sup>-1</sup>) were lower than those reported by Kishchuk (1998) (60–110 mg·kg<sup>-1</sup>), but the absolute values are sufficiently low that such between-study differences may reflect the inherent limitations of S-analyzers. The extremely low concentrations of adsorbed inorganic SO<sub>4</sub>-S may reflect both absolute S scarcity and the low concentrations of extractable sesquioxides in this moderately developed Luvisolic soil (P.T. Sanborn, unpublished data).

Foliar N concentrations in all three treatments indicated moderate to severe N deficiencies, based on thresholds (10.0–11.5 g·kg<sup>-1</sup> N) in current interpretive guidelines (Brockley 2001). These criteria suggested that total S was slightly to moderately deficient in the control and AS treatment (0.8–1.0 g·kg<sup>-1</sup> S), but adequate (>1.0 g·kg<sup>-1</sup> S) in the S<sup>0</sup> treatment, which differed significantly from the control. Foliar SO<sub>4</sub>-S concentrations in the control were within the 40–60 mg·kg<sup>-1</sup> range suggested to indicate moderate to severe S deficiency, a condition that would likely provide superior growth responses to fertilization with N + S than to S alone (Brockley 2000, 2001). Although these criteria were not designed for assessing fertilized stands, the elevated SO<sub>4</sub>-S concentrations in the 2002 foliage may indicate that a single S<sup>0</sup> treatment in 1990 had ameliorated S availability sufficiently that even after 12 years, this nutrient was no longer limiting. In contrast, the absence of any significant differences in foliar total S and SO<sub>4</sub>-S concentrations between the AS treatment and the control suggests that the use of a more immediately available S source has not provided a lasting improvement in the S status of this site.

These patterns in foliar total S and SO<sub>4</sub>-S concentrations are consistent with the strikingly higher concentrations of total S, ester SO<sub>4</sub>-S, and inorganic SO<sub>4</sub>-S, as well as S mineralization rates, in the forest floor under the S<sup>0</sup> treatment.

**Table 3.** Selected chemical properties and S-fraction concentrations in current-year lodgepole pine foliage, forest floor, and mineral soil (0–20 cm).

		Control		S <sup>0</sup> treatment		AS treatment	
		LS mean	SE	LS mean	SE	LS mean	SE
Pine foliage ( <i>n</i> = 15 for control and AS treatments; <i>n</i> = 14 for S <sup>0</sup> treatment)	Total N (g·kg <sup>-1</sup> )	10.67	0.267	10.84	0.277	11.13	0.267
	Total S (g·kg <sup>-1</sup> )	0.91a	0.025	1.02b	0.026	1.00ab	0.025
	Inorganic SO <sub>4</sub> -S (mg·kg <sup>-1</sup> )	46.07a	7.26	97.34b	7.26	57.18a	7.26
Forest floor ( <i>n</i> = 5)	Total C (g·kg <sup>-1</sup> )	288.7	24.17	322.1	24.17	255.2	24.17
	Total N (g·kg <sup>-1</sup> )	7.97	0.62	9.47	0.80	7.68	0.59
	pH (CaCl <sub>2</sub> )	4.51	0.10	4.30	0.10	4.51	0.10
Mineral soil ( <i>n</i> = 5)	Total S (mg·kg <sup>-1</sup> )	508a	35.8	1004b	99.3	527a	37.8
	C-bonded S (mg·kg <sup>-1</sup> )	303a	28.5	592b	87.0	330a	32.3
	Ester SO <sub>4</sub> -S (mg·kg <sup>-1</sup> )	192a	20.4	412b	64.2	179a	18.3
Mineral soil ( <i>n</i> = 5)	Inorganic SO <sub>4</sub> -S (mg·kg <sup>-1</sup> )	8.2a	1.3	44.8b	16.4	11.7a	2.2
	S mineralization (mg·kg <sup>-1</sup> )*	17.9a	4.7	76.3b	41.9	20.3a	5.7
	Total C (g·kg <sup>-1</sup> )	10.6	1.48	11.0	1.57	11.2	1.62
Mineral soil ( <i>n</i> = 5)	Total N (g·kg <sup>-1</sup> )	0.53	0.060	0.54	0.060	0.56	0.060
	pH (CaCl <sub>2</sub> )	4.80	0.06	4.66	0.06	4.82	0.06
	Total S (mg·kg <sup>-1</sup> )	43	5.4	43	5.4	40	5.4
Mineral soil ( <i>n</i> = 5)	C-bonded S (mg·kg <sup>-1</sup> )	15	4.3	14	4.3	11	4.3
	Ester SO <sub>4</sub> -S (mg·kg <sup>-1</sup> )	28	3.7	27	3.7	28	3.7
	Inorganic SO <sub>4</sub> -S (mg·kg <sup>-1</sup> )	0.7a	0.15	2.1b	0.15	1.1a	0.15
Mineral soil ( <i>n</i> = 5)	S mineralization (mg·kg <sup>-1</sup> )*	3.9	0.39	4.9	0.39	4.7	0.39

