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6. Page: 1

The in-text citation "Mobley, 2011" is not in the reference list. Please correct the citation, add the reference to the list, or delete the citation.

Thank you! Please add the reference: Mobley, M.L. 2011. An Ecosystem Approach to Dead Plant Carbon over 50 years of Old-Field Forest Development. PhD Dissertation, Duke University, Durham, North Carolina. [DOI: 10161/4992](https://doi.org/10.1139/4992)

7. Page: 2

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We have the correct in-text reference: Mobley et al., 2013 and the correct reference: Mobley, M.L., D.D. Richter, and P.R. Heine. 2013. Accumulation and decay of woody detritus in a humid subtropical secondary pine forest. Can. J. For. Res. 43:109–118. doi:10.1139/cjfr-2012-0222

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8. Did you wish to cite the Supplemental Tables S1 and S2? If so, where is the best place to insert this call? (perhaps page 4)

We would like to cite those tables as follows:

Page 4, under section of "Statistical Analysis", "...we chose the simpler model (Burnham and Anderson 2002) (Supplemental S1)." and "...as this autocorrelation coefficient is required for the MDC calculation (Supplemental S2)."

We found a typo for the subheading of "Statistical Analysis", omit one "a".

9. Please check the math and equations as their form as separate math items did not match the individual term as given in the text. Are any in error? I would be happy to correct them for you. Thanks.

We would like to change this text "The SD of the regression slope was computed as." to "The S_b was computed as.", and the following equation to " $S_b = ..$ "

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10. Page: 2

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Should this be the paper from 2013?

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11. Page: 2

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12. Page: 2

Reference "Granato, 2006" is not cited in the text. Please add an in-text citation or delete the reference.

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13. Page: 3

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15. Page: 3

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16. Page: 3

Reference "Richter, Markewitz, Heine, Jin, Raikes, Tian, Wells, 2000" is not cited in the text. Please add an in-text citation or delete the reference.

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How to Estimate Statistically Detectable Trends in a Time Series: A Study of Soil Carbon and Nutrient Concentrations at the Calhoun LTSE

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Quantifying rates of change in soil carbon and nutrients is essential to understanding the global carbon cycle as well as to guiding local management decisions. However, change in soils can be nonlinear, difficult to detect, and dependent on the depth of soil examined. The purpose of this study was to demonstrate how to quantify uncertainty in detection of soil change, which has major implications for the design of soil monitoring projects. We analyzed soils collected over five decades from a long-term soil-ecosystem experiment in the South Carolina Piedmont of the United States. We estimated the minimum detectable change (MDC) using a dataset of soil carbon, nitrogen, calcium, magnesium, and potassium concentrations at four soil depths (0–7.5, 7.5–15, 15–35, and 35–60 cm). Changes over time in the log-transformed concentration at each soil depth were fit with one of three response shapes (linear, exponential, and quadratic) with the best model fit determined by a corrected AIC. The MDC analyses were conducted using repeated-measures models. For soil depths in which elemental change was quadratic, we conducted MDC analyses separately on the decreasing and increasing limbs. We also used paired *t* tests to analyze the MDCs based on only the first and last sample dates. Greater sampling intensity was needed to detect equivalent proportional changes in elements at intermediate depths than at shallower or deeper depths. Our study demonstrates the importance of considering the pattern and rate of the expected change, and how that may vary by element and depth, when designing and evaluating soil monitoring studies.

Abbreviations: AIC, Akaike information criterion; MDC, minimum detectable change.

Understanding rates of change in soil carbon and nutrients is essential to quantifying the global carbon cycle as well as to improving local management decisions. However, change in soils may be slow, and spatial variability in forest soils can make these changes difficult to detect. Spatial heterogeneity in elemental abundance has been found to vary with soil depth (Heinze et al., 2018) and with land-use change from an agricultural field to pine forest (Li et al., 2010). Thus, the sampling intensity required to detect change can vary with soil depth or over time. Studies find different magnitudes and even different directions of change at deeper depths compared to surface depths (Richter and Markewitz, 2001; Mobley et al., 2015; James and Harrison, 2016). Sizeable and biologically important pools of carbon and nutrients can be stored in deep soils (Rumpel and Kögel-Knabner, 2011; Bacon, 2014). Because sampling soils at the frequency and replication necessary to detect change can be labor-intensive and expensive, careful consideration of the most appropriate sampling scheme and intensity is critical to cost-effective soil monitoring (Levine et al., 2014).

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Core Ideas

- Detecting change depends on soil variability, sampling effort and desired confidence.
- We illustrate detectable rates of change using data from the Calhoun Long-Term Soil-Ecosystem experiment.
- The sampling intensity needed to detect change varied with soil depth and chemical element.
- Experience at this LTSE can be used to improve long-term soil monitoring designs.

