

Using foliar variables to predict the response of lodgepole pine to nitrogen and sulphur fertilization

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Abstract: Six-year basal area responses to nitrogen (N) fertilizer, alone and in combination with sulphur (S), in 31 lodgepole pine (*Pinus contorta* Dougl. var. *latifolia* Engelm.) stands in the interior of British Columbia were compared with pre-treatment measures of foliar N and S and first-year increases in fascicle mass to determine the utility of these variables for predicting growth response and for determining appropriate fertilizer prescriptions. Results indicate that pre-fertilization levels of foliar N or inorganic sulphate-S (SO_4) may be more reliable than the fascicle mass screening method for predicting whether or not a significant stemwood response will occur following N fertilization. When combined, N and SO_4 levels in unfertilized foliage explained 68% of the variation in relative basal area response to fertilization with N alone. Foliar nutrients were also useful for determining whether or not lodgepole pine would respond incrementally to S when added in combination with N. Stands in which pre-fertilization foliar SO_4 was ≤ 60 mg/kg and N/S ratio was ≥ 13 did not respond significantly to N alone but always responded significantly to N + S. Conversely, a foliar SO_4 level > 60 mg/kg combined with a N/S ratio of ≤ 12 always resulted in a favourable response to N with no incremental benefit of added S.

Résumé : L'effet sur la surface terrière après six ans d'une fertilisation azotée (N), appliquée seule ou avec du soufre (S), dans 31 peuplements de pin lodgepole (*Pinus contorta* Dougl. var. *latifolia* Engelm.) situés dans la zone intérieure en Colombie Britannique a été étudié. Les résultats ont été comparés à des mesures de N et S foliaires prises préalablement aux traitements et à l'augmentation du poids des fascicules la première année dans le but de déterminer l'utilité de ces variables pour prédire l'effet de la fertilisation sur la croissance ainsi que la quantité appropriée de fertilisant à prescrire. Les résultats montrent que les niveaux préalables aux traitements de N ou S foliaires, sous forme de sulphate inorganique (SO_4), sont plus fiables que la méthode de dépistage qui consiste à peser les fascicules pour prédire si une fertilisation azotée va provoquer ou non une croissance importante de la tige. Combinés, les niveaux de N et de SO_4 dans le feuillage non fertilisé expliquent 68% de la variation dans la réaction relative de la surface terrière à la fertilisation azotée seule. Les nutriments foliaires étaient également utiles pour déterminer si le pin lodgepole réagirait positivement ou non à l'ajout de S en combinaison avec N. Les peuplements dans lesquels le niveau de SO_4 foliaire était ≤ 60 mg/kg et le rapport N/S était ≥ 13 avant la fertilisation n'ont pas réagi significativement à N seul mais ont toujours réagi significativement à N + S. À l'inverse, un niveau de SO_4 foliaire > 60 mg/kg combiné à un rapport N/S ≤ 12 a toujours été associé à une réaction favorable à N sans effet supplémentaire dû à l'addition de S.

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Introduction

Lodgepole pine (*Pinus contorta* Dougl. var. *latifolia* Engelm.) is the most widespread conifer in British Columbia, occurring in all but the coldest and driest of the 11 interior biogeoclimatic (BEC) zones (Meidinger and Pojar 1991). In 1996–1997, its harvested volume of 19.9×10^6 m³ accounted for 39% of the total harvest volume from interior forests (B.C. Ministry of Forests 1998), making it the leading commercial tree species in the province. The species regenerates naturally and abundantly following wildfires and clearcut harvesting. Lodgepole pine has also been extensively planted during the past two decades and presently accounts for approximately one half of all seedlings planted

annually in the B.C. interior. Large silvicultural investments have been made in young stands to increase the yield and value of merchantable wood produced from this sizable lodgepole pine resource. Although pre-commercial thinning and pruning operations may improve the value of harvested wood, they generally do not increase harvest volumes. Forest fertilization is unique, because if applied to appropriate species and sites, it not only increases the size of individual stems but also produces "extra" wood per hectare.

Extensive research has been undertaken to determine the nutritional status of lodgepole pine in the B.C. interior and to document the effectiveness of fertilization in improving the growth of immature stands (Brockley 1991a, 1996). These studies have confirmed that N deficiencies are widespread throughout the region and that N additions often have a substantial positive effect on tree and stand growth. However, the response of lodgepole pine following N additions is variable; some stands respond well, and others respond poorly. Sulphur (S) has been implicated in limiting the

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effectiveness of N additions in some instances (Brockley 1990, 1991*b*, 1995). On some sites, growth responses have been enhanced by combining S with N in fertilizer prescriptions (Mika et al. 1992; Brockley and Sheran 1994). However, methods are not presently available to reliably predict which stands will respond best to N fertilization or to identify stands where response can be improved by adding S in combination with N. The absence of a reliable prediction system is undoubtedly a major factor limiting the large-scale use of fertilizers in interior forest operations.

A positive correlation between the mass of needles (e.g., g/100 needles) produced during the first year after fertilization and subsequent stemwood response has been documented for several species (Camiré and Bernier 1981; Timmer and Stone 1978; Timmer and Morrow 1984; Valentine and Allen 1990). In British Columbia, the "screening" method has been widely accepted as a fairly reliable predictor of whether or not a significant stemwood response will occur following N fertilization of lodgepole pine (Weetman and Fournier 1982; Weetman et al. 1988). Fertilizer screening trials are frequently established in candidate stands to diagnose specific nutrient deficiencies and to finalize stand selections for large-scale fertilizer operations. However, it is still unclear whether or not the technique can be reliably used to predict the magnitude of stemwood response, to rank stands on the basis of fertilization response potential, or to identify which stands will respond better when other nutrients are added in combination with N.

In British Columbia, foliage samples are routinely collected from lodgepole pine candidate stands being considered for operational fertilization. Foliar nutrient analyses of these samples are compared with published diagnostic criteria to confirm N deficiencies and to infer whether other nutrients are either growth limiting or likely to become growth limiting following N additions. Pre-treatment foliar analysis is not presently used to predict the responsiveness of lodgepole pine to fertilization. However, empirical relationships and predictive indices, using pre-fertilization measures of foliar N and S, have been successfully developed for many tree species to identify sites that will respond to N fertilization and (or) to predict the magnitude of the response (Turner et al. 1977, 1979, 1988; Hopmans and Chappell 1994; Sikstrom et al. 1998). Because of the large differences in the amount of inorganic sulphate-S (SO_4) often found in the foliage of S-deficient and S-sufficient plants, SO_4 has been reported to be better than total S as an indicator of plant S status (Freney and Spencer 1967). The fact that N, S, and SO_4 levels in lodgepole pine foliage are often low, and that enhanced growth responses to combined N and S additions have been reported (Brockley and Sheran 1994), suggests that pre-treatment measures of N, S, and SO_4 may have utility as predictors of fertilization response for lodgepole pine in the interior of British Columbia.