**Note:** For ANOVAs performed on transformed data, the least squares (LS) means and standard errors (SE) were back-transformed to be expressed in meaningful concentration units, therefore soil S fractions may not sum to total S. Values in a row followed by a different letter are significantly different (Fisher's protected LSD, *p* < 0.05).

\*Cumulative 27-week net S mineralization under three fertilization treatments: control, S<sup>0</sup> (400 kg N·ha<sup>-1</sup> (urea) + 100 kg S·ha<sup>-1</sup> (elemental S)), and AS (400 kg N·ha<sup>-1</sup> + 100 kg S·ha<sup>-1</sup> (urea + ammonium sulphate)).

Although we did not attempt to measure residual unoxidized S<sup>0</sup> directly, given the time elapsed since fertilization, the fine particle size (100 mesh) of the applied elemental S and the reported rates of S oxidation in both field and incubation experiments (Janzen and Bettany 1986; Gal and Nyborg 1987; Gower et al. 1991; Donald and Chapman 1998; Watkinson and Bolan 1998), it is likely that most of the applied S<sup>0</sup> has oxidized. The slightly lower pH values in the S<sup>0</sup> treatment may indicate acidification resulting from this oxidation. Net S mineralization rates and ester SO<sub>4</sub>-S concentrations in the forest floor were highest in the S<sup>0</sup> treatment, and across all treatments these variables had a strong positive correlation (*r* = 0.985, Bonferroni-adjusted probability: *p* < 0.001), suggesting that the ester SO<sub>4</sub>-S fraction may be an important pool of relatively labile organic S, consistent with the model proposed by McGill and Cole (1981).

The apparent lack of any persisting elevation of forest-floor or mineral-soil total S and S-fraction concentrations under the AS treatment is consistent with observations made in other ecosystems. Beaton et al. (1969) observed little retention of fertilizer SO<sub>4</sub>-S in aerobically incubated forest-floor material from Vancouver Island. In agricultural fertilization experiments, long-term repeated applications of inorganic SO<sub>4</sub>-S have led to very limited net accumulation of soil S in the absence of accompanying additions of organic matter (Eriksen and Mortensen 1999; Knights et al. 2000, 2001).

A bioassay experiment conducted by Kishchuk (1998) reported S-fraction concentrations in both forest floor and surface mineral soil sampled 1 year after fertilization in response to selected treatments at this installation that included urea + AS and urea + S<sup>0</sup> at rates of 200 kg N·ha<sup>-1</sup> and

50 kg·S ha<sup>-1</sup>. Addition of SO<sub>4</sub>-S had little effect on soil S status, but total S and ester SO<sub>4</sub>-S concentrations in both forest floors and the mineral soils were significantly elevated with the addition of S<sup>0</sup>. Although we examined treatments with different fertilizer-application rates, and used a different mineral soil sampling depth (20 vs. 10 cm), the parallels in behaviour patterns of fertilizer S across more than a decade are striking.