Estimation of minimum detectable change (MDC) provides information about the sampling intensity needed to detect a change. This approach has been used to determine the number of replicate samples needed to detect changes in soils in natural forests (Johnson et al., 1990; Yanai et al., 2003) and croplands (Yan and Cai, 2008), after management activities such as selective harvesting (Homann et al., 2008; Holub and Hatten, 2019), tillage (Yang et al., 2008) and moldboard plowing (Necpálová et al., 2014), or to predict how soon a change in soil C and nutrients is likely to be detected in national soil monitoring networks (Smith, 2004; Saby et al., 2008). However, MDCs are often reported based on only one or two sampling dates, forcing an assumption that the change will be linear (Yanai et al., 2003; Homann et al., 2008). Moreover, quantifications of detectable soil change are commonly restricted to surface soil horizons (Smith, 2004; Saby et al., 2008; Necpálová et al., 2014; Knebl et al., 2015). Few studies have evaluated the effect of sampling intensity on detecting nonlinear changes or changes at multiple soil depths.

At the Calhoun Long-Term Soil-Ecosystem Experiment (LTSE), long-term, spatially replicated soil monitoring has revealed both depth-dependent and nonlinear changes in soil elemental concentrations over 50 yr of forest development. Topsoils increased in soil C and N during secondary forest development on old fields, while subsoils lost C and N (Mobley et al., 2015). When the young secondary forest was rapidly accumulating biomass, N and the exchangeable nutrient cations Ca, Mg, and K were depleted in the 0- to 60-cm mineral soil (Bacon, 2014). Later in forest development when biomass stopped accumulating, these depletions ceased, and nutrient concentrations either stabilized or began to re-accumulate (Bacon, 2014). Because elemental concentrations changed at different rates, and with linear, exponential, or quadratic relationships over time, depending on the element and soil depth, this dataset provides an opportunity to demonstrate multiple approaches to estimating the minimum detectable rate of concentration change.

This paper demonstrates methods to estimate statistically detectable trends in a time series of observations. We explore the effects of both spatial and temporal sampling intensity on detectable rates of change, as well as the difference in statistical power and the conclusions that result from accounting for a variety of patterns of change through repeated sampling compared to using only the first and last measurement. This study aims to provide useful methods and relationships that can be used to inform the design of other long-term soil monitoring programs.

MATERIALS AND METHODS

Site Description

The Calhoun Long Term Soil-Ecosystem Experiment (LTSE) at the Calhoun Critical Zone Observatory in Union County, South Carolina (34.608° N, 81.724° W) is strongly influenced by the agricultural land use history of the southeastern Piedmont-clearing of mixed-hardwood stands, a century of tillage accompanied by soil erosion and ecosystem carbon loss, and eventual agricultural abandonment. The time span of sam-

pling of the Calhoun LTSE includes planting and five decades of growth of even-aged loblolly (*Pinus taeda* L.) pine. The climate is subtropical (McKnight and Hess, 2008), with mean annual precipitation of 1185 mm and mean annual temperature of 15.2°C (Union 8S Station for 1977–2006; National Climatic Data Center, 2008). The soils are fine, kaolinitic, thermic Oxyaquic Kanhapludults in the Cataula series, derived from granitic gneiss (Richter and Markewitz, 1995). Soil A and E horizons extend to 20 to 40 cm and are sandy loams or loamy sands. The Bt horizons extend to 2 to 3 m and are sandy clay to sandy clay loam in texture and rich in kaolinite and sesquioxides (Richter and Markewitz, 2001). Weathering profiles extend to nearly 40 m (Richter and Markewitz, 1995; Bacon et al., 2012).

Sample Collection and Analysis

The Calhoun LTSE was established in 1957 as a loblolly pine productivity and spacing study, and has a randomized complete block design, with four blocks arrayed across a gentle slope (<3%) capturing differing erosional intensities on the landscape. Each block contains two 0.1-ha plots, planted at 2.4 and 3.0 m spacing. Additional plots within each block at 1.8 and 3.7 m spacing were sampled less frequently and are not used in this study. Previous studies have consistently shown spacing to be insignificant in explaining variation in biomass and soil properties (Bacon, 2014; Markewitz et al., 1998; Mobley, 2011; Mobley et al., 2013).

Soil samples from four depths (0–7.5, 7.5–15, 15–35, and 35–60 cm) were collected at nine sampling dates at intervals of 4 to 9 yr (1962, 1968, 1972, 1977, 1982, 1990, 1997, 2005, 2010; mean sampling interval = 5.75 yr), with the exception that the 15- to 35- and 35- to 60-cm depths were not sampled in 1968 and C and N were not analyzed in 1972. At each sampling date, 20 soil samples were collected from each plot with a 2.0-cm diameter punch tube and composited by depth across the plot. After each collection, samples were air-dried, sieved to 2 mm, and then stored in glass containers. Subsamples were pulverized and analyzed for total C and N analyses via elemental analyzer (Mobley et al., 2015). Carbonates are absent from these deeply weathered, strongly acidic Ultisols (pH < 5 in top 5 m of profile; Richter and Markewitz, 2001), so total C is taken to be soil organic C. Exchangeable Mg, Ca, and K were extracted with 1 M NH₄OAc at pH 7 (Suarez, 1996) and analyzed via atomic absorption spectroscopy for Mg and Ca and atomic emission spectroscopy for K (Bacon, 2014). As the Calhoun LTSE is a longitudinal experiment, soil chemical analyses have been conducted on multiple occasions, albeit with the same methods. To ensure the compatibility of these analyses and to check for sample storage effects, a set of quality control samples (including external standard reference materials, internal reference materials, and a subset of previously analyzed archived samples) were always analyzed alongside unknown samples (Markewitz et al., 1998; Bacon, 2014). Method detection limits were 0.13% for C, 0.02% for N, 0.2 mg kg⁻¹ for Mg, 1.5 mg kg⁻¹ for Ca and 2.0 mg kg⁻¹ for K. Additional details regarding sample processing, laboratory analysis, and quality controls were detailed by Bacon (2014) and Mobley et al. (2015).

