INORGANIC CONCENTRATIONS IN THE WOOD OF EASTERN REDCEDAR GROWN ON DIFFERENT SITES¹

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ABSTRACT

Samples of eastern redcedar (*Juniperus virginiana* L.) growing on soils derived from five parent materials—rhyolite, dolomite, limestone, sandstone, and chert—were analyzed for levels of inorganics in sapwood and heartwood. Eighteen elements were detected in sapwood using inductively coupled plasma optical emission spectroscopy. Neutron activation analysis was also used to determine concentrations of an additional six elements in heartwood. No difference was found between results obtained by the two analytical methods. Conventional wet chemistry techniques were used to determine nitrogen and sulfur concentrations in some samples.

Keywords: Eastern redcedar, inorganics, ICP, NAA, soil types.

INTRODUCTION

While, as a whole, levels of inorganic elements in wood and foliage are relatively small, there have been repeated efforts for many years to quantify these levels and relate them to specific sites and site deficiencies (Ellis 1965; Finn 1953; Hacskaylo et al. 1969). In more recent years, the levels of inorganics in foliage and wood have been used to ascertain the plant's response to atmospheric pollution and document changes in air-borne pollutants (Baes and Ragsdale 1981; Baes and McLauglin 1984; Bondietti et al. 1989; Guyette and McGinnes 1987; Guyette et al. 1989, 1991). Sophisticated instrumental analytical methods coupled with the use of dating techniques borrowed from the field of dendrochronology (Fritts 1976; Stokes and Smiley 1968) have permitted documentation and reconstruction of changes in soil chemistry as captured in the tree-rings of selected species.

Eastern redcedar (*Juniperus virginiana* L.) has several characteristics that make it and its wood desirable and usable, not only as a dendroclimatological indicator (Butler and Walsh 1988; Guyette et al. 1980; Weakly 1943), but also as a biogeochemical monitor. Redcedar can be found growing under a wide range of soil and climatic factors with correspondingly wide ranges in such traditional measures of growth such as dbh and height. For the purposes of research such as described in this pa-

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FIG. 1. Map of Missouri showing sample locations. In all cases more than one tree or remnant per location was obtained. The outlined area is the lead mining district of SE Missouri. R represents a rhyolite site; L, limestone; C, chert; D, dolomite; and S, sandstone.

per, this wide latitude in ecological amplitude is ideal. Additionally, redcedar can be a very long-lived species with documented life-spans in excess of 1,000 years (Guyette and Mc-Ginnes 1987). These very old trees grow on sites where relatively small changes in atmospheric deposition can have a major influence on the chemical composition of the soil.

The heartwood of eastern redcedar has a relatively low moisture content (McGinnes and Dingledein 1969), very low radial permeability (Choong and Fogg 1968; Stamm 1970), and is considered very durable. Its chemical composition has been shown to be stable over long time spans (McGinnes et al. 1983).

The research presented in this paper represents the results of elemental analyses of eastern redcedar samples grown on a wide variety of soils and parent bedrocks in the Missouri Ozarks. This study was part of a larger study utilizing redcedar growth increments to reconstruct long-term changes in soil chemistry (Guyette et al. 1989, 1991).

MATERIALS AND METHODS

Sites and soils

The overall objectives of the original study dictated that the trees that were sampled be located on sensitive sites across a wide geographic area (Guyette and McGinnes 1987; Guyette et al. 1989). The trees used in this study grew on sites that had shallow, residual soils over either sandstone, Burlington limestone, chert, rhyolite, or dolomite (Fig. 1). Rhyolite is composed of alkali feldspars, quartz, and matric pyroxenes and amphiboles that weather to form an acidic soil. Chert is a siliceous rock (usually an inclusion in other rock types) that also weathers to form an acidic soil. Sandstone is a sedimentary rock consisting of sand (usually quartz) held together by a cementing agent such as calcium or silica. Limestone is a sedimentary rock composed of calcite (calcium carbonate). Dolomite is a type of limestone consisting of double carbonates of calcium and magnesium. All sites were generally steep with very well-drained soils. Mean annual precipitation ranges from 92 to 112 cm.