Except for the treatment effect on soil inorganic SO<sub>4</sub>-S concentrations that was statistically significant but of very small absolute magnitude, these fertilization treatments appear to have had no lasting impact on the upper mineral soil. Our data do not allow us to calculate a complete mass balance for fertilizer fate, so there is more than one possible explanation, including leaching of soluble S below the sampling depth of 20 cm and retention of fertilizer S in older pine-needle cohorts and other above- and below-ground biomass components that were not sampled.

Such remaining uncertainties, along with the need to understand the nature of retained S in forest floor treated with elemental S, point out the limitations of relying exclusively on conventional methods of soil S fractionation. Recent advances in analytical methods (Prietzl et al. 2003) may enable progress in identifying the speciation of organic S in these soils, and new applications of stable-isotope methods to forest fertilization research should provide a much deeper understanding of the fate and behaviour of fertilizer S in lodgepole pine ecosystems in this region (Sanborn et al. 2005).

Shorter term (6-year) tree growth responses to these treatments showed less obvious differences related to S source (Table 5). Although height growth was not significantly im-

**Table 4.** ANOVA summary table of chemical properties of forest floors and mineral soils (0–20 cm) under three fertilization treatments, showing variance ratios ( $F$ ),  $p$  values, and mean square errors.

Source of variation	Forest floors			Mineral soils		
	df	$F$	$p > F$	df	$F$	$p > F$
Total C						
Treatment	2	1.912	0.190	2	0.048	0.953
Mean square error	12	2920.874		12	$2.31 \times 10^{-3}$	
Total N						
Treatment	2	2.019	0.175	0.108	0.898	
Mean square error	12	$9.49 \times 10^{-4}$		0.018		
pH (CaCl <sub>2</sub> )						
Treatment	2	1.323	0.302	2	1.839	0.201
Mean square error	12	0.048		12	0.021	
Total S						
Treatment	2	21.146	0.000	2	0.103	0.903
Mean square error	12	$1.21 \times 10^{-5}$		12	148.167	
C-bonded S						
Treatment	2	9.236	0.004	2	0.260	0.775
Mean square error	11*	$3.64 \times 10^{-5}$		12	90.100	
Ester SO <sub>4</sub> -S						
Treatment	2	13.471	0.001	2	0.027	0.973
Mean square error	12	$7.35 \times 10^{-5}$		12	68.933	
Inorganic SO <sub>4</sub> -S						
Treatment	2	14.290	0.001	2	22.847	0.000
Mean square error	12	$3.72 \times 10^{-2}$		12	0.109	
Cumulative S mineralization						
Treatment	2	4.504	0.035	2	1.648	0.233
Mean square error	12	$4.94 \times 10^{-3}$		12	0.759	

Note: C-bonded S = total S – HI-reducible S; ester SO<sub>4</sub>-S = HI-reducible S – inorganic SO<sub>4</sub>-S; cumulative S mineralization = cumulative net S mineralization over 27 weeks).

\*Transformation of a zero-valued outlier resulted in a missing value.

**Table 5.** Six-year growth increments in single-tree plots of lodgepole pine by treatment ( $n = 15$ ).

	Control		N-only treatment		S <sup>0</sup> treatment		AS treatment	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Height increment (m)	2.74a	0.48	2.28b	0.35	2.68a	0.41	2.73a	0.45
Basal area increment (cm <sup>2</sup> )	47.5a	12.9	51.4ab	11.5	60.4bc	14.3	68.2c	13.4
Volume increment (dm <sup>3</sup> )	21.8a	5.9	21.2a	5.3	26.8b	6.4	29.2b	7.0