This paper compares 6-year basal area responses to N fertilizer, alone and in combination with S, with pre-treatment measures of foliar N and S and post-fertilization (i.e., first-year) increases in fascicle mass in 31 lodgepole pine stands in the interior of British Columbia. The primary objective was to determine the utility of these foliar variables as predictors of growth response and for determining appropriate fertilizer prescriptions.

Methods

Study sites

The growth response and foliar data used for this analysis were collected from a subset of lodgepole pine fertilization research installations established by the B.C. Ministry of Forests from which 6-year growth response data are presently available.

The 31 study sites are located in the interior of British Columbia between 49°18' and 54°39'N and between 115°30' and 127°22'W. Twenty of the stands regenerated naturally following wildfire; the remainder were more-or-less equally divided between naturally regenerated, harvest-origin stands, and plantations. The harvest and planting of lodgepole pine in the interior of British Columbia did not become widespread until the early 1970s. Therefore, until recently, few stands of these origins were old enough to be considered for fertilizer operations.

All of the naturally regenerated harvest- and fire-origin stands were pre-commercially thinned prior to fertilization to provide opportunities for crown expansion. Some of the stands had been operationally thinned prior to installation establishment, and others were thinned at the time of installation establishment. Post-thinning densities cover the range typically used operationally throughout the region.

Twenty-seven of the installations are located within the Sub-boreal Spruce (SBS) or Montane Spruce (MS) BEC zones (Meidinger and Pojar 1991). This distribution reflects the abundance of immature lodgepole pine and pre-commercial thinning activity in these two zones. Two installations are located within the Interior Cedar-Hemlock (ICH) BEC zone; the remaining sites are within the Sub-boreal Pine Spruce (SBPS) and Interior Douglas-fir (IDF) zones. Stand age averaged 20 years (range 13–30 years) at the time of fertilization.

Soils were derived from either glacial till or glaciofluvial parent material. Parent materials were predominantly of mixed metamorphic-sedimentary lithology in the southeastern interior (often slightly calcareous) and predominantly of igneous intrusive lithology in the south-central and central interior. Except for those derived from glaciofluvial material, soils in the southeastern interior tended to be finer textured (silt-loam to loam) than those in the central interior (loam to sandy loam). Based on morphological criteria, soils in the ICH biogeoclimatic zone and in the wet cool central subzone of the SBS were classified as Humo-Ferric Podzols. The soils in all other installations were classified as either Brunisols or Luvisols (Agriculture Canada Expert Committee on Soil Survey 1987).

Experimental design

As shown in Table 1, a combination of conventional, fixed-area-plot fertilization research installations, "mini-plot" experiments, and "single-tree" fertilizer screening trials was used in this analysis. These installations were selected from 10 separate experimental projects that were established between 1981 and 1992. Because each experiment was established for specific objectives, the experimental designs, plot layouts, and treatments are not consistent. Preliminary results and descriptions of experimental designs and treatments for many of these experiments have been reported previously (Brockley 1989, 1990, 1991*b*, 1995; Mika et al. 1992; Brockley and Sheran 1994).

Nitrogen was the only nutrient added in 14 of the 31 installations. The other 17 installations received both N and N + S additions. Some of the experiments included additional treatments to those listed in Table 1 that are not relevant to the objectives of the present analysis.

Each conventional and mini-plot treatment consisted of an inner "measurement" plot surrounded by a treated buffer. In conventional installations, each treatment was applied to three treatment plots.

Table 1. Distribution of installations by experimental project, plot type, and treatment.

Experimental project No.	Plot type	No. of installations	Treatments*	Treatment plot size (ha)	Replicate plots per treatment	No. of trees measured per plot
886.01-1	Fixed area	10	C, N	0.059–0.091	3	50
886.01-2	Fixed area	3	C, N N+S	0.059–0.091	3	50
886.01-3	Fixed area	2	C, N, N+S	0.070–0.091	3	50
886.13	Fixed area	1	C, N, N+S	0.164	3	64
886.12	Miniplot	1	C, N, N+S	0.031	5	15
886.04-1	Single tree	3	C, N	0.008	15	1
886.04-2	Single tree	1	C, N, N+S	0.008	15	1
886.05	Single tree	1	C, N	0.008	15	1
886.09	Single tree	7	C, N, N+S	0.008	15	1
886.10	Single tree	2	C, N, N+S	0.008	15	1

*C, control; N, nitrogen only; N+S, nitrogen + sulphur.

Table 2. Fertilizer sources and application rates for nitrogen and sulphur by experimental project and treatment.

Experimental project No.	N only		N + S		S source(s) [†]	S rate (kg/ha)
	N source(s)	N rate (kg/ha)	N source(s)*	N rate (kg/ha)		
886.01-1	46:0:0	200				
886.01-2	46:0:0	200	46:0:0	200	Elemental S	58
886.01-3	46:0:0	200	46:0:0	134	21:0:0	75
			21:0:0	66		
886.13	46:0:0	200	46:0:0	156	21:0:0	50
			21:0:0	44		
886.12	46:0:0	300	46:0:0	234	21:0:0	75
			21:0:0	66		
886.04-1	46:0:0	200				
886.04-2	46:0:0	200	46:0:0	200	Elemental S	58
886.05	46:0:0	200				
886.09	46:0:0	134	46:0:0	134	21:0:0	75
	26:0:0	66	21:0:0	66		
886.10	46:0:0	200	46:0:0	167	Elemental S	37.5
			21:0:0	33	21:0:0	37.5

*46:0:0 (urea); 21:0:0 (ammonium sulphate, 24% S).

[†]Elemental S (finely divided S, 100% S); 21:0:0 (ammonium sulphate, 24% S).

Mini-plot treatments were replicated five times. The outer boundaries of adjacent treatment plots were separated by a minimum distance of 5 m. In single-tree trials, fertilizer treatments were applied to a 5-m radius area surrounding each single-tree plot. Each treatment was randomly assigned to 15 plots within each installation, and adjacent single-tree plots were separated by a minimum distance of 15 m.

In thinned installations, all treatment plots (including controls) were thinned in the same year and to the same post-thinning density.

