DENDROCHEMICAL RESPONSE TO SOIL FERTILIZATION

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ABSTRACT.—Use of chemical element content of tree rings to detect soil acid or base changes was tested at 13 sites of former forest fertilization trials in the eastern United States and Canada. Ammonium sulfate or nitrogen fertilization was the typical acidification treatment, while lime added with or without other fertilizer was the typical base treatment. Molar ratios of calcium/manganese and magnesium/manganese in bolewood were used to detect changes in tree ring tissue from 10 hardwood and 3 conifer species available at these sites. Base fertilization at all sites caused molar ratios to increase in all species tested and acid treatment caused ratios in tree rings to decrease. Diffuse-porous hardwood species and conifers with wide sapwood regions were not useful in preserving a chemical record of soil changes caused by fertilization. Approximate chronologies of soil chemical change due to fertilization were approximately preserved in tree cores from Japanese larch and in ring-porous hardwoods sampled up to 30 years after fertilization. Greater use of dendrochemical sampling is recommended to assess fertilizer and soil acidification effects on trees.

Dendrochemistry, or the study of the chemical element content of tree rings, has the potential for detecting and reconstructing environmental changes that influence soil chemistry over the life of the tree. The major limitation of the dendrochemical method for detecting soil chemical change has been the lack of controlled studies of changing soil environments and incomplete understanding of radial translocation and storage of elements in xylem. Natural variations in the availability of soil nutrients and trace metals with soil pH have been found in several tree species (DeWalle and others 1991, Guyette and others 1992, Mohamed and others 1997), but the responsiveness of tree-ring chemistry to soil chemical changes has been documented in only a limited number of studies and species (McClenahen and Vimmerstedt 1993, Kashuba-Hockenberry and DeWalle 1994, DeWalle and others 1999, Watmough and Hutchinson 1996).

Bondietti and others (1990) proposed use of molar ratios of aluminum (Al)/calcium (Ca) or Al/magnesium (Mg) in tree-rings as indicators of change. Studies by Cote and Camire (1995) and Mohamed and others (1997) showed relationships between available soil aluminum and tree-ring aluminum. DeWalle and others (1999) found Ca/Mn and Mg/Mn ratios in bolewood to be useful in studying impacts of soil acidification, while Watmough and Hutchinson (1996) showed Ca, Mg, Mn, and strontium (Sr) were responsive to surface soil pH changes. Shortle and others (1997) provided evidence that cation levels in tree-rings of red spruce (*Picea rubens* Sarg.) increased due to base cation mobilization induced by atmospheric deposition. Concepts of ecosystem acidification from atmospheric deposition with initial base cation mobilization followed by base cation depletion are well established in the literature (Aber and others 1989).

In this paper we combine past dendrochemical results largely related to effects of ecosystem acidification with data from other past fertilization trials where other types of soil base (liming)

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and acid fertilization occurred. The objectives of these studies were to:

1) demonstrate the responsiveness of tree-ring chemistry in a large number of tree species to the effects of both soil acidification and base addition conditions and
2) to assess the ability of tree rings to preserve a record of past soil chemical changes even where up to 30 years had elapsed since the original soil fertilization.

METHODS

Samples of bolewood from treated and control trees for chemical analysis were collected at a number of sites of past fertilization experiments summarized in table 1. Fertilization occurred several years to over 30 years prior to sampling. In most treatments the fertilizers were applied once to the soil surface in granular or powdered form without incorporation into the soil; however, treatments to whole watersheds at Bear Brook in Maine (site 7 in table 1) and Fernow and Clover Run in West Virginia (sites 5 and 6, table 1) were applied several times annually. Fertilization with ammonium sulfate, aluminum sulfate, NPK without lime, and nitrogen-only fertilizers were categorized as acid treatments. Treatments with lime and other base additions, either separately or in combination with NPK and other compounds were classified as base treatments.

In most instances, wood cores were collected from trees (bark to pith) using increment borers (table 1). Black locust (*Robinia pseudoacacia* L.) samples at Fernow (6d in table 1) were taken from approximately 5-cm thick wood discs cut at breast height from felled trees. Only healthy, overstory dominant or co-dominant trees were sampled, although for red maple (*Acer rubrum* L.) and black locust at Fernow (6c and 6d in table 1), only intermediate or suppressed canopy-position trees were available for sampling.