Note: The data are on file at the Kalmalka Forestry Centre, Research Branch, British Columbia Ministry of Forests, Vernon (E.P. 886.10, R.P. Brockley, 1996). The N-only treatment consisted of 400 kg N·ha<sup>-1</sup> (urea); the S<sup>0</sup> treatment consisted of 400 kg N·ha<sup>-1</sup> (urea) + 100 kg S·ha<sup>-1</sup> (elemental S); the AS treatment consisted of 400 kg N·ha<sup>-1</sup> + 100 kg S·ha<sup>-1</sup> (urea + ammonium sulphate). Values in a row followed by a different letter are significantly different (Fisher's LSD,  $p < 0.05$ ).

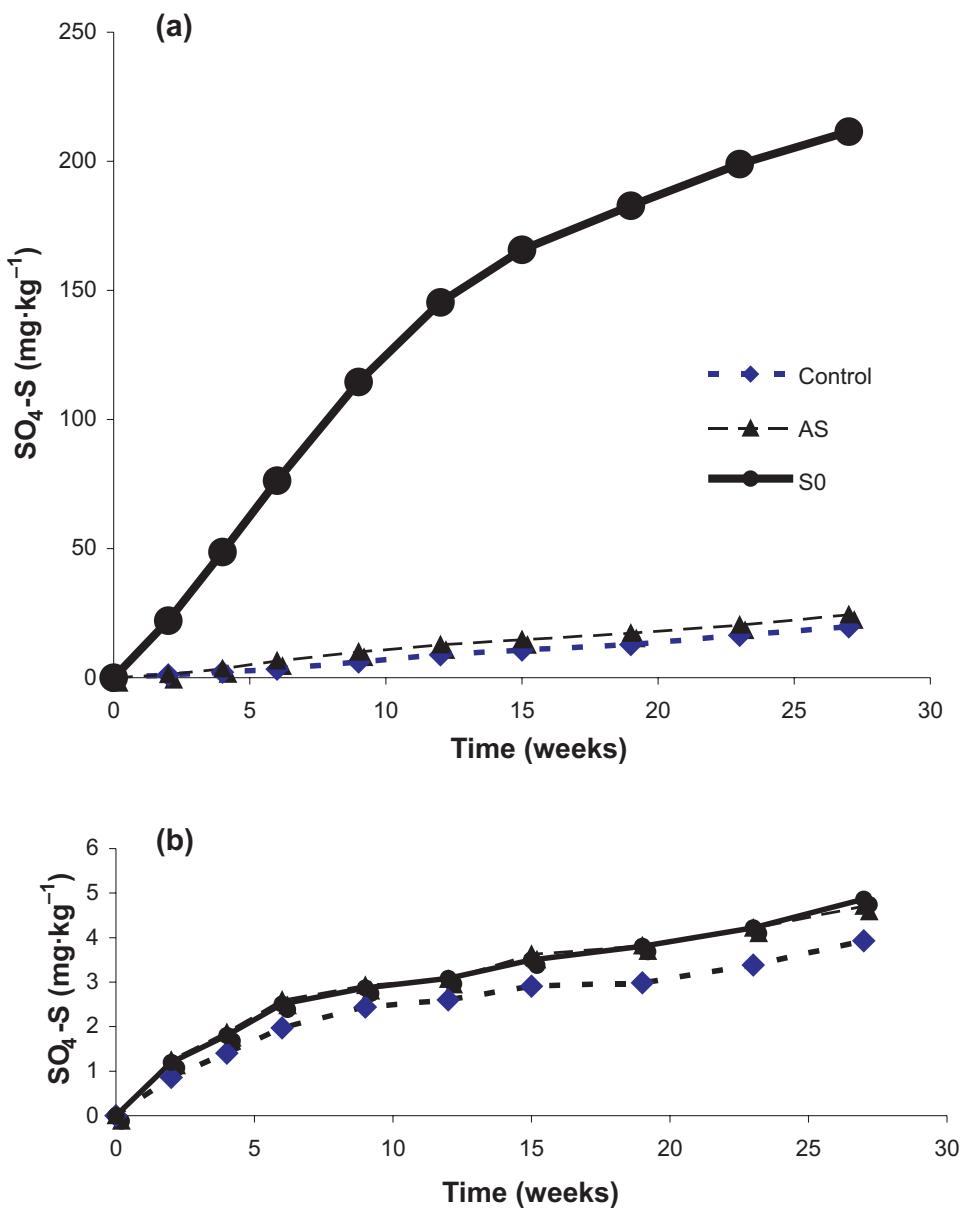
proved over the control by these N + S treatments, volume increment was significantly improved relative to both control and N-only treatments. Although the basal area and volume increments in the S<sup>0</sup> treatment were somewhat lower than in the AS treatment, these differences were not significant, suggesting that at these fertilization rates, the two S sources were equally effective in improving tree growth above that of the control. Tree growth measurements were not obtained after year 6 at this site, as single-tree experimental plots are not suitable for detecting treatment effects on growth over longer time periods. Area-based installations currently established elsewhere in central interior B.C. will allow any longer term growth responses to different S sources and application rates to be addressed.

