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SELECTED RELATIONSHIPS FOR TRACE METALS IN MAINE LOW ELEVATION SPRUCE-FIR FOREST FLOORS

by

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ABSTRACT

The O horizon (forest floor) of soils found in forested ecosystems ha a tremendous capacity to retain trace metals (e.g. Pb, Zn, Cu, Ni, Cd) derived from any source including atmospheric deposition. Results from the analysis of trace metals in the forest floor from three study areas northern Maine commercial spruce-fir stands showed significant, positive correlations were frequently evident between ash-free concentrations of individual pairs of trace metals. Only Pb concentrations at one location were found to vary significantly among soil drainage classes. Overall, ash-free trace metal concentrations were less than what has been found at sites receiving high loadings of atmospheric deposition in the northeast, such as in southern New England or at high elevations. Little historical data are available to adequately determine if Maine forest floor trace metal concentrations are greater than the natural levels expected in commercial forests, but it appears possible that current concentrations reflect contributions from the atmosphere above background conditions. Relatively large numbers of samples would be required for future studies of forest floor trace metal concentrations to obtain good estimates of the metal concentrations for comparisons among sites and over time.

INTRODUCTION

There is increasing concern about the potential effects of long-range transported air pollutants on natural ecosystems of the Northeast. The center of attention in this issue has evolved from a focus on "acid rain" effects on lakes, to the possibility of a number of air pollutants interacting to affect the health of forests. Much of the concern has centered around spruce-fir forest types with several locations in the eastern United States exhibiting an anomolous deterioration of red spruce (<u>Picea rubens</u> Sarg.) (Johnson and Siccama, 1983).

One component of air pollution which should be considered in assessing effects on forests is the deposition of trace metals. Numerous studies have reported levels of trace metals in New England forest floors that are high enough to suggest that several metals (e.g. Pb, Cu, Zn) are accumulating in these organic soil layers as a result of deposition from the atmosphere (Andresen <u>et al</u>., 1980; Friedland <u>et al</u>., 1984a; Friedland <u>et al</u>., 1984b; Johnson <u>et al</u>., 1982; Siccama <u>et al</u>., 1980; Reiners <u>et al</u>., 1975). Hanson <u>et al</u>. (1982) found a gradient of decreasing Pb and Zn forest floor concentrations in high elevation sites from Vermont through Maine to the Gaspe' Peninsula in Canada. These studies suggest that while the magnitude of forest floor trace metal accumulations in Maine may be less than those noted in southern New England, the possible role of trace metals in the future productivity of Maine forests should not be ignored.

Very little research has been done on trace metals in low elevation, commercial forests and almost no work to date has been done on Maine's extensive low elevation, commercial spruce and fir dominated forestlands. In this paper we report on trace metal levels in the forest floor at three low elevation spruce and fir sites in northern Maine, and some of the relationships between these trace metals and selected properties of the site. This paper expands on a publication quantifying forest floor trace metal levels at these study sites by Fernandez and Czapowskyj (1985).

THE STUDY

The study was carried out at three sites in northern Maine as shown Figure 1. Sites were selected to represent low elevation (< 350 m) spru or spruce and fir dominated forests typical of the commercial spruce-fir resources in the state. Site descriptions are as follows: (a) The Square Lake Experimental Area is located in township T16-R5 WELS in northern Aroostook County, Maine. The site is under a cooperative agreement with the U.S. Forest Service as a long-term research site currently dominated by black spruce (Picea mariana (Mill.) B.S.P.) resulting from regeneration following clearcutting in 1957. Soils are derived from glacial lacustrine and glacial till parent materials. Twel 17×24 m sample plots had been established at this site and treated wit various combinations of drainage, fertilization, thinning, and slash removal. Twelve forest floor samples were collected in 1982 from each o four study plots. All samples were separated into 01 and 02 horizons based on an ability to recognize the origin of the organic matter. (b) The Big Reed Pond study site is equally divided by the border betwee townships T8-R10 and T8-R11 in Piscataguis County, Maine. There are no roads or records of cutting in this area with the exception of some ceda removals about 1924. The area is dominated by the spruce-fir type (Pice rubens Sarg. - Abies balsamea (L.) Mill.); however, approximately one third of the sample sites was from hardwood or hardwood-dominated mixed stands. Soils are derived primarily from ablation till and compact basa till. Sixty-seven forest floor samples, collected in 1981, were retaine for trace metal analyses. These samples were a subset from a total of 1 samples collected systematically throughout this 2064 ha site (Young, 1982).