Two types of sampling with increment borers were conducted depending upon availability of trees and other constraints. In some areas, four cores were removed from each of five trees of each species in each treated or control area, while in other areas two or four cores were removed from 10 trees per species. Cores were extracted at breast height in different quadrants of the bole cross-section at all sites except one, which was sampled at stump height (site 4 in table 1). Wood samples were collected with clean, teflon-coated increment corers, handled only with gloved hands, placed in plastic straws and frozen until they were processed.

In the laboratory, cores were separated into 4- or 5-year growth increments to provide enough samples for analysis by inductively coupled plasma emission spectroscopy. Low-power magnification, clean gloves, and scalpels were used for core separation. Growth increments from the same years for each core were composites by tree for chemical analysis. Black locust wood samples, collected from each annual growth increment using a chisel, were combined by year for all treatment or control trees.

Most chemical analyses of the chemical element content of the wood were conducted at the Plant and Soil Analysis Laboratory on the Pennsylvania State University campus, but in some of the earliest sampling other contract laboratories were used. Although a suite of chemical element concentrations were obtained for the wood samples, the molar ratios Ca/Mn and Mg/Mn are stressed in this paper based on past research (DeWalle and others 1999). Data reported are from wood formed just after the last treatments were applied at each study site.

ACID VS. BASE RESPONSE

Dendrochemical responses to acid and base fertilizations were consistent for most tree species (figs. 1 and 2). Molar ratios of Ca/Mn and Mg/Mn in growth rings formed after the most recent fertilization generally showed increases for base fertilization (above 1:1 line) and decreases for acid fertilizations (below 1:1 line). High ratios in base fertilizations were caused by the combined effects of higher concentrations of Ca or Mg and lower concentrations of Mn. Conversely, lowered ratios in acid fertilizations were due to the combined effects of lower Ca or Mg concentrations and increased concentrations of Mn.

The highest Ca/Mn and Mg/Mn ratios for base fertilized trees relative to control trees occurred for red oak (*Q. rubra* L.) at the Laurel Run Acid Mine Site in Pennsylvania (site 12 in table 1), which received the greatest lime application of 28 tonnes ha^{-1} of pulverized limestone. Heavy liming at Cherry Springs in Pennsylvania (site 8) showed nearly equivalent increases in Ca/Mn and Mg/Mn in sugar maple (*Acer saccharum* Marsh.) and black cherry (*Prunus serotina* Ehrh.) trees. In acid fertilized experiments the largest differences between treated and control trees occurred in Japanese larch (*Larix leptolepis* Sieb. and Zucc.) at Clover Run (site 5) and yellow-poplar (*Liriodendron tulipifera* L.), red maple, black cherry, and black locust trees at the Fernow site in West Virginia (site 6).