These soil and foliar data have practical implications for nutritional management of S-deficient lodgepole pine in the

B.C. interior. Twelve years after fertilization, the low foliar N levels in response to these N + S treatments indicate that the stand might benefit from additional N fertilization. Based on the low foliar SO<sub>4</sub>-S levels in the AS plots, a combined application of N and S would likely be required to ensure a favourable growth response following refertilization of this stand if the previous treatment used AS as the S source (Brockley 2000). However, the higher foliar SO<sub>4</sub>-S levels in the S<sup>0</sup> treatment indicate that S could probably be excluded from the fertilizer prescription if S<sup>0</sup> had been used as the S source in the previous fertilization.

At a similar site within 20 km of the Cluculz Creek installation, we have estimated annual N-fixation rates of 10–15 kg·ha<sup>-1</sup> by Sitka alder (*Alnus sinuata* (Reg.) Rydb.) and observed potential imbalances with other nutrients, particularly S, in lodgepole pine foliage, apparently created by this

**Fig. 1.** Cumulative net S mineralization after a 27-week aerobic incubation of forest-floor material (a) and mineral soils (0–20 cm) (b) under control, ammonium sulphate (AS), and N<sup>+</sup> elemental S (S<sup>0</sup>) treatments established at Cluculz Creek, B.C., in 1990.



increased N availability (Sanborn et al. 2002; Brockley and Sanborn 2003). These earlier findings, combined with those of the present study, suggest that long-term amelioration of N and S deficiencies in lodgepole pine stands could be achieved by combining symbiotic N-fixation with S<sup>0</sup> fertilization. Additional field experiments should be established to quantify the stand-level growth responses to such combined treatments.

## Conclusions

Twelve years after fertilization (100 kg S·ha<sup>-1</sup> + 400 kg N·ha<sup>-1</sup>) of an S-deficient lodgepole pine stand in central interior B.C., there were significantly higher foliar SO<sub>4</sub>-S and forest-floor S-fraction concentrations and net S mineralization rates in the treatment that used S<sup>0</sup> compared with ammonium sulphate treatment. Treatment-related differences in S-mineralization rates and S-fraction concentrations were

largely absent from the surface mineral soil. Retained fertilizer S derived from the S<sup>0</sup> source was largely in organic form in the forest floor. The results of this study suggest that a single application of S<sup>0</sup> (with N) may provide prolonged amelioration of S deficiency in managed lodgepole pine stands while producing volume-growth rates that differ little from those obtained after fertilization with readily available ammonium sulphate. Further research should examine the mechanisms of S retention and the long-term fate and mobility of applied fertilizer S in these ecosystems.

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