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# Dendrochemical evidence for soil recovery from acidic deposition in forests of the northeastern U.S. with comparisons to the southeastern U.S. and Russia

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#### HIGHLIGHTS

• Dendrochemical patterns of Ca, Mg, and K were identified in US and Russian spruce.

• Patterns varied with wood transformations and soil chemistry.

• Patterns were consistent with decreased acid deposition and progressive recovery.

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#### ABSTRACT

A soil resampling approach has detected an early stage of recovery in the cation chemistry of spruce forest soil due to reductions in acid deposition. That approach is limited by the lack of soil data and archived soil samples prior to major increases in acid deposition during the latter half of the 20th century. An alternative approach is the dendrochemical analysis of dated wood to detect temporal changes in base cations back into the 19th century. To infer environmental change from dendrochemical patterns of essential base cations, internal factors that affect cation chemistry such as the maturation of sapwood and the spread of wood infection need to be recognized. Potassium concentration was a useful marker of these internal maturation and infection that could affect the concentration of essential base cations in wood. Dendrochemical patterns in samples of red spruce in the eastern United States and Norway spruce in northwestern Russia were used to determine how internal changes in base cations can be separated from external changes in root-zone soil to date major changes in the availability of essential base cations associated with a changing environment.

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#### 1. Introduction

Due to naturally high acidity and low base saturation of forest soil, red spruce (*Picea rubens* Sarg.) in the eastern United States and Norway spruce (*P. abies* L. (Karst.)) in northern Europe were particularly vulnerable to adverse effects of acidic deposition derived from industrial emissions (Tomlinson and Tomlinson, 1990). Acidic deposition tended to mobilize essential base cations, deplete the rooting zone, and increase stress for some tree species (Shortle and Smith, 2015).

The rate of emission increase of S and N oxides peaked in the

1960s, gradually decreased during the 1970s and 1980s as the 1970 Clean Air Act was implemented (NAPAP, 1993), and continued to trend downward in the US as the 1990 Amendment to the Clean Air Act was implemented (NAPAP, 2005). The chemical comparison of forest soils in the northeastern US and eastern Canada sampled in the 1980s-1990s and resampled in 2003–2014 indicated an early stage of reversal of the adverse effects if acidic deposition (Lawrence et al., 2015).

The resampling approach to detect changes in the root environment prior to the rapid increase of acidic deposition in the 1960s-1970s is limited by the lack of soil data and archived soil samples. However, dendrochemical analysis of dated wood has the potential to detect major changes in base cations well back to the early 19th century. Dendrochemical patterns integrate cation availability with internal processes of maturation and infection.





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Base cation movement follows a modified Donnan model of cation exchange along the flowpath from soil storage, root uptake, and stem translocation (Momoshima and Bondietti, 1990; Bondietti et al., 1990; Shortle and Bondietti, 1992; Shortle et al., 1997). The purely biophysical Donnan model is modified through active absorption by living cells, resorption during heartwood formation, and disruption by the internal spread of wound-initiated diseases of wood (Smith and Shortle, 1996; Watmough, 1997; Meerts, 2002; Smith, 2008; Smith and Shortle, 2001; Smith et al., 2009, 2014).

The woody stems of mature healthy spruce contains an outer band of living sapwood that surrounds a core of non-living heartwood. The width of the band of sapwood in terms of radial distance and number of rings varies in part due to ring width. The greater moisture content of sapwood compared to heartwood in freshly collected samples is visible as a dark water-soaked appearance in reflected light (Fig. 1A) and translucence in transmitted light. The transformation of sapwood into heartwood is accompanied by a reduction of wood moisture and K content, the latter due to the withdrawal of the symplast and K-rich parenchymal cytoplasm. Unlike many other conifer species, spruce heartwood formation is not accompanied by the deposition of colored extractive products, making dried sapwood and heartwood similar in appearance.

Extensive research on the electrical characteristics of wood in living trees determined that changes in electrical resistance relative to the outer living sapwood can be used to detect the spread of wood decay in spruce and fir trees (Shortle and Smith, 1987). This ionization occurs rapidly and long before any symptoms of decay are visible (Shortle, 1990) and is primarily related to increased concentrations of K ions (Shortle, 1982; Shortle and Ostrofsky, 1983), with concentrations of Mg and Ca ions increased to a lesser degree. Therefore, the concentration of K in dated wood can be used to detect the transformation of sapwood to heartwood in spruce and to detect the internal spread of wood decay that alters the mineral content of wood during the previsual stage of the decay process and disrupts the radial patterns of base cations used to infer external changes in root-zone soil. The alkaline earth elements Ca and Mg share series 2A of the periodic table and both occur as fixed divalent base cations in trees and forest soil. Translocation of Ca is largely apoplastic, following the bulk flow of xylem sap with ion displacement at exchange sites on the xylem cell walls (Bondietti et al., 1990; Smith and Shortle, 2001).