Table 1.—*Description of forest fertilization sites and denrochemistry studies*

Study and Ref. No.*	Species	Treatment	Amount, kg/ha	Year Treated	Year Meas.	Site	Trees per treat.	Cores per Tree
1 Acid	*Quercus rubra*	Ammonium nitrate	336 N	1977-78	1995	AR, Boston Mtns.	10	4
2 Base 2a 2b	 *Q. velutina,* *Pinus echinata*	NPK+ lime	1345 N 587 P 477 K 657 Ca 129 Mg	 1973 1973	 1995 1995	MO, Sinkin Exp. For.	7-10	4
3 Acid 3a 3b	 *Q. rubra,* *Liriodendron tulipifera*	NPK	448 N 194 P 93 K	 1970-71 1970-71	 1994 1994	WV, McGee Run, Mt. Zion Rd.	5	4
4 Acid	*Q. coccinea*	Aluminum sulfate	32.4 S	1988	1994	PA, State College	5	4
5 Acid 5a 5b 5c	 *Larix leptolepis,* *Q. rubra,* *Prunus serotina*	Ammonium sulfate	35 N 40 S per year	 1987 1987 1987	 1994 1992 1992	WV, Clover Run	5	4
6 Acid 6a 6b 6c 6d	 *P. serotina,* *L. tulipifera,* *Acer rubrum* *Robinia psuedoacacia*	Ammonium sulfate	35 N 40 S per year	 1989 1989 1989 1989	 1996 1996 1996 2000	WV, Fernow	5 9-10	4 discs
7 Acid 7a 7b 7c 7d	 *Acer saccharum,* *Fagus grandifolia,* *Picea rubens,* *Betula allegheniensis*	Ammonium sulfate	26.5 N 30 S per year	 1989 1989 1989 1989	 1994 1994 1994 1994	ME, Bear Brook	5	4
8 Base 8a 8b	 *P. serotina* *A. saccharum*	Liming	11.2 lime	 1985 1985	 1994 1994	PA, Cherry Springs	10	2
9 Base	*A. saccharum*	Potassium sulfate, lime	224 K 92 S as K2SO4, 500 as Ca and Mg CO3	1989	1994	Quebec, Univ. Montreal, Biol. Sta.	10	2
10 Base	*A. saccharum*	Maple Gro, dried blood, bonemeal, KCl, and dolomite	equiv. to 400 of 4-4-8 fert.	1988	1994	Quebec, Univ. Montreal, Biol. Sta.	5	4
11 Base	*Q. coccinea*	Liming	605 Ca as hydrated lime	1963-64	1990	PA, Black Moshannon	5	4
12 Base	*Q. rubra*	Liming	28000 Ca as ground limestone	1978-80	1988	PA Laurel Run Acid Mine site	10	4

**References: 1 = Snyder and others 1982, 2 = Harris and others 1980, 3 = Auchmoody and Smith 1977, 4 = L. McCormick, Penn State, School of Forest Resources personal communication, 5 = Kochenderfer and others 1995, 6 = DeWalle and others 1999, 7 = Norton and others 1994, 8 = Long and others 1996, 9 = Fyles and others 1994, 10 = B. Cote', U. Montreal personal communication, 11 = Kashuba-Hockenberry and DeWalle 1994, 12 = DeWalle and others 1995.*

Applications of ammonium sulfate several times annually for 8 years appeared to cause the greatest acidification at the Fernow and Clover Run sites.

Some treatments produced ratios that fell closer to the 1:1 line (figs. 1 and 2); differences between treatment and control were not always significantly different. Additions of N fertilizer or NPK fertilizer without lime, such as that in the Boston Mountains of Arkansas (site 1) or the McGee Run (site 3a) and Mt. Zion Road (site 3b) sites in West Virginia, showed evidence of modest acidification response (figs. 1 and 2). Four species at Bear Brook in Maine (site 7) showed modest acid response to treatment with ammonium sulfate treatment. However, unlike trees at Fernow or Clover Run, trees at Bear Brook were sampled after only 6 years of treatment, which was before base cation depletion was developed fully.

Modest base fertilizations of sugar maple in Canada (sites 9 and 10), scarlet oak (*Quercus coccinea* Muenchh.) at Black Moshannon in Pennsylvania (site 11) and shortleaf pine (*Pinus echinata* Mill.), and black oak (*Quercus velutina* Lam.) in Missouri (site 2) showed expected base fertilizer responses, but only modest to small departures from the 1:1 lines (figs. 1 and 2). Aside from the Bear Brook site, modest changes in bolewood chemistry to fertilization in these studies was probably related to the single applications and/or low amounts of fertilizer applied.

Scarlet oak at the State College site in Pennsylvania (site 4) that received aluminum sulfate fertilizer was the only species not exhibiting the expected response for either Ca/Mn or Mg/Mn. Aluminum sulfate was expected to produce an acidifying effect on the soil, but caused a small increase in Ca/Mn or Mg/Mn ratios in tree rings like a base addition. This effect may have been due to replacement of base cations by Al on soil exchange sites and the relatively high base saturation of this soil.