Fertilizer application

Fertilizer N and S sources and application rates for the various experimental projects are shown in Table 2. In all but one experiment, the N application rate was 200 kg/ha. Urea (46:0:0, N:P:K) was the primary N source in all experiments, although N + S treatments often used a combination of urea and ammonium sulphate (21:0:0) as sources of N. This obviously creates a confounding effect of N source when comparing growth responses obtained from the N and N + S treatments. In one experiment (experimental project (EP) No. 886.09), two N-only treatments, with different ratios of urea or ammonium chloride (26:0:0), were used to test whether

response to N was affected by N source. The ammonium chloride was used as a substitute for the ammonium sulphate in the N + S treatment. Results showed the effect of N source to be insignificant, which indicated that differences between N and N + S treatments were likely due to the added S rather than to differences in N source (Brockley and Sheran 1994). It is assumed that this conclusion applies to other experiments used in this analysis.

Sulphur was applied as ammonium sulphate or finely divided elemental S (S^o) at application rates between 50 and 75 kg/ha. In one experiment, the S^o was added separately by hand. Otherwise, the S^o was integral with a forest-grade urea prill (41:0:0:12, N:P:K:S).

All fertilizers were applied by hand to the entire treatment plot at the beginning of each experiment. Each plot was divided into a number of segments to facilitate uniform application, and pre-measured amounts of the specified fertilizers were applied to each segment. Except for one installation, fertilization was undertaken in the fall, prior to snowpack accumulation.

Foliar analysis

In all conventional, fixed-area installations, foliage samples were collected from 10 dominant or codominant trees within each

measurement plot immediately prior to fertilization and again after the first and third growing seasons. In three installations, samples were also collected after 2 years. In the mini-plot experiment, foliage was collected from five trees per plot before fertilization and after 1, 2, and 3 years. For each sampling year, one composite sample per measurement plot, consisting of equal amounts of foliage from each of the sampled trees, was prepared for chemical analysis. The dry mass of fascicles (g/100 fascicles) that were produced in the first year after fertilization by each of the sampled trees per plot was measured and recorded.

In the single-tree experiments, foliage was collected from the central tree within each plot before fertilization and after the first and third growing seasons. In four experiments, foliage was also collected after the second year. For most experiments, one composite sample per treatment, consisting of equal amounts of foliage from each of the 15 single-tree plots, was prepared for chemical analysis after each sampling year. In two experiments (EP Nos. 886.09 and 886.10), three composite samples per treatment, each composite consisting of equal amounts of foliage from five of the single-tree plots, were prepared for chemical analysis after each sampling year. In all single-tree experiments, the dry mass of fascicles (g/100 fascicles) that were produced in the first year after fertilization by each of the 15 trees per treatment was measured and recorded.

All samples consisted of current year's foliage taken from near the base of the upper one third of the live crown. Samples were frozen prior to oven-drying at 70°C for 16–24 h. Dried, composite samples were ground in an electric coffee grinder prior to shipment to a commercial laboratory for chemical analysis. The same commercial laboratory was used for all experiments and measurement periods.

Dried foliage samples were digested using a variation of the sulphuric acid – hydrogen peroxide procedure described by Parkinson and Allen (1975). The digests were analyzed colorimetrically for N using the Berthelot (phenol–hypochlorite) reaction (Weatherburn 1967) in a Technicon autoanalyzer II. Total S was determined with a Leco SC-132 sulphur analyzer. Inorganic SO₄ was extracted with 0.1 M HCl (1 g foliage per 20 mL of HCl boiled for 20 min) followed by hydriodic acid (HI) reduction of the extract and bismuth colorimetry using the procedure of Johnson and Nishita (1952).

To document interlaboratory variation in N, S, and SO₄ analyses, the analytical results from the commercial laboratory used in this study were recently compared with another commercial laboratory. Both laboratories are used extensively by forestry clients in British Columbia for the nutrient analysis of conifer foliage. After oven-drying and grinding, homogeneous subsamples of 81 individual lodgepole pine foliage samples collected from fertilizer experiments not reported on in the present study were submitted to both laboratories. The two laboratories used different methods of extraction and (or) determination for N, S, and SO₄. In the second laboratory, foliage was digested using the Kjeldahl method (Bremner and Mulvaney 1982) followed by colorimetric determination of N in a Technicon autoanalyzer II. Wet combustion with nitric and perchloric acids, followed by determination with an inductively coupled plasma spectrophotometer (ICP), was used for total S analysis in the second laboratory. Both laboratories used 0.1 M HCl for extraction of SO₄. However, the second laboratory did not use boiling acid, and the extract was shaken for 20 min prior to determination with an ICP.

Measurements

In conventional and miniplot fixed-area installations, diameter at breast height (DBH) measurements were made on all trees within each measurement plot at the time of establishment and after six growing seasons. In single-tree installations, the central tree within each plot was measured for DBH at establishment and after six

years. All DBH measurements were taken with a steel diameter tape. Breast height measurements were taken at 1.30 m above the ground, measured from the uphill side of the tree.

Data analysis

Two measures of fertilizer response were used in this analysis: mean first-year fascicle mass and mean individual-tree 6-year basal area (BA) increment. Although ultimately not as useful to the forest manager as area-based fertilization response, individual-tree growth response analyses generally give more sensitive measures of growth-response potential. In area-based analyses, the positive effects of nutrient additions on tree growth can be easily masked by the non-treatment-related plot variability (e.g., mortality, level of growing stock) that is inherent to forest research field experiments.

For individual installations, first-year fascicle mass was analyzed by analysis of variance (ANOVA), using the general linear models procedure (SAS Institute Inc. 1989). Because pre-fertilization fascicle masses were not determined, all analyses assumed that between-treatment differences in first-year fascicle mass were solely treatment related. Within each measurement plot, BA increments were calculated for all trees alive after 6 years. For each tree, BA increment was taken as the difference between the initial and 6-year stem measurement. Increments were adjusted by covariance analysis, using initial BA as the covariate. For fascicle mass and BA analyses, two single degree-of-freedom contrasts were undertaken; one tested average control response versus average response to N alone, and the other tested average N alone response versus average N + S response. A probability threshold of $p = 0.05$ is used throughout the text for inferring statistical significance. For comparative purposes, the difference between treated and control values for each installation was expressed relative to the control value (control = 100).

For each installation, the mean foliar N, total S, and SO₄-S concentrations and N/S ratios in current-year needles were calculated based on all available data from unfertilized trees during the first 3 years of the experiment. The foliar data for all treatment plots were used in the case of pre-fertilization foliar sampling. For post-fertilization foliar sampling, only the data from control plots were used in the calculations.