(c) <u>The Weymouth Point</u> study site is located at Weymouth Point, township T4-R12 WELS, Piscataquis County, Maine along Chesuncook Lake. The site consists of a pair of adjacent watersheds with similar slope and aspect.

Forest floor samples used for trace metal analyses were collected in 1980 from the treatment watershed (48 ha) prior to harvesting. Samples were collected from twelve 20 x 20 m study plots with 01 and 02 horizons separated on the basis of ability to recognize the origin of the organic matter. The sampled watershed is predominantly characterized by the spruce-fir type growing on soils from the Chesuncook catena (coarse loamy soils developed from basal till). (Smith, 1984).

Forest Floor Analyses

Forest floor samples were air-dried prior to analysis. Subsamples were oven-dried at 105°C to permit the data to be expressed on an oven-dry weight basis. All visible rock fragments were removed from the forest floor samples prior to grinding in a Wiley Mill with a #40 mesh screen. Samples were ashed at 450°C overnight, digested in 10 ml of 1:1 (water/conc. acid) HNO3, boiled on a hot plate for 20 minutes, filtered through Whatman #42 filter paper, and brought to a final 50 ml volume with distilled-deionized water. Weight loss from the overnight ashing procedure was used to approximate the percentage of total organic matter in the forest floor. Forest floor digests were analyzed for Ca, Cu, Mg, Mn, P and Zn by inductively-coupled plasma emission spectroscopy (ICP); Al, Cd, Pb, and Ni by flame atomic absorption spectrophotometry (AAS) and K by flame emission spectrophotometry (AES). Forest floor pH was measured by electrode in distilled water at a 1:5 sample to solution ratio prior to arinding. Sulfate in the forest floor digest was analyzed by the methylthymol blue method on a Technicon Autoanalyzer.

Forest floor elemental concentrations are presented on an ash-free basis. We believe forest floor trace metal data should be expressed as either ash-free concentrations (best suited to evaluate the quality of the substrate for microbiological activity) or content (best suited to evaluate accumulation trends over time). Sample replicates and National Bureau of Standards orchard and pine tissue were utilized for quality



control. The data were statistically analyzed using SAS (Helwig, 1982) on the University of Maine at Orono's central computer facilities.

RESULTS AND DISCUSSION

Descriptive Statistics

Current ash-free trace metal concentrations and percent organic matter in the forest floor for the study sites are shown in Table 1. These data provide (a) a baseline for comparing future studies on trace metals in Maine forest floors to permit evaluations of possible trends over time, and (b) a perspective on current trace metal burdens relative to other regions. Compared to published trace metal studies in other regions, Maine forest floor trace metal concentrations appear lower than concentrations reported at high elevations in southern New England, or at sites affected by local point sources of trace metal emissions. Care should be exercised in comparisons with other published data, however, since whole-soil rather than ash-free concentrations are often reported. No historical trace metal concentration data are available for the region studied to adequately determine if significant trace metal accumulations have occurred in recent years.

Soil Drainage Relationships

During sampling of the forest floor at the Big Reed Pond study site, soil series were identified which permitted a comparison of both soil type and drainage to trace metal concentrations. Soil drainage is known to affect the accumulation of organic matter in the forest floor due to both soil moisture effects on tree growth and soil microbiological activity. Table 2 illustrates the trace metal concentrations at the Big Reed Pond study site partitioned according to soil drainage class and soil series. No statistically significant differences were found for trace metal concentrations among drainage classes with the exception of Pb. In the

Horizon	n	OM	Cd	Cu	Ni	Pb	Zn
		- % -			mg kg ⁻¹		
			<u>Squar</u>	<u>e Lake</u>			
01	47	91(5)a	0.70(32)a	5.7(56)b	6.7(35)b	51(29)a	45(22)a
02	47	76(15)b	0 . 59(42)b	7 . 4(38)a	11.5(39)a	37(33)b	40(25)b
			<u>Big R</u> e	ed Pond			
0(1+2)	67	80(11)	2.12(78)	8.0(52)	11.2(93)	77(41)	86(51)
			Weymout	<u>h Point</u>			
01	72	95(1)a	0.81(56)a	6.3(23)a	4.3(31)b	53(25)a	73(50)a
02	72	90(6)b	0.90(70)a	5.8(42)a	5.9(41)a	57(45)a	63(54)a