As expected, treatment with nitrogen or NPK fertilizers caused an acidification response in tree-ring chemistry. Liming application with nitrogen fertilizer showed a base response

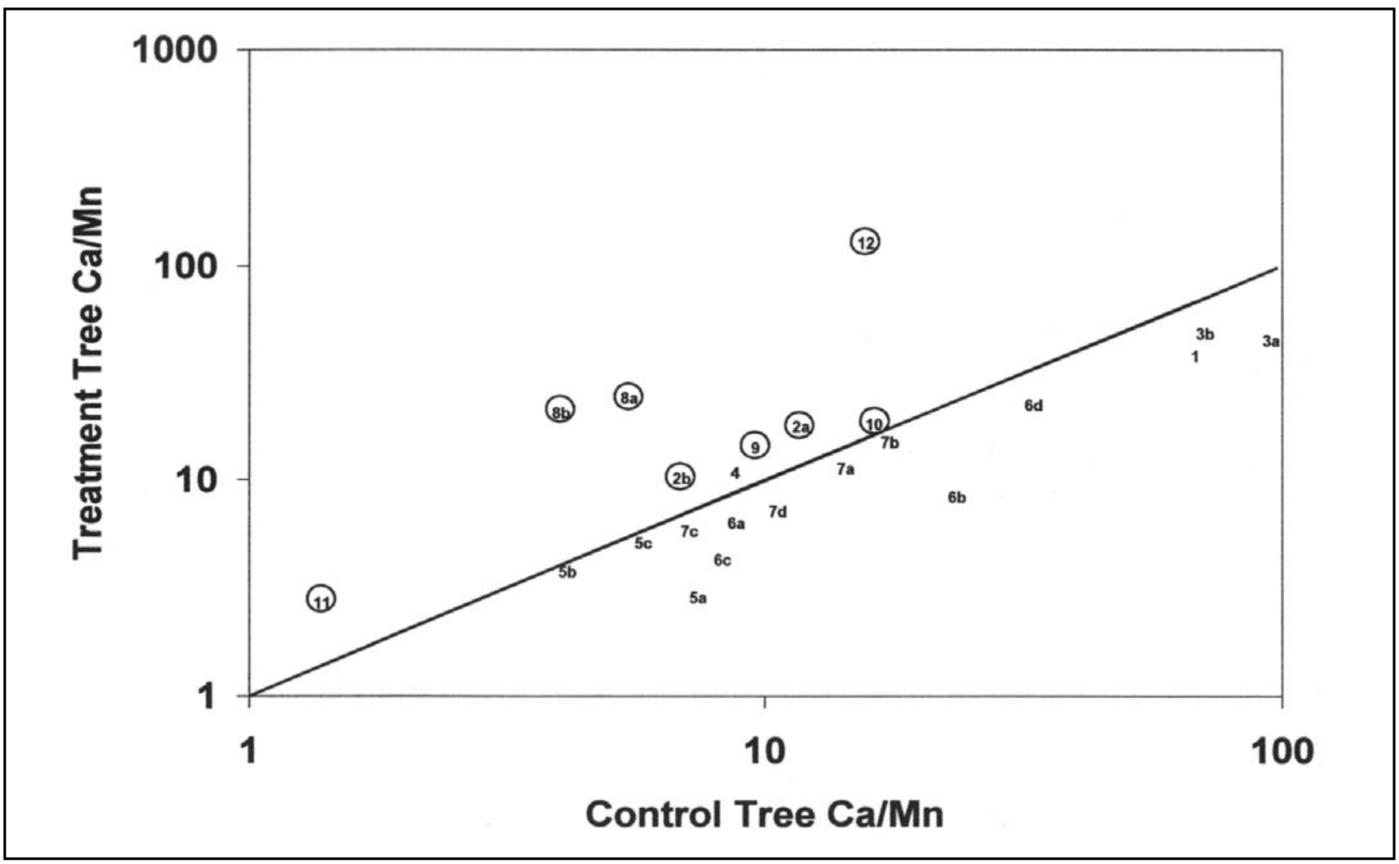

Figure 1.—*Comparison of Ca/Mn molar ratios in bolewood from treated and control trees at sites of forest acid and base fertilization trials. Data were derived from wood growth segments formed after the last treatment. Circles around points denote base cation fertilization, while points without circles denote acidifying fertilization. Numbers and letters of points are keyed to treatments, species, and sites described in table 1.*

indicating that the lime overcame the acidifying effects of N-fertilization.