TABLE 1. Soil pH and extractable element concentrations (ppm) with standard deviations for soils derived from five bedrock types.

pH Ca Mg	Limestone	Chert	Dolomite	Sandstone	Rhyolite 4.25 (0.5)		
pН	6.9 (0.84)	4.9 (0.8)	6.6 (0.3)	5.4 (0.6)			
Ca	4,616 (2,050)	754 (540)	3,871 (983)	1,873 (915)	593 (525)		
Mg	70 (21)	181 (207)	817 (382)	428 (406)	70 (35)		
Zn	5.3 (4.8)	5.8 (2.6)	5.1 (1.8)	4.8 (1.9)	5.1 (1.6)		
Fe	19.6 (19.4)	43.6 (17.1)	13.1 (3.3)	31.4 (18.2)	60.1 (29.4)		
Р	5 (3)	4 (1)	2 (1)	7 (17)	16 (9)		
Cu	9.1 (2.9)	8.6 (3.6)	17.6 (4.2)	30.1 (32.2)	0.9 (0.6)		
Mn	1.0 (0.6)	1.1 (0.4)	1.9 (1.4)	2.4 (3.3)	21.9 (14.8)		
K	98 (29)	62 (17)	188 (45)	105 (65)	117 (37)		

Element	Occurrence .	Detection limit	Mean	Range	Standard deviation	
AP	common on acid sites	2.0	3.32	<2-18	4.73	
As	none	3.0	nd ¹			
В	common on acid sites	2.0	10.3	<2-60	13.5	
Ba	all samples	0.03	10.2	0.2-31	8.77	
Bi	none	3.0	nd			
Ca	all samples	2.0	1,347	962-1,690	167	
Cd	uncommon, acid-smelter sites	0.04	0.11	< 0.04-0.23	0.06	
Со	none	0.3	nd			
Cr	none	0.9	nd			
Cu	all samples	0.2	1.14	0.6-1.8	0.26	
Fe	nearly all samples	0.9	2.27	<0.9-6.0	1.19	
K	all sapwood samples	30.0	478	280-950	135	
Li	nearly all samples	0.04	0.085	< 0.04-0.12	0.02	
Mg	all samples	0.9	103.0	51-188	27.6	
Mn	all samples	0.2	25.70	2.1-131	28.6	
Mo	none	0.9	nd			
Na	common	1.0	16	<1-46	13.2	
Ni	rarely on acid smelter sites	0.2	0.34	<0.2-0.5	0.098	
Р	all sapwood samples	10.0	71	<10-130	19.5	
Pb	common near smelters, roads	0.4	2.1	<0.4-9.6	2.25	
Sb	none	3.0	nd			
Se	none	3.0	nd			
Si	common	0.2	23.9	<0.2-93	28.9	
Sn	none	3.0	nd			
Sr	all samples	0.09	4.69	1.1-14.1	3.42	
Ti	none	0.09	nd			
Tl	none	5.0	nd			
V	none	0.3	nd			
W	none	1.0	nd			
Zn	all samples	0.2	2.0	0.8-4.0	0.68	

TABLE 2. ICP inorganic elements and average detection limits (ppm) in eastern redcedar sapwood from 67 trees.

nd = Not detected.

Basic physical and chemical data are given in Table 1 for all five sites.

Soil acidity was measured as pH with a glass Ag/AgCl combination electrode. Ten-gram samples of soil were mixed with 10 ml of 0.01 M CaCl₂. The solution was stirred every 10 minutes for 30 minutes, and the pH electrode was immersed in the soil solution. Salt pH measurements were used in this study because it is a better measure of potential acidity and is less affected by suspension effects in measurement. Independent soil pH measurements were taken by a soil testing laboratory in addition to those taken in the field. Soil pH for the 67 tree sites ranged from 3.1 to 7.9 and had a mean of 5.28.

Soil tests were run to determine exchangeable Ca, Mg, P, Cu, Mn, Zn, K, and Fe to provide a control for differences in the amount of an element available to the roots.² Soil was extracted with chelated DTPA for the elements Fe, Zn, Cu, and Mn (Lindsay and Norvell 1969). Extraction for Ca, Mg, and K was done with ammonium acetate. The Bray I phosphorous test was used.

While sample trees were taken from all five sites, the majority of the trees were sampled in two areas. This was because the primary emphasis of the original study was the influence of air-borne pollutants on soil chemistry. One area was within the lead mining district of Southeast Missouri, while another area was

² Soil tests were performed by the University of Missouri–Columbia's Regional Soil Testing Laboratory, Department of Agronomy, 27 Mumford Hall, Columbia, MO 65211.