Table 1 - Ash-free trace metal and organic matter concentrations in three low elevation, commercial spruce-fir sites in Maine. $^{\rm l}$

1 Numbers in brackets are coefficients of variation. Means followed by the same letter are not significantly different between 01 and 02 horizons at that site for the 0.05 level of confidence.

Table 2 -	 The relationship of the Big Reed 	between soil Pond Study Si	drainage a te. ¹	and ash-free	trace metal	concentrations	in the O	horizon

Drafnage Class	Sof1 Sertes	n	МО	Cd	Cu	Ni	Pb	Zn
			- % -			_mg kg ⁻¹		
very poorly	Imlos, (Bag)	б	81a	2.8a	8.2a	14.6a	54b	59a
poorly	Monarda	6	79a	2.2a	6.5a	11 . la	103a	80a
somewhat poorly	Telos	11	77a	2.9a	9.8a	15 . 1a	63b	96a
moderately well	Chesuncook	11	82a	1.8a	7 . la	8.3a	81ab	.77a
well	Elliotsville, Beckett	13	78a	1.8a	7.6a	8.0a	82ab	90a
somewhat excessively	Monson	18	82a	1 . 9a	8.4a	12.2a	78ab	95a
excessively	Misery	2	85 a	2.3a	6 . 3a	8.8a	69ab	71a

I Values followed by the same letters are not significantly different at the 0.05 level of confidence using Duncan's Multiple Range Test.

case of Pb, there were significant differences among drainage classes, but no consistent trend was evident. Further research using a larger number of samples should be carried out to better define possible trace metal concentration trends associated with soil drainage class.

Relationships Among Trace Metals

Tables 3 and 4 show correlations between individual trace metals and organic matter content at the Big Reed Pond and Weymouth Point Watershed study sites. Overall, there was a highly significant, positive correlation among trace metal concentrations. Significant negative correlations with organic matter content could reflect a dilution of trace metals with increasing forest floor biomass, if trace metals are primarily associated with organic materials. An alternate explanation may be that in the digests of forest floor samples, trace metals are derived primarily from mineral soil particles, and digest with lower trace metal concentrations simply reflecting less contamination of forest floor materials by mineral soil particles. The net result is a negative correlation between trace metal concentrations and percentage of organic matter in the forest floor.

lable 3 - Simple correlation coefficients (r) for a	ash-tree
trace metal concentrations and organic ma	atter in
the forest floor at the Big Reed Pond st	udy site.

	Cd	Cu	Ni	Pb	Zn
OM	-0.76***	-0 58***	0 67***	-0 64***	0.25**
Cd	-0.70	+0.85***	-0.07 +0.86 ^{***}	-0.04 +0.54 ^{***}	-0.35 +0.43 ^{***}
Cu			+0.71***	+0.46***	+0.37**
Ni				+0.50***	+0.30**
Pb					+0.34**

*,**,*** Significant at the 0.05, 0.01, and 0.001 levels of confidence, respectively.

<u>.</u>	Cd	Cu	Ni	РЪ	Zn
			<u>Ol Horizon</u>		
MO	-0.23*	-0.62***	-0.36**	-0.10	-0.33**
Cd		+0.52***	+0.20	+0.13	+0.85***
Cu			+0.29**	+0.18	+0.57***
N 1				+0.17	+0.65***
РЬ					+0.20
		۵	2 Horizon		
ОМ	-0.34**	-0.27*	-0.40***	-0.46***	-0.40***
Cd		+0.44***	+0.31**	+0.52***	+0.90***
Cu			+0.39***	+0.34**	+0.44***
N1				+0.48***	+0.30**
РЬ					+0.62***

Table 4 - Simple correlation coefficients (r) for ash-free trace metal concentrations and organic matter in the forest floor at the Weymouth Point Watershed study site.