TREE-RING CHRONOLOGIES

Bolewood sampling to establish the chronology of chemical changes was limited by the width of sap conducting tissue in trees. Trees with wide sapwood tended to show no consistent trends due to fertilization, while those with narrow sapwood showed patterns related to past soil changes. The pattern of Ca/Mn ratios that existed in yellow-poplar cores extracted 30 years after acid treatment with NPK fertilizer at the Mt. Zion site in West Virginia and in scarlet oak cores extracted 25 years after lime treatment at the Black Moshannon site in Pennsylvania are shown for illustration purposes (figs. 3 and 4, respectively). Yellow-poplar is a hardwood species with diffuse-porous wood and a wide band of sapwood, while scarlet oak is a ring-porous hardwood with a moderately wide sapwood zone. The precise width of sapwood and heartwood regions at the time of treatment are not known for either site.

Yellow-poplar showed reduced ratios due to treatment with NPK fertilizer indicating acidification; however, wood tissue formed up to 20 years before and over 30 years after treatment showed effects of treatment (fig. 3). Although yellow-poplar trees appeared quite sensitive to soil chemical change (figs. 1 and 2), the chronology of molar ratios preserved in tree rings shows no evidence of the exact time of treatment. This species clearly cannot be used to date changes in site fertility. Other diffuse-porous hardwood and most conifer species tested, showed similar lack of ability to preserve a chronology of soil chemical change.

Scarlet oak, with its narrower sapwood region than yellow-poplar, showed a different response dendrochemical chronology to lime application (fig. 4). Ratios were similar in treated and control trees prior to treatment. After treatment, relatively high Ca/Mn ratios occurred in rings formed immediately after treatment (1963-1969 segment) and the maximum liming effect appeared about a decade after treatment (1970-1974 segment). The timing of maximum liming