Element	Half-life	Cross-section (barns)	Mean	Avg. detection limit
As-76	1.10 d	4.30	7.0 ppb	5 ppb
Au-198	2.69 d	98.80	0.6 ppb	0.005 ppb
Ba-131	11.8 d	13.50	19.7 ppm	0.012 ppm
Ca-47	4.53 d	0.25	910.0 ppm	4 ppm
Co-60	5.27 у	37.0	20.6 ppb	2 ppb
Fe-59	44.5 d	1.23	4.2 ppm	
Hg-203	44.6 d	4.90	4.7 ppm	2 ppb
K-42	12.36 h	1.30	121.0 ppm	35 ppm
Mo-99	2.78 d	0.51	44.7 ppb	30 ppb
Na-24	14.97 h	0.53	6.6 ppm	0.1 ppm
Rb-86	18.6 d	0.91	132.8 ppb	30 ppb
Sr-85	64.8 d	1.32	5.8 ppb	
Zn-65	243.8 d	0.82	830.5 ppb	3 ppb

TABLE 3. NAA parameters for and concentrations of heartwood inorganic elements monitored in this study.

located 80 km upwind (southwest) from the center of the mining district (Fig. 1). In both areas, trees were sampled that grew on two soil types: acidic soils over rhyolite, and more neutral soils over dolomite.

Sampling

Over 100 trees-both live and dead-were used in this series of studies. These trees ranged in age from <25 to >700 years old and dated from the present for the live trees back to 1118 AD for remnant samples. For the live trees, reference cores were first taken with a 4-mm increment borer to determine the place of extraction for the large core and for later use in growth analysis and dating. These trees were then sampled for chemical analyses by extracting a 17-mm-diameter core from the tree bole. Cross sections were taken from dead trees. For both the cores and the cross sections, annual growth increments were crossdated to insure the correct dating of the annual increments and assign dates to the remnant (dead) wood (Stokes and Smiley 1968).

Analytical measures

The overall objectives of the principal study in part dictated the choice of analytical measures. Since we were looking for particular inorganic elements in a very large number of samples, the techniques used were chosen both for cost and reproducibility. One of the drawbacks to the sampling procedures employed was that matched samples were not always available from every core or cross section for comparison of different analytical techniques. However, some matched samples were run to provide a limited comparison within a given site.

Selected wood samples were analyzed by inductively coupled plasma optical emission spectroscopy (ICP).³ Samples (0.5 to 1.0 g) were prepared for analysis using a nitric acid-perchloric acid digestion procedure. ICP analyses were conducted with a Jarrell-Ash Model 1100 Mark III with 40 analytical channels, controlled by a Digital Equipment Company 11/ 23+ computer. The instrument was standardized with a series of seven standards containing 36 elements. The detection limit shown in Table 2 was obtained by taking ten integrations of the "zero" standard; three times the standard deviation of the mean was used as the detection limit.

Additional samples were analyzed using neutron activation analysis (NAA). It has been used in several studies to determine inorganic levels in wood and charcoals (Cutter et al. 1980; Meyer and Langwig 1973; Osterhaus et al.

³ ICP analyses done at the University of Missouri's Environmental Trace Substances Laboratory, Route 3, Columbia, MO 65203.

Substrate	Al	В	Ba	Ca	Cu	Fe	К	Mg	Mn	Р	Sr	Zn
Rhyolite	6.8	19.1	19.3	1,352	1.1	1.9	470	98	50	73	8.3	2.3
(27)	(23	23	27	27	27	27	27	27	27	27	27	27)
Dolomite	0.6	3.1	1.6	1,296	1.5	1.8	490	108	6	78	1.7	1.9
(19)	(2	14	16	19	19	14	19	19	19	19	19	19)
Sandstone (7)	0.3	3.0	3.4	1,173	1.4	3.4	530	101	17	60	2.2	1.8
	(1	4	7	7	7	7	7	7	7	6	7	7)
Chert	0.6	3.0	7.1	1,495	1.0	3.1	489	126	12	61	3.1	1.9
(10)	(2	8	10	10	10	9	10	10	10	10	10	10)
Burlington limestone (4)	nd ²	2.3 (2	2.2 4	1,502 4	1.0 4	1.8 3	388 4	52 4	6 4	63 4	2.7 4	1.4 4)

TABLE 4. Mean sapwood inorganic concentrations (PPM) determined using ICP sorted by underlying substrate.

¹Numbers in parentheses are the number of samples for that soil type in which a given element was detected.

 2 nd = Not Detected.