Statistical Analysis

Except where otherwise indicated, statistical analyses were performed in SAS 9.4 (SAS Inst., Inc., 2013). To characterize temporal trends in element concentrations measured repeatedly over time, we fit linear and quadratic regression models in *proc mixed* and exponential models in *proc nlmixed* with repeated measures by plot. We estimated models separately for each combination of five chemical elements and four soil depths. We used log-transformed concentrations as the dependent variable and year as the independent variable for linear and exponential equations, and year and year \times year as the two independent variables for quadratic equations (Supplemental Text S1). In each case, we chose the model with the lowest AICc, which is the Akaike information criterion (AIC) that corrects for small sample sizes. If two models had similar AICcs (differing by <2), we chose the simpler model (Burnham and Anderson, 2002) (Supplemental Table S1). Residuals of all models were normally distributed according to the Shapiro–Wilk test; residuals of models fit to untransformed concentrations were not normally distributed. First-order autocorrelation coefficients were quantified using the Durbin–Watson test to determine if the concentration measurements were related to measurements from the previous sampling date, as this autocorrelation coefficient is required for the MDC calculation (Supplemental Table S2). Including block as an independent variable did not improve the model performance according to the AICc, so we decided to omit blocks in this analysis.

We calculated MDC from log-transformed elemental concentrations. Methods of calculating sample size to detect trends for data with repeated measures are available for linear regression (Dang et al., 2008; Guo et al., 2013; Green and MacLeod, 2016). Sample sizes for nonlinear trends would be difficult to interpret, as they would vary over time. To calculate the MDC for quadratic temporal trends, we used the lowest point of the quadratic equation to define a break point, and then fit two linear trends to each part of the dataset using SAS *proc autoreg*. For linear and exponential trends, we used

$$\text{MDC} = N \cdot t_{[n(N-2)]} \cdot S_b \sqrt{\frac{1+p}{1-p}}$$

where N is the number of sampling dates; n is the number of replicates at each sampling date; $t_{[n(N-2)]}$ is the two-tailed t -value defined by Student's t -distribution, based on the degrees of freedom and an α of 0.05; S_b is the standard deviation (SD) of the regression slope; and p is the autocorrelation coefficient for autoregressive lag 1, AR(1) generated from *proc autoreg* (Spooner et al., 2011). The S_b of the regression slope was computed as

$$S_b = \sqrt{\frac{\text{MSE}}{n \sum_{i=1}^N (m_i - \bar{m})^2}}$$

where MSE is the mean square error for the regression; $m_i = 1, 2, \dots, N$; and $\bar{m} = (N+1)/2$ (Spooner et al., 1987). We used the MSE and p from our statistics and computed the MDC as a function of the number of plots (n) and the number of sampling dates (N). The MDC as a percentage change between sampling dates is calculated as

$$\frac{1-10^{-\text{MDC}}}{N} \times 100$$

for a decreasing trend and

$$\frac{10^{\text{MDC}}-1}{N} \times 100$$

for an increasing trend (Spooner et al., 2011). To report the MDC in units of annual percentage change, we divided this by the average number of years between sampling dates for each element and soil depth.

We also calculated the MDC using raw, untransformed data from the first and last sampling year (1962 and 2010) as a paired design in MINITAB 17 (MINITAB, Inc., State College, PA, 2000). This MDC as an annual percentage change is calculated as

$$\frac{(\text{MDC}) / (\text{mean concentration at first date})}{(\text{number of years from first to last dates})} \times 100$$

We then compared the MDCs estimated from the full time series to those estimated from two sampling dates using a t test, paired by the five elements at four soil depths ($n = 20$).

To provide context for the MDC results, we characterized the mean spatial variation in elemental concentrations for each depth using the coefficient of variation (CV) across the eight replicate plots, averaged across the sampling dates. Similarly, we characterized the interannual variation as the CV across seven to nine sampling dates, depending on the element and sampling depth, averaged across the eight plots. To exclude the temporal trend from the interannual variation, we detrended the dataset using the models with time as a predictor (described above). Two-way ANOVA with Tukey's honestly significant difference was used to test for differences in the spatial or interannual CV associated with the chemical element (five levels) and soil depth (four levels) in SAS. The CVs were log-transformed in all of the analyses to meet the assumption of normality of the residuals.