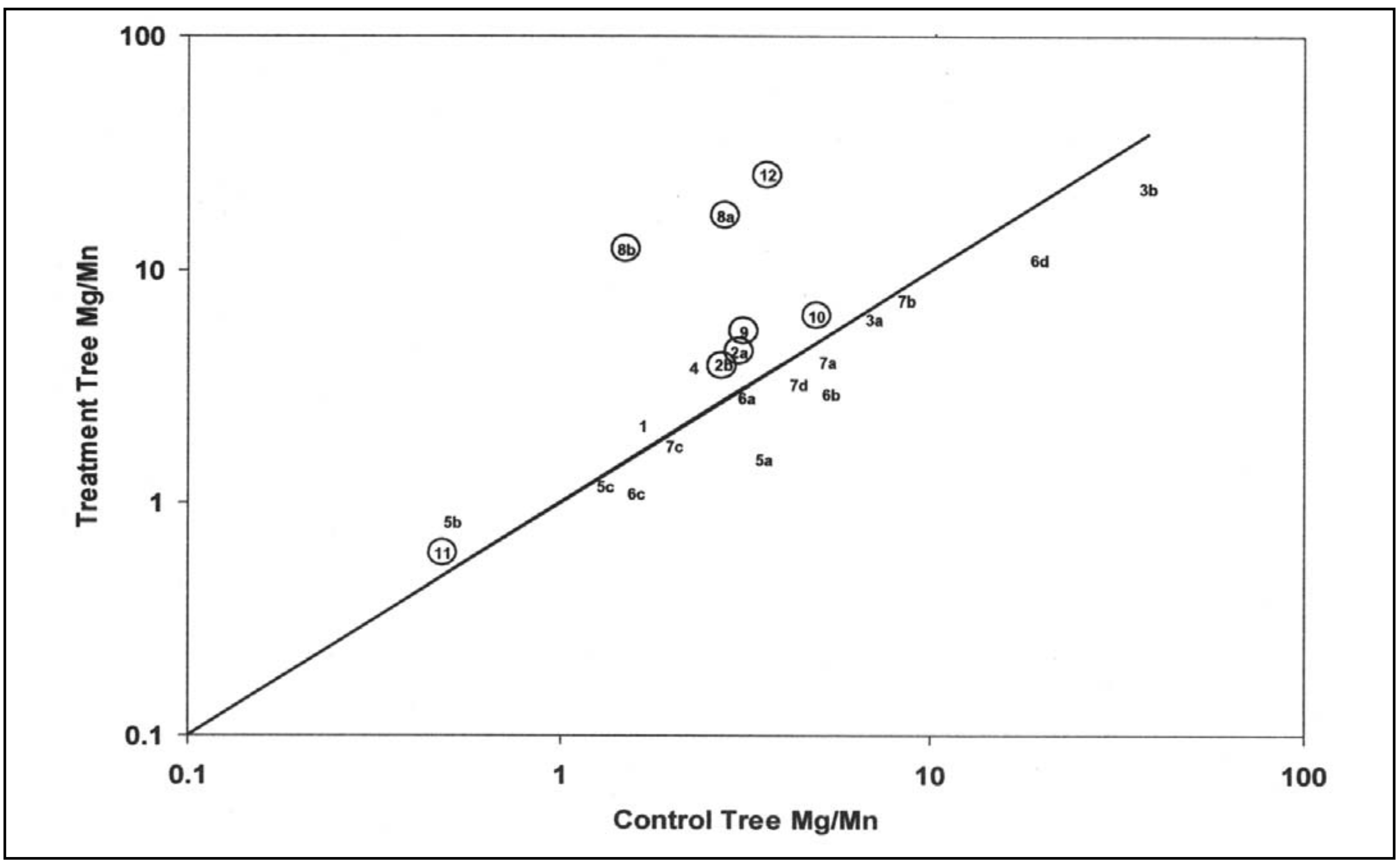

Figure 2.—*Comparison of Mg/Mn molar ratios in bolewood from treated and control trees at sites of forest acid and base fertilization trials. Data were derived from wood growth segments formed after the last treatment. Circles around points denote base cation fertilization, while points without circles denote acidifying fertilization. Numbers and letters of points are keyed to treatments, species, and sites described in table 1.*