1975: Slocum et al. 1978; Young and Guinn 1967). While certain of the inorganic elements detected in NAA were the same as in ICP, the detection limits of NAA (Table 3) were far lower than for ICP (Table 2). The wood to be irradiated was split from the core, skinned with a stainless steel scalpel blade to remove surface contaminants, and sealed in pure quartz vials. Samples were irradiated for 45 h in a thermal neutron flux of 5×10^{13} thermal neutrons cm⁻² sec⁻¹ at the University of Missouri's Research Reactor (MURR).⁴ In this study, activity counts were taken at 5 d. Activation parameters for elements analyzed using this technique are given in Table 3 (Pagden et al. 1971).

Sulfur concentrations in the heartwood were measured turbidimetrically as $BaSO_4$ (Wall et al. 1980). This procedure requires a minimum of 10 g of wood per sample. Nitrogen levels were done using the micro-Kjeldahl technique.⁵

It should be pointed out that all of the detection limits reported in this paper are actually average detection limits. This is because of numerous factors including sample size, sample moisture content, and in the case of NAA, interference from other elements and fluctuations in radiation intensity.

RESULTS AND DISCUSSION

The results of the sapwood ICP anlyses are shown in Table 4; those of the heartwood ICP analyses in Table 5; and those from the heartwood NAA analyses in Table 6. When dealing with these and subsequent data and analyses, one pertinent question becomes immediately obvious-are observations or samples in which no element was detected, i.e., the level was below the detection limit, treated as zeros when calculating statistics or are they treated as missing data? In this study we have chosen to treat these samples as zeros. This has the effect of reducing the mean (sometimes below the average detection level). Therefore, Table 4 and subsequent tables also indicate the number of samples in which the element was detected.

The other point that needs to be clearly made is that we know that the appearance of at least some of the elements detected in the heartwood is directly correlated with anthropogenic activities, i.e., mining, smelting, farming, etc. For example, levels of Cd, Pb, Mo, and S all changed dramatically following the beginning of the lead mining and smelting industry in Missouri. Lead and Cd were found only in trees growing on acid (pH < 4.6) soils over rhyolite substrate (Table 2). No lead was detected in trees growing both in and out of the

⁴ NAA performed by Michael D. Glascock, Senior Research Scientist at MURR.

⁵ Both the sulfur and the nitrogen analyses were done by the University of Missouri's Agricultural Experiment Station Chemistry Labs.

Substrate	Al	В	Ba	Ca	Cu	Fe	К	Mg	Mn	Р	Sr	Zn
Rhyolite	5.7	14.6	14.2	654	0.7	1.0	101	69	17	19	5.3	0.8
(21)	(19	16	21	21	21	20	21	21	21	21	21	21)
Dolomite	0.5	2.2	1.9	634	0.6	2.7	69	70	4	20	1.1	0.5
(13)	(3	9	12	13	12	9	12	13	13	11	13	11)
Sandstone (2)	1.5	3.5	12.8	537	1.0	1.6	59	50	8	28	5.7	0.7
	(1	1	2	2	2	2	2	2	2	2	2	2)
Chert (5)	0.6	3.2	6.4	819	0.5	1.3	96	77	8	22	2.9	0.3
	(1	4	5	5	4	3	4	5	5	5	5	4)

TABLE 5. Mean heartwood inorganic concentrations (PPM) determined using ICP sorted by underlying substrate.^{1,2}

¹ Numbers in parentheses are the number of samples for that soil type in which a given element was detected.

² There were no usable ICP heartwood samples on Burlington limestone.

lead mining district on more basic soils over dolomitic substrates. Thus, soil acidity is a very important factor influencing xylem Pb concentrations. Plots of Pb levels show that no lead was detected until the amount of lead smelted annually exceeded 100,000 Mt/yr. This occurred about 1900 (Guyette et al. 1991). Similarly, levels of Mo in redcedar rings declined starting in 1860. This was coincident with the start of coal mining and lead smelting in Missouri and also with the increase in S levels in the annual rings (Guyette et al. 1989).

As would be expected, inorganic levels were considerably higher in the sapwood (Table 4) than in the heartwood (Tables 5 and 6) for elements such as Ca, K, Mn, and P, which are actively involved in metabolic processes and are readily translocatable.

The levels of all the inorganics to a limited

extent reflect their underlying substrate. For example, relatively low amounts of Mg were found in the sapwood of the trees growing on Burlington limestone, itself low in magnesium relative to the competing calcium cation (Table 1).