*,**,*** Significant at the 0.05, 0.01, and 0.001 levels of confidence, respectively.

The Weymouth Point Watershed data suggest that the strong positive correlation among trace metals in the forest floor results largely from significant correlations between concentrations in the 02 horizon, since Cd and especially Pb showed no significant correlations with some of the other metals in the 01 horizon. This likely reflects the strong retention of Cd and Pb by organic materials which comprise most of the forest floor volume. Table 6 indicates that significant positive correlations between trace metals should not be expected for 02 horizons at all sites, since

the Square Lake Management Area data showed non-significant relationships between at least one pair of trace metals for the metals studied. The general trend for all sites relative to relationships among trace metals suggests that no negative correlations among the concentrations of the trace metals studied are to be expected in forest floor materials.

Relationships With Other Elements

A more intensive investigation of trace metals in the 02 horizon samples from the Square Lake Management Area was carried out to explore trace metal relationships with other elements in the forest floor. Table 5 presents 0 horizon trace metal data as whole-soil concentrations, ash-free concentrations, and content. Content was calculated using bulk density data for this site from Czapowskyj (unpublished data).

Table 5 - A comparison of forest floor trace metal concentrations, ash-free concentrations, and contents at the Square Lake Management Area study site.

Horizon	Cd	Cu	Ni	Pb	Zn					
-1 Concentration (mg kg)										
01	0.64	5.2	6.1	46.3	41.1					
02	0.45	5.6	8.5	27.8	30.5					
Ash-free concentration (mg kg ⁻¹)										
01	0.70	5.7	6.7	51	45					
02	0.59	7.4	11.5	37	40					
	-		_2.							
	<u>Cc</u>	ontent (mo	<u>, m⁻ 4)</u>							
01	2.65	21.7	26.6	201	178					
02	2 30	20 0	45 2	142	157					
02	2.30	29.0	43•Z	142	121					

Additional elements were measured in the O2 horizon at the Square Lake Management Area (Table 6) and a correlation matrix for ash-free concentrations of all elements measured is shown in Table 7. Table 7 shows Mg was significantly and positively correlated with all the trace metals. Calcium showed a significant positive correlation with Zn only, whereas K showed a significant positive correlation with all the trace metals except Zn. This might be expected since the concentration of Ca and K in these types of horizons is known to be negatively correlated (Fernandez and Struchtemeyer, 1985).

Table 6 - Ash-free concentrations of selected elements in the 02 horizon at the Square Lake Management Area site (n=47).

	Ca	к	Mg	Mn	A1	Fe	\$0 ₄	-
Mean (mg kg ⁻¹)	1083	1444	549	37	8333	6443	4826	-
C.V. ¹ (%)	58	51	55	55	34	59	29	

¹ Coefficients of variation.

Aluminum was not significantly correlated with any of the trace metals, however Fe showed a significant positive correlation with Ni, Pb and Zn. If assume that Al is less likely to be deposited in significant amounts from the atmosphere, the lack of correlations with Al could reflect differences in the sources and mechanisms of retention in the soil for these metals. The lack o wet and dry deposition information for these metals in remote forested region in Maine prohibits conclusions which relate forest floor concentrations to atmospheric loading of each of the metals measured. If Al and trace metal mobilities were affected by pH, a strong correlation with pH would be expected. Indeed, many of the elements showed significant positive

	Cu	Ni	РЬ	Zn	Ca	Mg	К	P	A1	Fe	S04	рН	ОМ
Cd	+0.27	+0.30*	+0.31*	+0.39**	+0.10	+0.17	+0.04	-0.16	-0.20	-0.08	-0.13	+0.11	-0.03
Cu		+0.57***	+0.31*	-0.11	-0.16	+0.20	+0.42**	+0.20	+0.20	+0.45**	+0.49***	+0.40**	-0.21
١i			+0.17	-0.01	-0.04	+0.53***	+0.46**	+0.11	+0.20	+0.30*	+0.39**	+0.43**	-0.44**
РЪ				+0.21	-0.09	+0.15	+0.17	+0.19	+0.08	+0.32*	+0.28*	-0.11	-0.18
Zn					+0.39**	+0.26	-0.09	-0.21	-0.27	-0.15	-0.23	-0.17	+0.11
a						-0.09	-0.24	-0.07	-0.07	-0.09	-0.38**	+0.01	+0.17
lg							+0.76***	+0.05	+0.15	+0.50***	+0.57***	+0.50***	+0.50***
(+0.25	+0.35*	+0.71***	+0.79***	+0.56***	-0.84***
									+0.93***	+0.63***	+0.42**	+0.31**	-0.19
1										+0.60***	+0.41**	+0.42**	-0.30*
e											+0.67***	+0.50***	-0.64***
50,												+0.54***	-0.64**
4													-0.47***