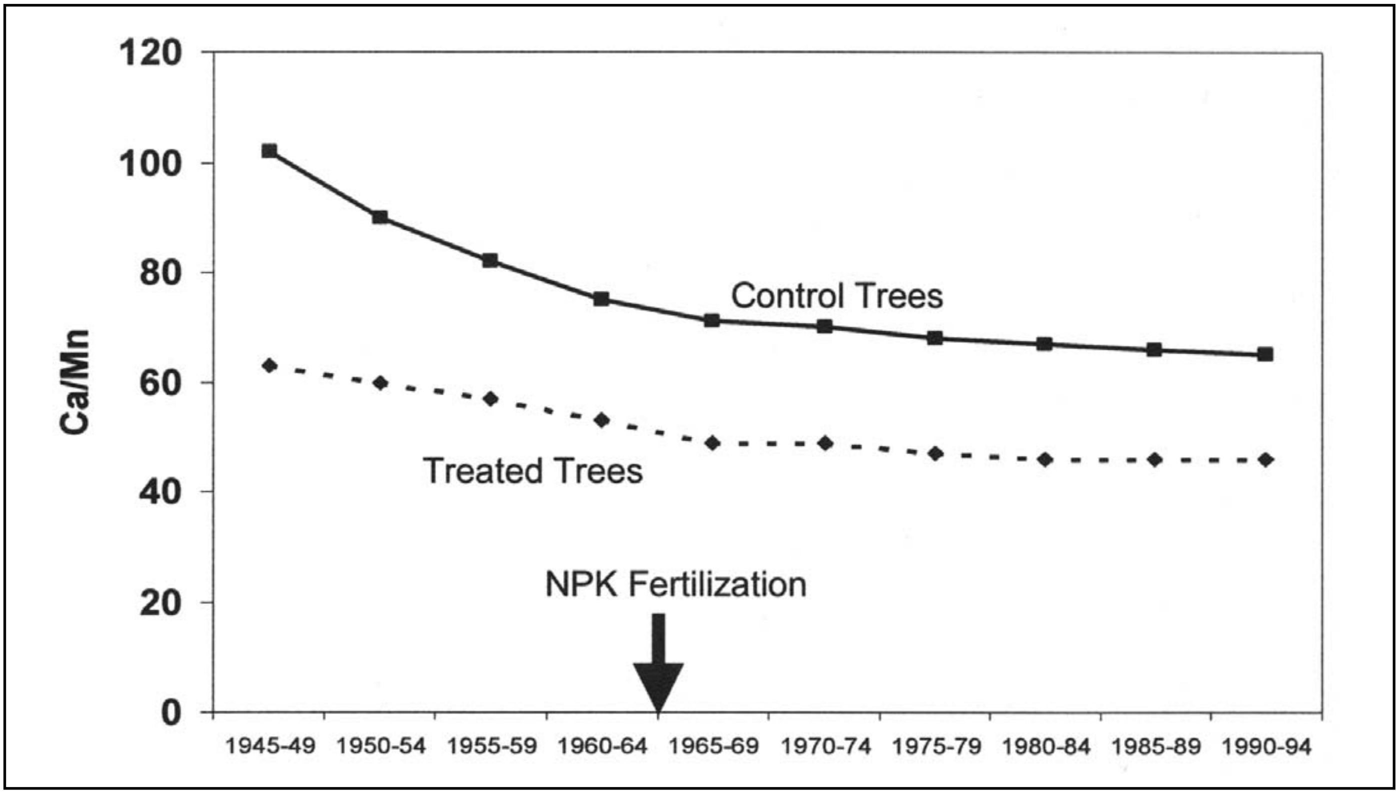

Figure 3.—*Pattern of mean Ca/Mn molar ratios in bolewood from treated and control yellow-poplar trees subjected to one-time NPK fertilization at the Mt. Zion, WV site. Arrow indicates year of NPK fertilization.*