To examine relationships between 6-year relative BA response (dependent variable) and various independent variables (first-year fascicle mass, foliar N, S, SO₄, and N/S for unfertilized trees), exploratory curve fitting with linear and curvilinear models using one and two independent variables was undertaken using SigmaPlot (SPSS Inc. 1997). In the absence of compelling evidence indicating any of these relationships were curvilinear, the stepwise regression procedure in the SAS statistical package was used to select the "best" linear model (SAS Institute Inc. 1989). A significance level of 0.15 used for the entry and retention of independent variables.

Results

Fascicle mass response to N and N + S fertilization

Fascicle mass response to fertilization with N alone was statistically significant ($p < 0.05$) in 23 (74%) of the 31 installations (Table 3). Overall, fascicle mass response to N fertilization averaged 26% (range –4 to 110%).

Seventeen installations were fertilized with both N and N + S. When averaged, a combined application of N + S resulted in a larger fascicle mass response than N alone (43 vs. 20%, respectively). Eleven (65%) of 17 installations responded significantly to N alone; 94% (16 of 17) responded significantly to combined N + S fertilization.

Table 3. Mean relative first-year fascicle mass responses and 6-year basal area responses to N and N + S fertilization with mean unfertilized foliar nutrient concentrations by experimental project and installation.

Experimental project No.	Installation	Fascicle mass response (control = 100)			Basal area response (control = 100)			Unfertilized foliar			
		N	N+S	N vs. N+S	N	N+S	N vs. N+S	N (g/kg)	S (g/kg)	SO ₄ (mg/kg)	N/S ratio
886.01-1	1	155*			131*			10.9	1.00	101	11.0
886.01-1	2	141*			142*			11.2	1.02	105	11.0
886.01-1	3	158*			145*			9.7	0.95	126	10.3
886.01-1	4	210*			176*			9.6	0.93	170	10.4
886.01-1	5	151*			134*			10.6	0.97	117	11.0
886.01-1	6	139*			170*			9.3	0.84	77	11.1
886.01-1	7	118*			132*			10.0	0.90	74	11.2
886.01-1	8	123*			114			9.9	0.86	47	11.5
886.01-1	9	132*			124			9.8	0.88	51	11.2
886.01-1	11	112			135*			10.3	0.93	76	11.1
886.04-1	1	108			108			11.2	0.98	11	11.4
886.04-1	2	140*			134*			10.1	1.02	151	9.9
886.04-1	3	127*			159*			9.7	0.94	102	10.3
886.05	1	117*			133*			10.6	1.00	71	10.6
886.01-2	12	140*	152*		177*	169*		10.0	0.89	115	11.3
886.01-2	13	117*	127*		116	127*		11.2	0.92	58	12.4
886.01-2	15	118*	120*		112	131*	**	12.0	0.91	40	13.3
886.01-3	17	107	169*	**	123	147*		11.1	0.87	50	12.7
886.01-3	18	123*	182*	**	125*	133*		9.9	0.79	53	12.6
886.04-2	4	104	112*		111	125*		12.0	0.95	42	12.6
886.09	1	129*	140*		111	121*		12.4	0.90	72	13.9
886.09	2	118	143*	**	116*	112*		12.0	0.85	71	14.4
886.09	3	120*	144*	**	118	139*	**	11.2	0.78	53	14.4
886.09	4	113	138*	**	133	167*	**	12.0	0.86	55	14.0
886.09	5	96	166*	**	113	140*	**	12.6	0.91	45	13.9
886.09	6	117	119		109	109		12.2	0.97	59	12.6
886.09	7	124*	133*		150*	145*		9.8	0.84	100	11.8
886.10	1	136*	137*		115	113		11.9	0.96	57	12.3
886.10	2	124*	157*	**	130*	127*		11.5	0.89	49	12.9
886.12	1	120*	151*	**	105	119*		11.3	0.84	42	13.4
886.13	1	154*	163*		117*	125*		11.3	0.91	54	12.5

Note: For N and N + S treatments, values marked with an asterisk are significantly different from control ($p < 0.05$). For N vs. N + S, two asterisks show response to N + S is significantly different than N alone ($p < 0.05$).

Eight (47%) of the 17 installations showed significantly larger fascicle mass response to N + S than to N alone (Table 3). For these stands, average responses to N and N + S were 15 and 56%, respectively.

Basal area response to N and N + S fertilization

Basal area response following fertilization with N alone was statistically significant ($p < 0.05$) in 17 (55%) of the 31 installations (Table 3). Response to N fertilizer varied considerably between installations as shown by the frequency distribution of relative BA response (Fig. 1). Overall, 6-year BA response to N fertilization averaged 26% (range 5–77%). Almost one quarter of the installations produced BA responses in excess of 40%. However, BA response in eight of the installations was less than 15%, indicating marginal benefit from the application of N fertilizer for those installations.

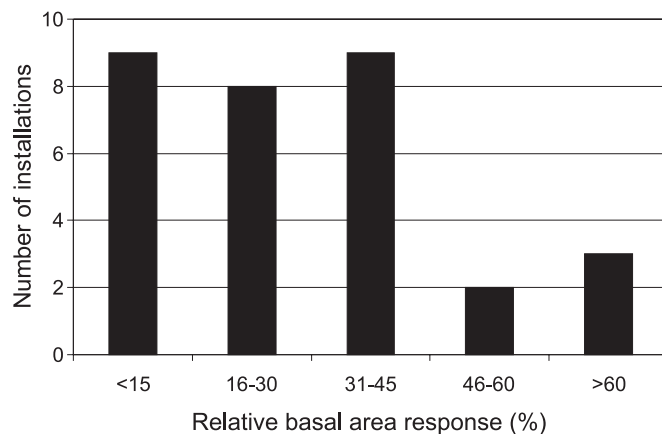
In the 17 installations fertilized with both N and N + S, the combined N + S application resulted in a larger mean 6-

year BA response than N alone (28 vs. 19%, respectively). Six (35%) of the 17 installations responded significantly to N alone; 88% (15 of 17) responded significantly to combined N + S fertilization. A BA response of more than 30% was obtained in one half of the installations fertilized with N + S. A similar response was achieved by less than one fifth of the stands fertilized with N alone.

In the six installations that responded significantly to N alone, average BA responses to N and N + S were 33 and 32%, respectively. In the 11 installations that did not respond significantly to N alone, average 6-year BA responses to N and N + S were 13 and 26%, respectively.

Four (22%) of the 17 stands showed significantly larger 6-year growth responses to N + S than to N alone. In five other stands, the response to N + S was statistically significant, whereas response to N alone was not. For those nine stands showing some positive effect of added S, average responses to N and N + S were 14 and 32%, respectively. Two stands did not respond to either N or N + S fertilization.