To examine the relationship between CVs (spatial or interannual) and MDCs, we applied Pearson correlation tests using the observations for five elements at four depths as replicates ($n = 20$). We conducted this test for the MDCs that were calculated from the full time series and also for the MDCs that were calculated from the paired comparison of the first and last sampling dates.

RESULTS

Temporal Trends in Soil Element Concentrations

Changes in soil element concentrations from 1962 to 2010 differed by element and sampling depth (Fig. 1; Table 1). Most temporal changes at most depths were best fit with linear regression models, although K at 15 to 35 cm was best fit with an exponential, and N at 0 to 7.5 and 7.5 to 15 cm and Mg, Ca and K at 0 to 7.5 cm were best fit with quadratic functions (Table 1). Total C concentration increased at 0 to 7.5, 7.5 to 15 and 15 to 35 cm from 1962 to 2010. Total N concentration initially decreased and then increased at 0 to 7.5 and 7.5 to 15 cm, with model-estimated break points at 1984 and 1986 respectively, but did not change significantly over time at 15 to 35 cm. At 35 to

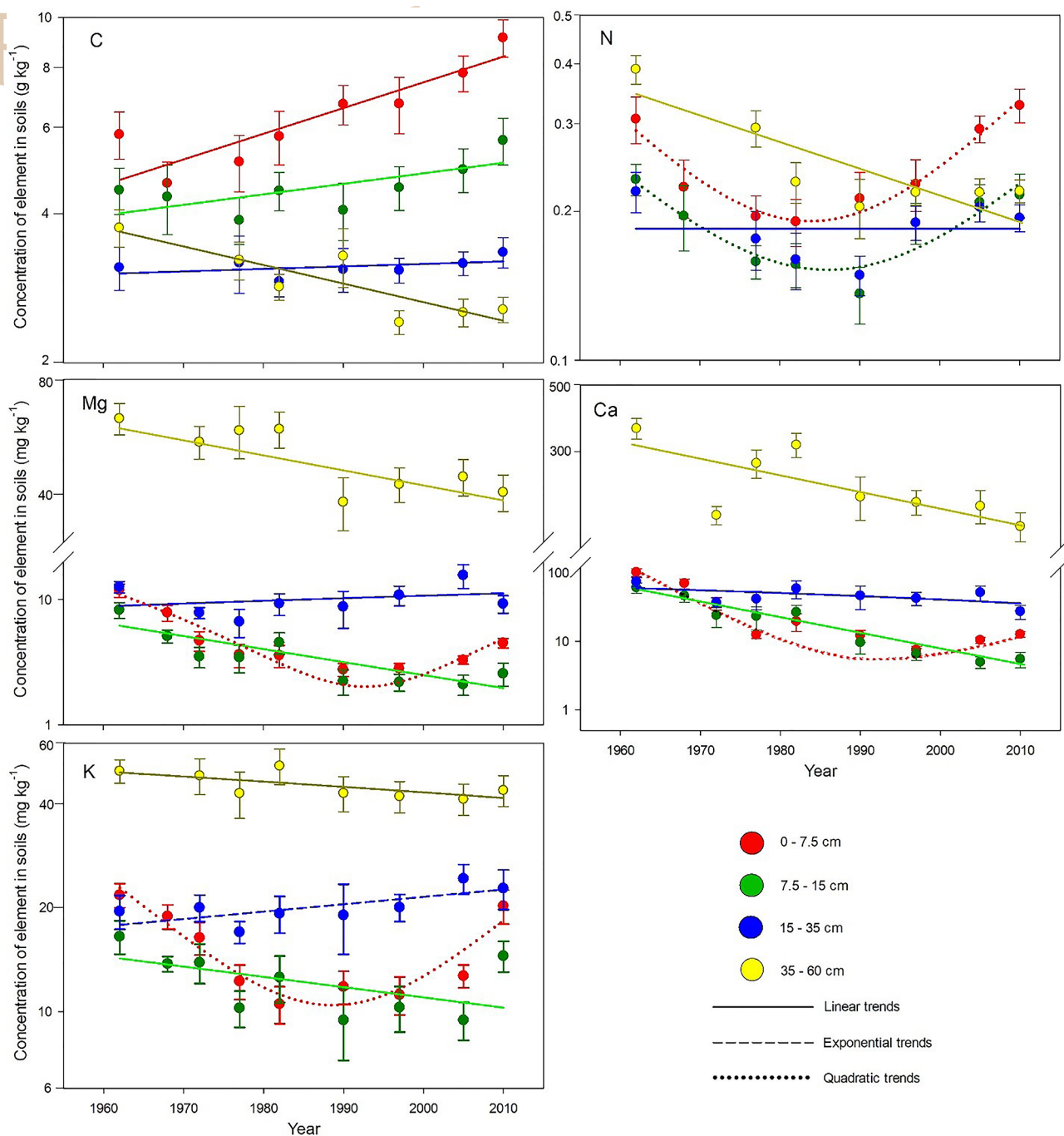


Fig. 1. Best fit regression models, selected by AICc, to describe changes from 1962 to 2010 in concentrations of five elements (total C and N, and exchangeable Mg, Ca, and K), plotted on logarithmic scale at four soil depths (denoted by different colors) at the Calhoun LTSE. Solid, dashed, and dotted lines indicate linear, exponential, and quadratic model trends, respectively.