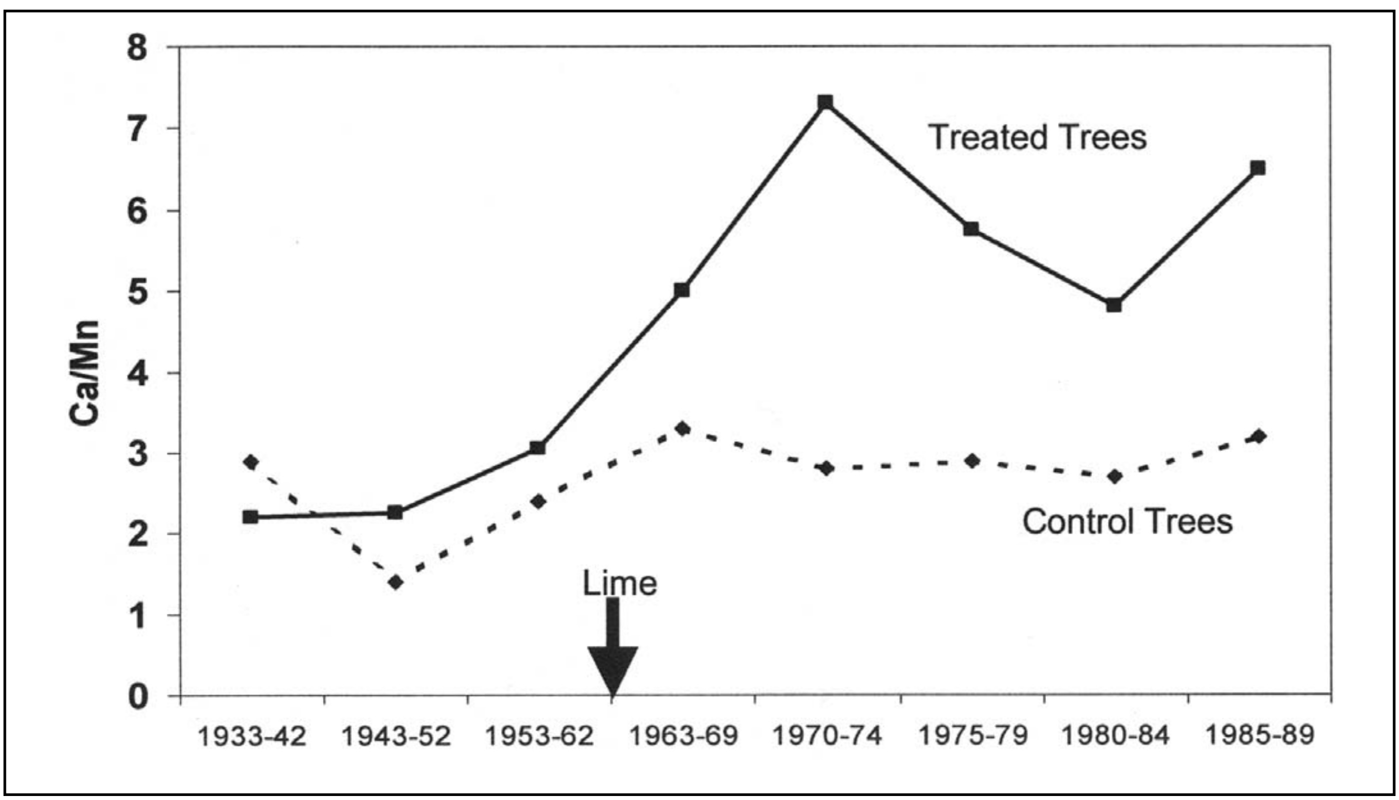

Figure 4.—*Pattern of mean Ca/Mn molar ratios in bolewood from treated and control scarlet oak trees subjected to one-time liming at the Black Moshannon, PA site. Arrow indicates year of liming.*

effect a decade after treatment is possible considering that lime was surface applied and not incorporated into the soil. Gradual convergence of ratios in treated and control trees in wood formed after 1974 was also suggested and this chronology of change after lime treatment was preserved in the bole for up to 25 years after treatment. Thus, it appears that scarlet oak, unlike yellow-poplar, can be used to approximately date changes in soil fertility and plant nutritional status.

All tree species studied were categorized according to their ability to preserve a chronology of chemical changes caused by soil fertilization in tree rings (table 2). Categories are tentative since the magnitude of the treatment and other unknown site conditions also may have influenced the patterns of chemical element changes detected. Since cores were generally divided into segments of 5 years or more, more precise timing of changes could not be determined. The longevity of fertilizer effects on trees is not well known and assessing the effects of forest fertilization could be one of the most useful applications of dendrochemisty.

Japanese larch was the best species tested at providing an exact record of the timing of soil chemical changes. Sapflow in this species appears to be confined to one or two growth rings without much radial translocation of elements (DeWalle and others 1999). Black locust was expected to preserve a good chronology by virtue of its narrow sapwood (4 to 5 rings) and extensive heartwood, but Mg/Mn and Ca/Mn ratios did not show expected trends for unknown reasons.

Table 2.—*Rating of tree species for ability to preserve a dendrochemical chronology*

Species	Chronology preservation
Larix leptolepis	Very good
Quercus rubra	Good
Q. velutina	Good
Q. coccinea	Good
Prunus serotina	Fair
Pinus echinata	Poor
Fagus grandifolia	Poor
Picea rubens	Poor
Betula allegheniensis	Poor
Liriodendron tulipifera	Poor
Acer saccharum	Poor
A. rubrum	Poor

Species with ring-porous wood and narrow sapwood bands, such as the oaks and black cherry, were useful at preserving at least an approximate record of the time of soil chemical changes. Diffuse-porous hardwood species and two other conifers tested, red spruce and shortleaf pine, did not appear to preserve change chronology in any consistent manner.

CONCLUSIONS

Molar ratios Ca/Mn and Mg/Mn in bolewood in a wide variety of deciduous and coniferous species are sensitive indicators of changes in the soil chemical environment due to either acid or base fertilizer treatments. Nitrogen-only, ammonium sulfate, and NPK fertilization of trees without lime caused an acidification response or reduction in Ca/Mn and Mg/Mn ratios in bolewood. Fertilization with lime caused increases in bolewood Ca/Mn and Mg/Mn ratios.

Approximate chronologies of soil fertility changes due to fertilization were preserved for up to 30 years in tree rings of ring-porous hardwood species, such as the oaks and black cherry, and conifers like Japanese larch. However, diffuse-porous hardwoods, black locust, red spruce, and shortleaf pine did not preserve a marked chronological record. Despite limitations imposed by some tree species, careful application of dendrochemistry offers promise as a tool for assessing impacts of fertilization and soil acidity changes.

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