Comparisons across two distinct regions of the eastern US and a third region in northwestern Russia has the potential to increase understanding of internal and external processes that contribute to dendrochemical patterns. The major objectives of this study were to: (1) determine how differences in the periodic growth rate can affect the dating of a major change in root-available Ca and how it differs from Mg; (2) compare the dendrochemical records of red spruce in the northeastern U.S to those in the southern Appalachians, and to Norway spruce at locations with archived soil data in northwestern Russia; and (3) determine how the dendrochemical record changed from the 1980s to the 1990s as acid deposition decreased and the availability of Ca increased from wood decay following red spruce mortality observed in the late 1970s, 1980s, and early 1990s.

#### 2. Materials and methods

#### 2.1. Sample collection and analysis

Decadal dendrochemical trends for the dominant base cations were determined for canopy-dominant or codominant spruce in the northeastern and southeastern US and in northwestern Russia (Fig. 2). Sampled trees were >30 cm in diameter at breast height. Increment cores (12-mm diameter) were extracted at breast height and the visible boundary between sapwood and heartwood marked in four collections. The northeastern red spruce collection was taken from 11 field locations across northern New York, Vermont, New Hampshire, and Maine in 1992–1993 in conjunction with forest soil analysis (David and Lawrence, 1996; Shortle et al., 1997) and from two additional locations in Waterville Valley, NH, in 1994 (Shortle and Bondietti, 1992). In brief, all northeastern and southeastern US sites were dominated by red spruce. Average tree age ranged from 96 to 175 years. Elevation ranged from 80 to 975 m and all soils were classified as spodosols.

The southeastern red spruce collection was taken at the Great Smoky Mountains National Park in Tennessee and North Carolina in 1995 in the vicinity of previous sampling and chemical characterization of spruce fine-roots (Smith et al., 1995). The northwestern Russian collection of Norway spruce was taken in 2001–2002 from a southern boreal forest of mixed Norway spruce and Scots pine (Pinus sylvestris). The Russian terrain was flat and low-lying, east of the Bay of Finland. Soil chemistry was determined at this location and is included in this report. At the two Russian sites (Luga and Lisino), exchangeable Ca and Al were determined for forest soil at depths 0–10 cm and 10–20 cm below the top of the mineral soil. In addition, intact soil monoliths collected in 1926 and 1964 were available to provide soil samples from the same layers as in the 2001-2002 sampling. The archived and newly collected soil samples collected were analyzed together to evaluate changes in soil chemistry over time. Soil methods are presented in Lawrence et al. (2005).

The northeastern red spruce re-sample collection was taken in 2003–2004 from six locations previously sampled for the northeastern red spruce collection, as described above and in Lawrence et al. (2012). The resample collection was made to test the persistence of any dendrochemical trend identified in the 1992–1993 collection and to detect changes that may have occurred due to decay of deadwood that produced Ca-rich organic matter during the decade between samples (Shortle et al., 2012).

For all collections, increment cores were mounted in grooved wood blocks, sanded, and cross-dated. Decadal boundaries of cores free of visible internal defects or indication of wood infection were marked. The radial distance of each decadal band was measured to the outer limit of the sapwood.

The dated wood samples were drilled from each mounted core using a Ti-coated drill bit 0.64 cm in diameter. Cores were drilled towards the center of each decadal band and the resulting shavings were taken as the sample for that decade. Because of variation in ring width, the number of rings contributing shavings varied among decadal samples. For each decade, 25 mg of drill shavings was placed in a 15-mL acid washed glass test tube to which 6 mL of 10 mM HCl was added. Cations were extracted from the shavings in three freeze-thaw cycles (Minocha and Shortle, 1993; Shortle et al., 1997). The extract was filtered with a 45-µm syringe filter and the concentration of the major inorganic cations, Ca, K, Mg, was determined by direct-coupled plasma atomic emission spectroscopy (DCP-AES) from 1991 to 1996, and thereafter by inductively coupled plasma atomic emission spectroscopy (ICP-AES).

#### 2.2. Sample screening

Increment cores lacking clean decadal bands due to wood defects, visible infection, or elevated K concentration (concentration > outermost sapwood band) were screened out of further analysis. The northeastern red spruce cores collection from 123 trees yielded cores with variable numbers of decadal bands (n = 14 in 1840s to n = 104 in 1900s, and n = 123 from 1910s to 1980s). To compare the effect of sapwood width on the dendrochemical record of decadal bands, the northeastern red spruce collection was subdivided for some

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