Fig. 1. Distribution of relative 6-year basal area response (% increase above control) to fertilization with N alone.



Fascicle mass as a predictor of BA response

Fifteen (88%) of the 17 installations with a significant relative BA response to N alone also had a significant increase in first-year relative fascicle mass (Table 3). Basal area response was significant in only two (25%) of the eight installations in which fascicle mass response was not significant. Conversely, eight (35%) of the 23 installations with a significant fascicle mass response did not show a significant BA response.

All eight installations in which first-year fascicle mass response to N fertilization was $\geq 40\%$ showed a significant BA response. The average relative BA response for these installations was 42%. For installations in which first-year fascicle mass response was $\leq 20\%$, only four (29%) of 14 showed a significant BA response. For these 14 installations, the mean BA response to N alone was only 16%.

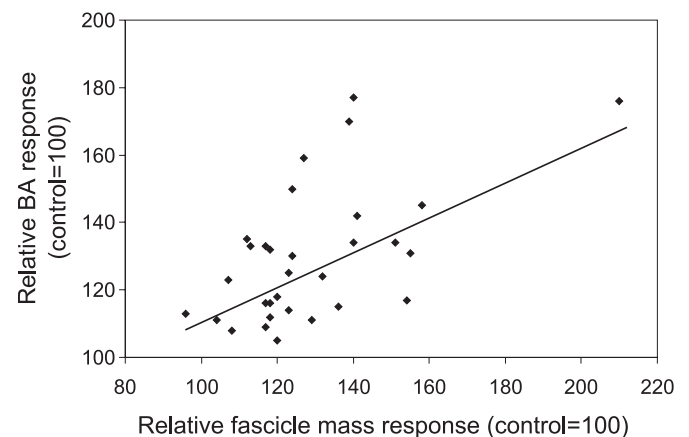
As shown in Fig. 2, first-year changes in relative fascicle mass following fertilization with N alone were significantly and positively related to 6-year relative BA response. However, fascicle mass explained only 34% of the variation in BA response.

For the 17 installations fertilized with both N alone and N + S, relative first-year fascicle mass differences between fertilization with N alone and N + S were plotted against relative differential BA responses. Although the relationship was weakly positive ($p = 0.09$), differential fascicle mass responses accounted for only 18% of the observed variance in the differential BA response for the 17 installations. On the basis of statistical significance ($p < 0.05$), however, fascicle mass correctly predicted whether or not added S would have a positive impact on BA increment in 11 (65%) of the 17 installations (Table 3). In three installations, the positive effect of added S on BA increment was not predicted based on fascicle mass responses to N and N + S treatments. Also, three installations that showed a significant fascicle mass response to S fertilization did not show a significant BA response.

Foliar measures of N and S as predictors of BA response

Basal area response to fertilization with N alone was significant in 13 (87%) of 15 installations with ≤ 11 g/kg of N in the foliage of unfertilized trees (Table 4). The average BA

Fig. 2. Relationship between relative 6-year basal area response and first-year fascicle mass response to fertilization with N alone ($n = 31$). The regression line is fit from the following equation: BA response (control = 100) = $61.0 + 0.53 \times$ fascicle mass (control = 100), $R^2 = 0.34$, $SE_E = 16.4$.



response in these 15 installations was 42%. However, only one (14%) of the seven installations with ≥ 12 g/kg of foliar N responded significantly to N additions. In these installations, BA responses averaged 13%.

All eight installations in which pre-fertilization foliar N/S ratio was ≤ 11 showed a significant BA response to fertilization with N alone (Table 4). The average BA response in these installations was 42%. Conversely, only one (14%) of seven installations in which pre-fertilization N/S ratio was ≥ 13 showed a significant BA response following N fertilization. The average response for these 15 installations was 14%. Installations in the top response quartile (i.e., the eight most responsive installations) had an average pre-fertilization N/S ratio of 10.9. Foliar N/S ratios in the bottom response quartile averaged 12.8.

All nine installations with ≥ 80 mg/kg of SO_4 in unfertilized foliage showed a significant BA response to fertilization with N alone (Table 4). The average BA response in these installations was 47%. However, only three of 16 (19%) installations with ≤ 60 mg/kg of SO_4 showed a significant BA response. The average response for these 16 installations was only 15%. Installations in the top response quartile had an average SO_4 concentration of 109 mg/kg. Foliar SO_4 levels in the bottom response quartile averaged only 45 mg/kg.

The prediction of significant BA response to fertilization with N alone was especially good when foliar N and SO_4 levels were used in combination. All 12 installations with ≤ 11 g/kg foliar N and >60 mg/kg foliar SO_4 showed a significant 6-year BA response to fertilization with N alone. The 6-year relative BA response in these 12 installations averaged 46%. Conversely, none of the five installations with ≥ 12 g/kg foliar N and ≤ 60 mg/kg foliar SO_4 responded significantly to fertilization with N alone. The average BA response in these installations was only 13%.

From scatter diagrams, relationships between 6-year relative BA response following N fertilization and foliar levels of N, S, and SO_4 and N/S ratio in unfertilized trees were examined using regression analyses. As illustrated in Fig. 3, an overall trend of declining BA response was observed with increasing N concentration in the foliage of unfertilized trees

Table 4. Number of installations showing significant ($p < 0.05$) first-year fascicle mass responses and 6-year basal area responses following fertilization with N alone by unfertilized foliar N, SO₄, and N/S classes.

	Foliar N (g/kg)			Foliar SO ₄ (mg/kg)			Foliar N/S ratio		
	≤11	>11 to <12	≥12	≤60	>60 to <80	≥80	≤11	>11 to <13	≥13
No. of installations	15	9	7	16	6	9	8	16	7
No. showing significant fascicle mass response	14	7	2	10	4	9	8	11	4
No. showing significant BA response	13	3	1	3	4	9	8	8	1
Mean BA response above control (%)	42	19	13	15	28	47	42	24	14

Fig. 3. Relationship between relative 6-year basal area response to fertilization with N alone and the concentration of N in foliage of unfertilized trees ($n = 31$). The regression line is fit from the following equation: BA response (control = 100) = $285.8 - 14.36 \times N$ (g/kg), $R^2 = 0.49$, $SE_E = 14.4$.