correlations with pH. For Cu, Ni, Pb, Al and Fe the positive pH relationship may result from a lesser tendency for the leaching loss of these metals at higher pH values.

If the deposition of trace metals is closely associated with acid precipitation, we also might expect forest floor SO₄ to be related to trace metal concentrations. Significant positive correlations were found between SO₄ and Cu, Ni, and Pb. Sulfate was also significantly and positively correlated with Fe, Al, Mg, K and P while the only significant negative correlation was found to be with Ca. Hanson et al. (1982), in their study of forest floor composition along a precipitation pH gradient across New England, concluded Ca and Mn seemed to be progressively leached from soils with decreasing precipitation pH. They also concluded K and Mg probably show a similar leaching effect only when precipitation is at or below an average pH 4.0. Since precipitation pH averages in northern Maine are closer to 4.5, it is reasonable to expect the correlations found among SO4, Ca, Mg, and K in accordance with Hanson et al.'s hypothesis. It should be noted that numerous environmental factors change along a north-south transect across New England and further research which better evaluates ecosystem complexity along with a more geographically widespread forest floor sample population are required to better support this leaching hypothesis.

Several authors have suggested that trace metal accumulation in the forest floor, particularly of Pb, may inhibit the rate of organic matter decomposition (Andresen et al., 1980; Ruhling and Tyler, 1973; Siccama et al. 1980). We found no evidence in correlations between trace metals and organic matter to suggest this is occurring in Maine forest floors. However, if trace metals reduced decomposition rates, then organic matter itself may be accumulating in the forest floor, resulting in the dilution of metal concentrations. Correlation coefficients for 02 horizon data at the Square Lake Management Area between organic matter percentage and the content (mg m⁻²) of Cd, Cu, Pb, Ni, and Zn were +0.20, -0.16,

+0.28***, -0.38***, and +0.20, respectively. We could speculate that the highly significant, positive correlation for Pb reflects a Pb inhibition of organic matter decomposition along with a dilution of Zn. Again, further investigations of these phenomena are necessary and drawing any conclusion at this time is clearly premature.

A Perspective on the Data For Lead

This investigation was primarily intended as a descriptive evaluation of current trace metal levels in low elevation, commercial spruce dominated forested ecosystems in Maine. It is useful in providing a perspective on the Maine data to compare concentrations found in this study to other study sites reported in the literature. Figure 2 shows a comparison of forest floor Pb concentrations at several locations. The low elevation forest floor mean for Maine taken from this study is shown here as whole-soil concentration to permit a comparison with other forest floor Pb data available in the literature. From Figure 2, it appears that current Pb concentrations in the forest floor at low elevation sites in northern Maine are greater than Pb concentrations reported for areas in northern Norway (Allen and Steinnes, 1980), presumably not affected by significant atmospheric inputs of Pb, and greater than the concentration reported for a buried O horizon in New Hampshire (Johnson et al., 1982) which could be taken to represent pre-settlement forest floor Pb concentrations. Lead concentrations from southern Norway (Allen and Steinnes, 1980), in areas which receive significant atmospheric Pb inputs, and the high elevation mountain site from western Maine (Hanson et al., 1982), both are greater than the low elevation average from this study. These comparisons suggest that current Pb concentrations in Maine low elevation forest floors may include accumulations due to modern atmospheric Pb deposition, but are not affected to the extent apparent for sites receiving high loading rates of Pb from the atmosphere. If trace metal accumulations in the forest floor play a role in potential

atmospheric deposition effects on forested ecosystems, it is critical that the mechanism of effect be defined and that dose-response relationships for the individual metals and pollutant interactions be established.