60 cm, both soil C and N concentrations decreased from 1962 to 2010. Concentrations of exchangeable Mg, Ca and K at 0 to 7.5 cm initially decreased and then increased, with break points estimated at 1991, 1996, and 1988, respectively. From 7.5 to 60 cm, concentrations of exchangeable Mg, Ca and K decreased throughout the time series, with the sole exception of exchangeable K at 15 to 35 cm, which increased.

Minimum Detectable Change

At the Calhoun LTSE, the 7.5- to 15- and 15- to 30-cm depths had larger MDCs than the 0- to 7.5- and 35- to 60-cm

depths (Table 1). Elements differed in MDCs, but at the greatest depth, they were all relatively low. MDC estimates from only the first and last sampling dates tended to underestimate the MDC compared to using the full time series ($p = 0.06$ using a two-tailed paired t test), especially in cases of quadratic trends (Table 1). Splitting the quadratic trends into two linear trends makes it difficult to compare the MDCs to those based on linear trends fit to the full time series, because of the reduction in sampling intensity. For linear trends using the full time series, we found that using only half of the dataset overestimated the MDCs by 14% on average, depending on the element and soil depth (data not shown).

Table 1. Model types, actual rates of change, and minimum detectable change (MDC), using the full time series and using the first and the last sampling date, for five elements at four soil depths at the Calhoun Long-Term Soil-Ecosystem experiment.

| Soil depth | Element | Fitted model type with estimated year of break point where applicable† | Actual annual change from the model | Annual MDC calculated from the full time series | Actual annual change from two dates | Annual MDC calculated from two dates |
|------------|---------|--|-------------------------------------|---|-------------------------------------|--------------------------------------|
| cm | | | % | | | |
| 0–7.5 | C | Linear | 1.4‡ | 0.9 | 1.2 | 0.6 |
| | N | Quadratic, 1985 | -1.9, 3.5 | 0.9, 0.6 | 0.1 | 0.3 |
| | Mg | Quadratic, 1991 | -2.7, 4.5 | 0.6, 0.5 | -1.3 | 0.7 |
| | Ca | Quadratic, 1996 | -2.8, 5.8 | 0.7, 0.5 | -1.8 | 0.9 |
| | K | Quadratic, 1988 | -2.4, 3.2 | 0.5, 0.4 | -0.1 | 0.9 |
| 7.5–15 | C | Linear | 0.5 | 1 | 0.5 | 0.6 |
| | N | Quadratic, 1986 | -1.7, 3.5 | 0.6, 0.9 | -0.1 | 0.3 |
| | Mg | Linear | -1.5 | 1 | -1.4 | 0.9 |
| | Ca | Linear | -1.9 | 1 | -1.9 | 1.1 |
| | K | Linear | -0.4 | 0.8 | -0.3 | 0.8 |
| 15–35 | C | Linear | 0.2 | 0.6 | 0.2 | 0.5 |
| | N | Linear | -0.1 | 0.7 | -0.2 | 0.4 |
| | Mg | Linear | -0.1 | 0.8 | -0.6 | 0.8 |
| | Ca | Linear | -1.2 | 1 | -1.3 | 0.5 |
| | K | Exponential | Factor of 1.0002 | 0.5 | 0.4 | 0.6 |
| 35–60 | C | Linear | -0.7 | 0.5 | -0.7 | 0.4 |
| | N | Linear | -0.9 | 0.5 | -0.9 | 0.4 |
| | Mg | Linear | -0.8 | 0.6 | -0.7 | 0.5 |
| | Ca | Linear | -1 | 0.5 | -1.1 | 0.4 |
| | K | Linear | -0.3 | 0.6 | -0.3 | 0.2 |

† Break point was defined as the lowest point of the quadratic equation.

‡ Values in bold indicate that the actual change exceeded the MDC.

Minimum detectable changes (Fig. 2; Supplemental Fig. S1 and S2) depended on the variability of elemental concentrations and the sampling intensity in space and time. The sampling intensity required to achieve a particular MDC varied with sampling depth and element, as illustrated in Fig. 2 with C and Ca at 15 to 35 and 35 to 60 cm. Both soil C and Ca required greater sampling effort in the intermediate depth (15–35 cm) than the greatest depth (35–60 cm) to achieve a similar MDC. Ca required greater sampling effort than C at 15 to 35 cm, but this difference was less marked at 35 to 60 cm (Fig. 2). The power to detect a change improved (the MDC was reduced) with both increasing numbers of plots and increasing numbers of sampling dates. In this Calhoun LTSE dataset, a greater improvement would be achieved by adding more sampling dates rather than more plots per sampling date. For example, for C at 15 to 35 cm depth, decreasing MDC from 4 to 3% would require adding four more plots per date but only one more sampling date (Fig. 2).