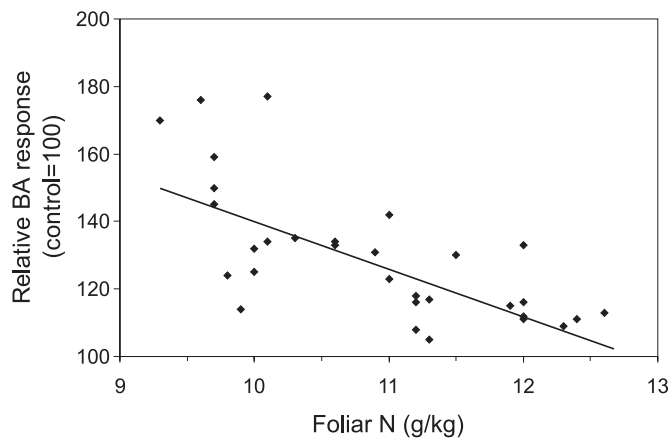
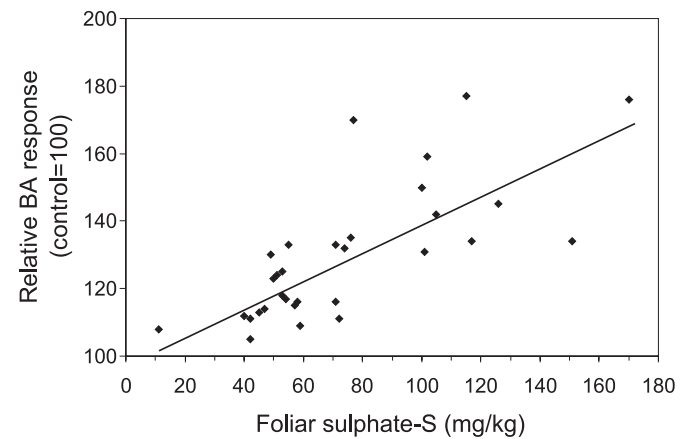


Fig. 4. Relationship between relative 6-year basal area response to fertilization with N alone and the concentration of SO₄-S in foliage of unfertilized trees ($n = 31$). The regression line is fit from the following equation: BA response (control = 100) = $98.9 + 0.41 \times SO_4$ (mg/kg), $R^2 = 0.55$, $SE_E = 13.6$.



($r = -0.70$). Conversely, there was clearly a positive relationship ($r = 0.74$) between relative BA response and foliar SO₄ (Fig. 4). Individually, each of these foliar variables explained approximately one half of the observed variance in relative BA response between installations. A negative relationship was also observed between relative BA response and pre-fertilization N/S ratio in foliage ($r = -0.59$). However, there was no observed relationship between relative BA response following N fertilization and foliar total S ($r = 0.02$).

A multiple regression model using two independent variables (foliar N and SO₄) explained 68% of the variation in relative 6-year BA response. Both of the independent variables were highly significant ($p < 0.01$):

$$[1] \quad \text{BA response (control = 100)} = 204.6 + 0.29 \times \text{SO}_4 \text{ (mg/kg)} - 8.85 \times \text{N (g/kg)}$$

$$SE_E = 11.6, R^2 = 0.68$$

For the 17 installations that were fertilized with both N and N + S, the magnitude of the relative BA response difference between N alone and N with S was only weakly related ($p = 0.14$) to the level of N in the foliage of unfertilized trees ($r = 0.37$). Despite this weak relationship, the level of N in unfertilized foliage was useful in identifying installations that benefited from added S. As shown in Table 5, installations with ≥ 12 g/kg foliar N rarely responded significantly to fertilization with N alone but usually showed significant BA

responses to combined applications of N + S. In fact, almost one half of installations with ≥ 12 g/kg foliar N showed significantly greater responses to combined applications of N + S than to N alone. Conversely, all installations with ≤ 11 g/kg foliar N responded significantly to N alone with little additional benefit from added S.

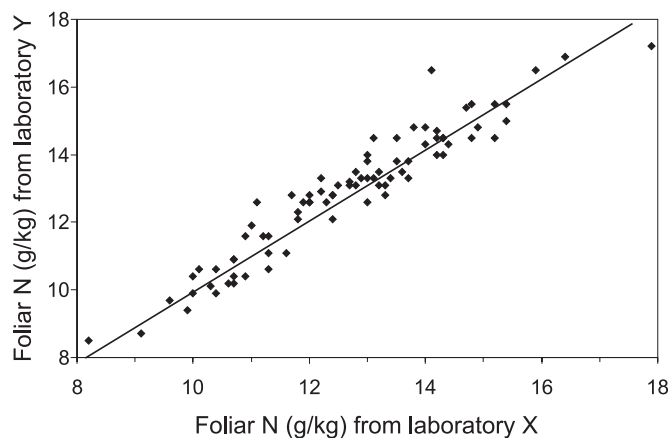
The magnitude of the relative difference in BA response between N alone and N + S was quite strongly related to the level of SO₄ in the foliage of unfertilized trees ($r = -0.61$). Installations with ≥ 80 mg/kg of SO₄ in foliage responded significantly to fertilization with N alone, and larger responses were not achieved by adding S in combination with N (Table 5). Whereas only one quarter of installations with ≤ 60 mg/kg SO₄ responded significantly to N alone, more than four fifths of them responded significantly to combined additions of N and S. In fact, approximately one third of installations with ≤ 60 mg/kg foliar SO₄ showed significantly greater responses to combined applications of N + S than to N alone.

The magnitude of the difference in relative BA response between N alone and N + S was positively related to the N/S ratio in unfertilized foliage ($r = 0.55$). Installations in which pre-fertilization foliar N/S ratio was ≤ 12 responded significantly to fertilization with N alone, and added S did not result in greater response (Table 5). Whereas only one (14%) of seven installations in which N/S ratio was ≥ 13 responded significantly to N alone, all seven of these installations responded significantly when N was added in combination

Table 5. Number of installations showing significant ($p < 0.05$) 6-year basal area responses following N and N + S fertilization by unfertilized foliar N, SO₄, and N/S classes.

	Foliar N (g/kg)			Foliar SO ₄ (mg/kg)			Foliar N/S ratio		
	≤11	>11 to <12	≥12	≤60	>60 to <80	≥80	≤12	>12 to <13	≥13
No. of installations	3	7	7	13	2	2	2	8	7
No. showing significant BA response to N	3	2	1	3	1	2	2	3	1
No. showing significant BA response to N+S	3	6	6	11	2	2	2	6	7
No. showing significantly larger response to N+S than to N alone	0	1	3	4	0	0	0	0	4
Mean BA response to N alone (% above control)	50	17	15	16	13	61	61	16	14
Mean BA response to N+S (% above control)	48	25	30	26	16	55	55	22	29

Fig. 5. Relationship between N concentration in foliage samples submitted to two different commercial laboratories ($n = 81$). The regression line is fit from the following equation: N (laboratory Y) = 0.07 + 1.01 × N (laboratory X), $R^2 = 0.92$, $SE_E = 0.57$.



with S. In fact, over one half of installations in which N/S ratio in unfertilized foliage was ≥ 13 showed significantly greater responses to combined applications of N + S than to N alone (Table 5).