Nitrogen levels in heartwood from nine trees growing on dolomite and limestone averaged 1,210 ppm, while sulfur levels in the same trees averaged 94 ppm (Table 7). In a previous paper, we demonstrated that sulfur levels in heartwood were less than 50 ppm prior to industrial development in the mid-1800s (Guyette et al. 1989).

One other question that always arises is: What concentrations should be used when different analytical techniques produce different values for trees/samples known to be from the same site. The ICP and NAA data presented

	Element												
Substrate	As	Au	Ca	K	Mo	Na	Ba	Co	Fe	Hg	Rb	Sr	Zn
	(ppb)	(ppb)	(ppm)	(ppm)	(ppb)	(ppm)	(ppm)	(ppb)	(ppm)	(ppb)	(ppb)	(ppm)	(ppb)
Rhyolite	1.3	0.83	1,026	131	19.8	8.2	21.3	24.9	4.2	4.1	96	7.4	973
(10)	(2	9	10		5	10	10	10	10	8	8	10	10)
Dolomite	2.3	0.36	851	110	33.6	6.1	30.1	9.8	4.3	4.7	18	1.3	602
(8)	(2	8	8	8	6	8	8	8	8	6	3	7	8)
Sandstone	1.9	0.72	815	134	41.7	6.8	13.7	32.1	4.9	1.5	194	9.2	980
(8)	(2	8	8	7	7	8	8	8	8	4	7	8	7)
Chert (1)	0	0.85	813	100	83.0	4.7	3.6	4.5	3.9	0	58	1.9	340
Limestone	1.5	0.05	996	23	4.5	0.7	2.4	3.9	1.4	2.2	34	1.4	253
(2)	(1	2	2	2	1	2	2	2	2	2	2	2	2)

TABLE 6. Mean heartwood inorganic concentrations determined using NAA sorted by underlying substrate.¹

¹Numbers in parentheses are the number of samples for that soil type in which a given element was detected.

 TABLE 7.
 Sulfur and nitrogen concentrations (ppm) in eastern redcedar heartwood growing on dolomite and limestone.

Ele- ment	n	Number trees	Range	Mean	Standard deviation
Ν	72	9	800-1,600	1,210	160
S	75	9	30-240	94	26

us with a similar dilemma in this study. The data presented in Table 8 show that there was no significant difference between the means of samples compared by the two analytical methods in levels of Ca, K, Sr, and Zn in matched samples. Correlations between the concentrations of those elements in matched samples determined by NAA and ICP were all significant at the 1% level.

Fletcher and Ochrymowych (1955) stated that there was a positive relationship between levels of Ca, Mg, and P in soil and levels in of these elements in the foliage, stems and roots of redcedar seedlings and mature trees. They suggested that soils with high amounts of exchangeable Ca in the soil promoted root and stem development. Cutter and Guyette (1990) found that there was a direct correlation between sap pH and soil pH for redcedar grown in Missouri. Since the pH of the sap solution is directly determined by the chemical makeup of the sap, it would not be surprising that the levels of inorganics in the wood would reflect this. Soils analyses were performed and these results (Table 1) tend to bear out this hypothesis.

SUMMARY

Samples of eastern redcedar (Juniperus virginiana L.) growing on soils derived from five parent materials—rhyolite, dolomite, limestone, sandstone, and chert—were analyzed for levels of inorganics in sapwood and heartwood. These samples included both living trees and dead remnant sections. Eighteen elements were detected in sapwood using inductively coupled plasma optical emission spectroscopy. These included Al, B, Ba, Ca, Cd, Cu, Fe, K, Li, Mg, Mn, Na, Ni, P, Pb, Si, Sr, and Zn.

TABLE 8. Means and standard deviations of ICP and NAA matched samples.

	NAA	ICP	n	
Са	722 ± 212	747 ± 229	36	
К	$340~\pm~224$	$359~\pm~235$	35	
Sr	1.3 ± 0.3	1.5 ± 0.3	36	
Zn	0.5 ± 0.2	0.4 ± 0.2	9	

Neutron activation analysis was also used to determine levels Ca, K, Sr, and Zn as well as levels of six additional elements in heartwood—As, Au, Co, Hg, Mo, and Rb. No difference was found between results obtained by the two analytical methods for Ca, K, Sr, and Zn levels in matched samples. Levels of nitrogen and sulfur were measured in some samples using traditional wet chemistry methods. Soil type and pH as well as anthropogenic activities were shown to influence the presence and amount of several elements detected in heartwood.

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