Spatial and Interannual Variation in Soil Element Concentrations

Spatial and interannual variability of soil element concentrations differed by element ($p < 0.001$ in ANOVA for both space and time), with the exchangeable base cations Ca and Mg having higher CVs across plots and across years than total C and N (Fig. 3). Variability also depended on sampling depth (both $p < 0.001$ in ANOVA), with the lowest variability at the greatest depth (35–60 cm) and the greatest variability at intermediate depths (7.5–15 cm and 15–35 cm).

Combinations of elements and sampling depths with higher spatial or interannual variation had higher MDCs estimated from the paired first and last sampling dates ($p < 0.001$ for both spatial and temporal CVs in Pearson correlation tests). Cases with higher spatial variation had higher MDCs calculated from the full time series ($p = 0.002$), but for interannual variation, there was not a significant relationship between the CV and the MDCs ($p = 0.2$).

DISCUSSION Detectable Changes

It is important to quantify detectable rates of change, especially when observations fail to exceed them. It is not enough to report that a significant change was not detected; it is more useful to know whether the lack of significance was attributable to the actual change being relatively small or to the MDC being relatively large due to high variability in the data. If the MDC is large, then it is important to recognize that a meaningful change was not detectable. For example, the first published N budget at the Hubbard Brook Experimental Forest in New Hampshire, USA assumed that there was no change in mineral soil N over time. Based on that assumption, the authors concluded that an input of $14.2 \text{ kg N ha}^{-1} \text{ yr}^{-1}$ by biological N fixation was necessary to balance the ecosystem N budget (Bormann et al., 1977). Subsequently, it was recognized that the uncertainty in soil N change far exceeded this supposed rate of N fixation (Binkley et al., 2000; Yanai et al., 2013).

Minimum detectable change analysis is a useful tool not only for reporting confidence in long-term estimates of soil

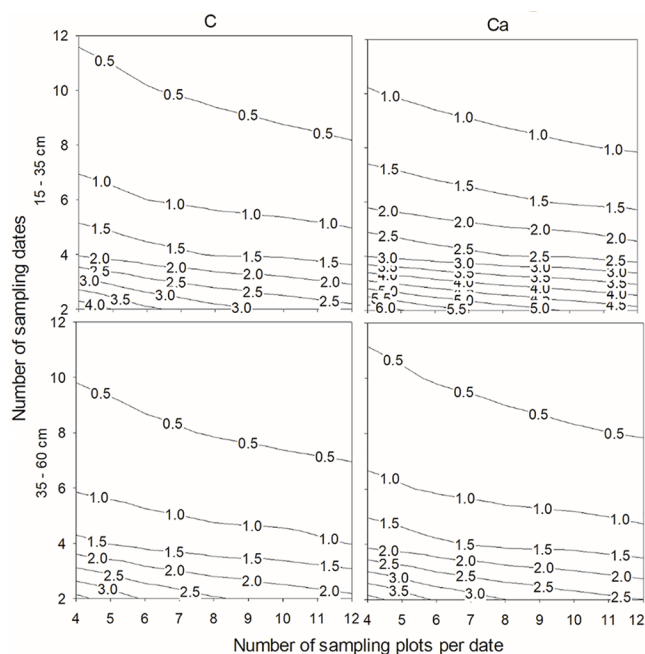


Fig. 2. Minimum annual percentage change detectable (contours) at various temporal (vertical axes) and spatial (horizontal axes) sampling intensities for concentrations of total C and exchangeable Ca at two soil depths for which trends were best fit with linear models.

change but also for guiding the design of future monitoring studies. Specifically, it is important to know the sample size required to detect the expected magnitude of change. In the case of switchgrass plots in the southeastern United States, >100 soil replicates would be required to detect a change of 2 to 3% in SOC stocks with an α of 0.10 (Garten and Wulfschleger, 1999). To detect a 60% change in carbon and nutrients in mineral soils at the Hubbard Brook Experimental Forest with α of 0.05 would require 23 to 47 soil replicates (Johnson et al., 1990). These estimates are based on a single return measurement; our Calhoun study demonstrates that more sampling dates can provide a more accurate assessment of soil change.

The positive correlation between spatial variation and MDCs in this study is consistent with other studies based on five forested or cultivated sites in Virginia (Conant and Paustian, 2002), three sites in Tennessee and western Washington, USA (Conant et al., 2003), and 13 sites across the north central United States (Necpálová et al., 2014). The fact that total C and N had lower variability than the exchangeable base cations in this study could reflect a natural uniformity in biological C and N fixation at the 0.1-ha scale, which are constrained by the distribution of solar energy, and a greater rate of uptake and turnover relative to soil pools (Bacon, 2014). The observation of lower variability in total C and N than exchangeable base cations was reported in a more spatially explicit study including the same 0.1-ha study plots (Li et al., 2010). Lower spatial variability in total C and N than in base cations was also observed in leaf litter across northern hardwood stands (Yanai et al., 2012; Yang et al., 2017); leaf litter both reflects soil nutrient availability to plants and also contributes plant-derived nutrients to soils.