The prediction of significantly larger BA responses to N + S over those achieved with N alone was especially good when pre-fertilization foliar SO₄ levels were combined with foliar N/S ratios. None of the lodgepole pine stands in which pre-fertilization foliar SO₄ was ≤ 60 mg/kg and foliar N/S ≥ 13 responded significantly to fertilization with N alone ($n = 5$). However, these five stands all responded significantly to N + S, and in four (80%) of five cases, the response to N + S was significantly greater than N alone. The relative BA responses to N and N + S were 14 and 36%, respectively, in these five stands. Conversely, a foliar SO₄ level >60 mg/kg combined with a N/S ratio ≤ 12 ($n = 2$) resulted in a favourable response to N with no incremental benefit of added S.

Interlaboratory comparison of foliar nutrient analyses

The foliar N, S, and SO₄ analyses from the commercial laboratory used in this study (laboratory X) were compared with another commercial laboratory (laboratory Y) to test whether the utility of these foliar variables as response predictors is influenced by inter-laboratory variation. As shown in Fig. 5, the N analyses for the two laboratories were in close agreement. The regression had an R^2 of 0.92, and the slope and intercept did not depart significantly from 1.0 and

0, respectively. However, the relationships between the S and SO₄ results from the two laboratories were relatively weak ($r = 0.45$ and $r = 0.64$, respectively). The S results from laboratory X were generally considerably higher than from laboratory Y. Conversely, laboratory X generally reported much lower foliar SO₄ levels than laboratory Y.

Discussion

Results from the 31 fertilization research installations reported in this study confirm that N is an important growth-limiting nutrient in interior lodgepole pine forests. The foliar N levels are typical of those commonly reported for the species (Weetman and Fournier 1982; Yang 1985). According to foliar diagnostic criteria suggested by Ballard and Carter (1986), even those stands with N levels at the upper end of the range reported in this study are slightly to moderately N deficient. Average relative BA gains from N additions compare favorably with those reported previously for lodgepole pine in western Canada (Yang 1985, 1998; Weetman et al. 1988; Preston and Mead 1994). However, the response of lodgepole pine to N fertilization in this study was extremely variable. Although other stand and site factors are undoubtedly involved, results indicate that S deficiencies, either induced or aggravated by N fertilization, may have a strong controlling influence on lodgepole pine growth response following N additions. On some sites, 6-year BA responses were improved by adding S in combination with N. These results are consistent with preliminary indicators of S deficiencies in lodgepole pine previously reported by Brockley and Sheran (1994). A requirement for S in addition to N was also indicated by Yang (1985) for 30-year-old lodgepole pine in Alberta.

Many studies have suggested that nutrient limitations and fertilization response potential can be estimated with reasonable accuracy from first-year increases in the needle mass of fertilized trees (Weetman and Fournier 1982; Timmer and Morrow 1984; Valentine and Allen 1990). The positive relationship between needle mass response and subsequent stemwood response in determinant species is the basis of the "screening" approach to fertilizer response prediction. Timmer and Morrow (1984) reported that first-year increase in fascicle mass of jack pine (*Pinus banksiana* Lamb.) was strongly correlated ($r = 0.88$) to 6-year BA increment in a $N \times P \times K$ factorial experiment. Yang (1998) also reported a strong relationship ($r = 0.91$) between first-year needle mass and 10-year mean relative stand volume increment in a

lodgepole pine thinning \times fertilization factorial trial in Alberta. In both cases, however, the treatments were applied to a single site, and as many as one half of the treatments in each trial did not include N. The non-nitrogen treatments responded similarly to the unfertilized controls, which is not surprising if, as is generally reported, N is the most growth-limiting nutrient in northern temperate and boreal forests. Therefore, the strong relationship between BA response and first-year needle mass in these experiments can be largely explained by the dichotomy in response between fertilized and unfertilized treatments. In an analysis of 15 lodgepole pine fertilizer screening trials in British Columbia, first-year fascicle mass explained only 19% of the variation in 4-year relative BA response from $N \times P \times K$ factorial combinations across these sites (Weetman et al. 1988). Despite this weak relationship, the authors concluded that the screening method offered a fairly reliable prediction of lodgepole pine fertilization response potential. On the basis of statistical significance, first-year needle mass correctly predicted 4-year BA response (or lack of it) to N fertilization in 12 (80%) of the 15 trials. In another study, Valentine and Allen (1990) reported that changes in needle mass correctly predicted a significant 4-year DBH response to N additions in eight of nine stands of loblolly pine (*Pinus taeda* L.). The screening technique also correctly predicted whether or not stands would respond significantly to phosphorus fertilization in seven of the nine stands.

On the basis of statistical significance ($p < 0.05$), fascicle mass response correctly predicted 6-year BA response (or lack of it) to fertilization with N alone in approximately two thirds of the installations in this analysis. In only two instances did changes in needle mass fail to identify responsive stands. In one of these cases, the 6-year BA response was significant but small; thus, the incorrect prediction risks little in potential growth gains. Fascicle mass was also fairly reliable in predicting whether or not added S would have a positive impact on BA increment. Fascicle mass correctly predicted three of the four stands in which BA responses to $N + S$ were significantly greater than N alone. However, approximately one third of the installations in which fertilization with N alone significantly increase in first-year needle mass did not show a significant BA response to added N. Also, the fascicle mass differences between N and $N + S$ were, on average, considerably higher than the differential BA responses, thereby over-estimating the long-term positive effects of S additions on tree growth. In fact, significant BA responses to added S did not materialize in more than one half of the installations in which S significantly increased first-year fascicle mass over than achieved with N alone. In an earlier study, one quarter of stands with a significant fascicle mass response did not show significant 4-year BA responses to N fertilization (Weetman et al. 1988). Clearly, there is some risk of not recouping fertilization investment costs when the screening technique is used to select candidate sites for fertilizer operations or to make fertilizer prescriptions based on the statistical significance of fascicle mass response. The reliability of the method in predicting significant BA responses to N and to $N + S$ was improved when only those installations with large fascicle mass responses were considered. However, the likelihood of committing a type II error (i.e., failure to identify responsive

stands) is increased when installations with smaller fascicle mass responses are ignored.

Whereas first-year fascicle weight explained only one third of the variation in 6-year relative BA response to added N, foliar N and SO_4 each explained approximately one half of the variation. When combined, these two foliar variables were particularly useful for predicting whether or not lodgepole pine stands would respond significantly to fertilization with N alone.