To date, evidence of modern forest decline phenomena has been primarily reported at high elevation sites in the Northeast. No causal mechanism for this decline has yet been defined, but atmospheric deposition of N, S, and trace metals are considered to be possible contributing factors due to the significantly greater rates of deposition of atmospheric substances which occur in high elevation forests. If trace metals are shown to play a role in the health of forests at high

elevation sites, it would be useful to estimate the time required for low elevation forest floor trace metal accumulations to reach levels similar to those presently reported for areas subject to greater trace metal deposition rates. Table 8 illustrates a simple approach to estimate this using Pb content at the Square Lake Management Area and data from the literature. The time periods calculated suggest that decades to centuries would be required before northern Maine forest floor Pb content could be expected to reach contents evident today at high elevation sites or in southern New England. Clearly the mechanism of any possible effect of trace metals on forest productivity needs to be defined to evaluate the importance of these estimated time periods. We also would emphasize these are only estimates based on some key assumptions; (a) that 10 mg m⁻²yr⁻¹ represent the sum of wet and dry Pb deposition in Maine forests, and (b) that 90 percent retention adequately approximates both annual Pb retention and Pb residence in the forest floor over time. Current estimates of Pb residence times in northeastern forest floors range from hundreds to thousands of years.

Trace Metal Variability

Research on soils in forested ecosystems is often hampered by the high level of heterogeneity found in soil properties. It is useful to have a quantitative understanding of the variability of soil properties to better design research and evaluate data. Table 8 summarizes the sample sizes which would be required, based on our data, to adequately quantify the ash-free concentrations of Cd, Cu, Ni, Pb, and Zn at the 0.05 confidence level within 10 percent of the mean. On the other hand we can say for example that given our sample sizes for this study at Big Reed Pond, our estimates of mean ash-free concentrations at the 0.05 confidence level were within $\pm 10\%$ for Pb, but only within $\pm 19\%$ for Cd and $\pm 23\%$ for Ni. Clearly the intended use of data in soils research needs to be carefully considered in determining the appropriate experimental design and sample population sizes.

Table 8 -	Estimated years required for forest floor Pb content at the
	Square Lake Management Area to reach contents comparable to other site
	reported in the literature.

Site Description	Elevation	Forest Floor Content	Years ¹	Reference	
	- meters -	- mg m ⁻² -			
Square Lake Management	183	343	-	(this study)	
Mt. Moosilauke, NH (spruce-fir zone)	1036	510	19	Reiners et al., 1975	
Central Massachusetts (white pine stands)	varied	1161	91	Siccama et al., 1980	
Camel's Hump, Vermont				Friedland et al., 1984	
a - Northern hardwood zone	below 730	843	56		
b - Transitional zone	730 - 850	1280	104		
c - Boreal forest zone	850 - 1200	2000	184		

Assumes 90 percent retention in the forest floor of a total Pb $10 \text{ mg m}^{-2}\text{yr}^{-1}$.

<u></u>											
Horizon	n	ОМ	Cd	Cu	Ni	Pb	Zn				
All Samples	305	5	367	83	256	76	122				
				Squa	are Lake						
01	47	1	41	125	49	34	19				
02	47	9	71	58	61	4 4	25				
		Big Reed Pond									
0(1+2)	67	5	243	108	346	67	104				
				Weymou	ith Poin	<u>t</u>					
01	72	1	125	21	38	25	100				
02	72	1	196	71	67	81	117				

Table 8 - Sample sizes needed to determine ash-free trace metal concentrations \pm 10% at the 0.05 confidence level.

CONCLUSIONS

The current concentration and content of Cd, Cu, Ni, Pb, and Zn in the forest floor of low elevation, commercial forests in northern Maine suggests that these metals may be at levels in excess of what could be expected for background conditions. Atmospheric deposition would be the probable source of trace metal burdens in excess of natural concentrations. While above expected background levels, trace metal levels reported here are well below those reported in the literature for sites thought to be more heavily affected by atmospheric deposition. Further assessment of the significance of these data relative to forest

productivity in northern Maine will require the results of investigations to determine (a) the mechanisms whereby trace metals may affect tree growth individually, in combinations, and when interacting with other environmental stress factors, (b) the subsequent dose-response relationships, and (c) the nature of interactions between trace metals and other elements or microbial processes in the soil.

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