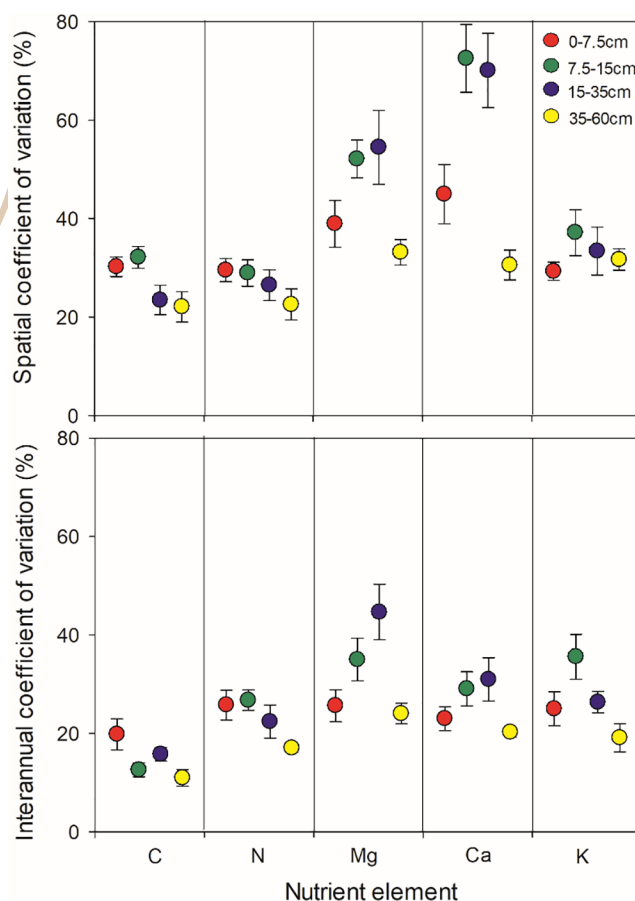


Fig. 3. Spatial variability (mean and standard error of spatial CVs across nine sampling dates) and interannual variability (mean and standard error of interannual CVs across eight replicate plots) for five elements at four soil depths. The interannual CV is detrended and based on the residuals of the regressions shown in Fig. 1. The spatial CV includes systematic variation among plots that is accounted for in the repeated-measures analysis.

Change in the Soils of the Calhoun LTSE Carbon and Nutrients

During the establishment phase of forest development (Peet and Christensen 1987), trees accumulate biomass rapidly and litterfall creates a thick organic horizon. This aboveground demand and accumulation can lead to rapid depletion of mineral soil nutrients. Decreases in soil carbon and nutrient concentrations might be explained by any of a number of factors, including increased rooting depth, greater water uptake, better aerated soils, and mineralization of soil organic matter, possibly primed by root-derived carbon inputs (Cheng and Kuzyakov, 2005; Mobley et al., 2015). As the Calhoun forest entered a transition phase in the 1990s, tree biomass loss to mortality exceeded biomass accumulation of the remaining trees (Peet and Christensen, 1987; Mobley, 2011). Thus tree demand for nutrients was reduced at the same time as decomposition of woody debris and the organic horizon returned N, Ca, Mg and K to the top layers of mineral soil, leading to reaccumulation of those elements at those depths (Bacon, 2014; Mobley et al., 2015). These loss and reaccumulation patterns of N and cations were best described by quadratic trends in concentrations (Fig. 1). Exchangeable cations (Mg, Ca and K) have not reac-

accumulated below 7.5-cm depth, indicating a net ecosystem loss of those elements from subsurface horizons (Bacon, 2014).

Advice for Monitoring Design

Sampling schemes may be designed with multiple goals, such as monitoring multiple elements at multiple soil depths. Our work shows that the MDC will differ by both element and depth if their variability in concentration differs. In the case of soil sampling, the effort allocated to field sampling should be determined by the soil depth that has the highest MDC and thus requires the most sampling to detect change. In our study, for example, the intermediate depths (7.5–35 cm) require more sampling plots per date or more sampling dates than the other soil depths. Thus, if the deepest soils were particularly labor intensive to sample, and the benefit of reduced field effort was deemed greater than the “cost” of an unbalanced sample set, the deep soils could be collected less frequently than the surface and intermediate soils. Note that if the primary goal is to monitor the total elemental content of the soil, then attention is also needed to detecting changes in bulk density, not just concentration.