Studies with Douglas-fir (*Pseudotsuga menziesii* (Mirb.) Franco) have reported strong negative relationships between stemwood responses to N fertilizer and pre-treatment levels of N in foliage. Turner et al. (1988) reported that the level of N in the foliage of unfertilized Douglas-fir accounted for 61% of the variation in 4-year BA response to N fertilizer. Hopmans and Chappell (1994) showed that 8-year relative BA response was strongly related to the level of N in the foliage of unfertilized Douglas-fir at 11 sites, accounting for as much as 94% of the variation among sites. In the present analysis, the weaker relationship between foliar N and stemwood response ($r^2 = 0.49$) may be partially attributable to N-induced S deficiencies and other non-nutritional factors, which may have a strong controlling influence on the response to N fertilization on many interior sites. Interestingly, these results are the same as those reported for Scots pine (*Pinus sylvestris* L.), in which foliar N accounted for 49% of the variation in 5-year stem volume response across 28 sites (Sikstrom et al. 1998).

Studies with radiata pine (*Pinus radiata* D. Don) and Douglas-fir have used pre-fertilization foliar SO_4 to predict whether or not stands will respond to N additions (Turner et al. 1977, 1979). The usefulness of foliar SO_4 in predicting fertilization response is based on the constant ratio between total N and organic S in the foliage of conifers (Kelly and Lambert 1972). Any S in excess of that required to balance foliar N in protein formation accumulates in the foliage as inorganic SO_4 -S. Therefore, in the absence of other nutritional or non-nutritional constraints, stands with inadequate foliar N and large reserves of foliar SO_4 (indicating S sufficiency) will likely respond well to N fertilization. Conversely, stands with low pre-fertilization SO_4 reserves (indicating S deficiency) will likely not respond to N additions even if foliar N levels indicate N deficiency. By modifying deficiency and sufficiency SO_4 levels developed for radiata pine, Turner et al. (1979) used pre-fertilization foliar SO_4 to identify Douglas-fir stands that were responsive and unresponsive to N fertilization. The correct prediction was made in 17 of 19 stands. However, in a recent fertilization study in British Columbia, Carter et al. (1998) reported that SO_4 was not useful in separating Douglas-fir responding and non-responding stands.

Based on the results of this study, pre-fertilization foliar N and SO_4 levels and N/S ratios may also have value in predicting whether or not lodgepole pine will respond to added S. Lodgepole pine stands in which pre-fertilization foliar SO_4 was ≤ 60 mg/kg and N/S ratio was ≥ 13 did not respond significantly to N alone but always responded significantly to $N + S$. On the other hand, a foliar SO_4 level > 60 mg/kg combined with a N/S ratio < 12 never resulted in a significant incremental S response. These results, combined with the fact that the screening method is more expensive and requires at

least 1 year's lead time prior to operational fertilization, indicate that foliar nutrient assessment using pre-fertilization foliar levels of N and SO₄ and N/S ratios may have greater utility than first-year increases in fascicle mass for assessing fertilization response potential and for making appropriate fertilization prescriptions. However, additional fertilization response data are needed to fully assess the utility of these foliar variables in developing reliable predictive tools. Response data are especially needed from lodgepole pine stands with low pre-fertilization levels of foliar N (<11 g/kg) and low to intermediate levels of SO₄ (<80 mg/kg) and from stands with intermediate to high foliar N (>12 g/kg) and high foliar SO₄ (>80 mg/kg).

The data presented here are from a variety of fertilization research experiments that were established over a number of years in the interior of British Columbia. As such, the reader is cautioned to consider the possibility that the results may be confounded by differences in such things as experimental approach (single-tree vs. area-based), stand origin (natural vs. plantation), post-thinning density, year or season of fertilization, and fertilizer source among the various experiments. On the other hand, the fact that relatively strong relationships were developed from such a diverse group of experiments may lend some strength to their validity.

In this study, foliage samples were submitted to a single commercial laboratory over a number of years. The methods used by the laboratory for the extraction and detection of foliar N, S, and SO₄ have not changed over time. Replicate samples of standard reference materials certified by the National Bureau of Standards, as well as in-house reference materials, were routinely submitted along with lodgepole pine foliage samples to document the accuracy and precision of analytical results. Within- and among-year accuracy and precision of N analyses were consistently high. Although certified standards for foliar S and SO₄ were unavailable, the within- and among-year precision of both analyses was also very good.

Results from the inter-laboratory comparison indicate that choice of commercial laboratory would likely not affect the utility of foliar N as a predictor of lodgepole pine fertilization response potential or interpretations with respect to foliar N status. However, based on the results from our inter-laboratory comparison, differences in analytical methodology for determining total S and SO₄ in conifer foliage may be problematic in terms of prediction and interpretation.

The lower values for total S reported by laboratory Y in our inter-laboratory comparison can likely be attributed to the different analytical methodologies used by the two laboratories. Recovery of S from wet combustion methods with nitric and perchloric acids (used by laboratory Y) has often been a problem because of gaseous losses of S or incomplete oxidation (Randall and Spencer 1980). However, S recovery with the Leco method (used by laboratory X) has been reported to be high (Guthrie and Lowe 1984). Based on results reported by Richter and Johnson (1983), it is unlikely that the slightly different HCl extraction methods used by the two laboratories for SO₄ analysis would significantly affect the amount of S contained in the extracts. However, since the HCl extraction method is not specific to inorganic SO₄ and can recover S from inorganic and soluble organic forms of S (Johnson and Nishita 1952), the amount of S recovered

from extracts will vary depending on which S fractions are measured. Laboratory Y used an inductively coupled plasma (ICP) spectrophotometer, which measures organic and inorganic forms of S in extracts. In contrast, the HI-reduction – bismuth colorimetric method used by laboratory X is specific to inorganic S. This likely explains why the SO₄ levels reported by laboratory Y were generally much higher than those reported by laboratory X. In another recent inter-laboratory comparison, determination of SO₄ by ion chromatography (also specific to inorganic S) following HCl extraction gave similar results as the HI-reduction method. Additional work is needed to quantify and evaluate between-laboratory differences in methodology and results for foliar S and SO₄ analyses.

The ability to reliably select responsive stands and to make fertilizer prescriptions based on the predicted magnitude of growth responses to N and S additions would remove much of the uncertainty surrounding lodgepole pine fertilizer operations in the interior of British Columbia. Future and recently established lodgepole pine fertilization research installations will be used to test and refine predictive models using pre-fertilization foliar nutrient variables.

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