Upon returning these samples to the laboratory, MDCs could be used to guide investments in analytical effort. ~~it might be possible that not all elements need to be analyzed from every sample.~~ to detect significant trends. In our study, the MDCs were usually similar for the elements analyzed within a soil depth, and differential analytical effort might not be warranted. Analytical effort will not differ for elements sharing an analytical method, even if they differ in MDC, or at least no expense will be spared by not analyzing for the extra elements. For soil and plant tissue samples, total C and N concentrations are generally obtained from a single sample by flash combustion, and exchangeable cations are commonly extracted into solution and analyzed by inductively coupled plasma emission spectroscopy. Thus, there is often no additional cost involved in analyzing for N if samples are being analyzed for C, or in analyzing for K if sample analysis is required for Mg or Ca. In cases where costs per analyte are significant (such as radioisotope analyses), decisions about sampling intensity should be made independently for each analyte.

Our study allowed a comparison of trend detection based on samples from two points in time versus from a time series of eight or nine sampling dates. Obviously, two points is not sufficient to determine whether a trend is nonlinear. In many cases, particularly where change turned out to be nonlinear, using only two points in time underestimated the MDC compared to the estimate based on the full time series. Sampling only two points in time carries the additional risk that even the direction of change may not be consistent with the long-term trend. When variability in observations over time is high, it is possible for two points in time to seriously misrepresent the long-term trend, as occurred over seven sampling dates of forest floor base cations at the Hubbard Brook Experimental Forest (Yanai et al., 1999). It is common for MDCs to be calculated based on one point in time and assuming constant variance (Yanai et al., 2003). In studies where monitoring is ongoing, MDCs should be recalculated as more information becomes available.

CONCLUSION

Detectable difference and power analysis can help determine the sampling effort and designs that are required to detect a specified change or rate of change in soil element concentrations over time. Optimal sampling schemes depend on the elements of interest and the depth of soil sampling, because they affect spatial and interannual variation. Sampling schemes must also consider the expected pattern of change, and incorporate more temporal observations to better capture any nonlinear ecosystem dynamics. Designing effective monitoring schemes is essential to the advancement of knowledge in soil and ecosystem science.

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SUPPLEMENTAL MATERIAL

Supplemental material is available with the online version of this article. The supplemental document contains Text S1, example SAS code used for statistical analyses; Table S1, AICc for three regression models by soil depth; Table S2, First-order autocorrelation coefficients for five elements at four soil depths; Fig. S1, Minimum annual percentage change detectable at various temporal and spatial sampling intensities for five elements at four soil depths; and Fig. S2, Minimum annual percentage change detectable at various temporal and spatial sampling intensities for N, Mg, Ca, and K.

REFERENCES

- Bacon, A.R., D.D. Richter, P.R. Bierman, and D.H. Rood. 2012. Coupling meteoric ^{10}Be with pedogenic losses of ^9Be to improve soil residence time estimates on an ancient North American interfluve. *Geology* 40:847–850. doi:10.1130/G33449.1
- Bacon, A.R. 2014. Pedogenesis and anthropogenesis on the Southern Piedmont. Diss., Duke Univ., Durham, NC. <http://hdl.handle.net/10161/8727>.
- Binkley, D., Y. Son, and D.W. Valentine. 2000. Do forests receive occult inputs of nitrogen? *Ecosystems* 3:321–331. doi:10.1007/s100210000029
- Bormann, F.H., G.E. Likens, and J.M. Melillo. 1977. Nitrogen budget for an aggrading northern hardwood forest ecosystem. *Science* 196:981–983. doi:10.1126/science.196.4293.981
- Burnham, K.P., and D.R. Anderson. 2002. Model selection and multimodel inference: A practical information-theoretic approach. 2nd ed. Springer, Germany.
- Cheng, W., and Y. Kuzyakov. 2005. Root effects on soil organic matter decomposition. In: S.F. Wright and R.W. Zobel, editors, *Roots and soil management: Interactions between roots and the soil*. ASA, CSSA, and SSSA, Madison, WI. p. 119–143.
- Conant, R., and K. Paustian. 2002. Spatial variability of soil organic carbon in grasslands: Implications for detecting change at different scales. *Environ. Pollut.* 116:S127–S135. doi:10.1016/S0269-7491(01)00265-2
- Conant, R.T., G.R. Smith, and K. Paustian. 2003. Spatial variability of soil carbon in forested and cultivated sites: Implications for change detection. *J. Environ. Qual.* 32:278–286. doi:10.2134/jeq2003.2780
- Dang, Q., S. Mazumdar, and P.R. Houck. 2008. Sample size and power calculations based on generalized linear mixed models with correlated binary outcomes. *Comput. Methods Programs Biomed.* 91:122–127. doi:10.1016/j.cmpb.2008.03.001

- Garten, C.T., and S.D. Wullschlegel. 1999. Soil carbon inventories under a bioenergy crop (switchgrass): Measurement limitations. *J. Environ. Qual.* 28:1359–1365. doi:10.2134/jeq1999.00472425002800040041x
- Green, P., and C.J. MacLeod. 2016. SIMR: An R package for power analysis of generalized linear mixed models by simulation. *Methods Ecol. Evol.* 7:493–498. doi:10.1111/2041-210X.12504
- Guo, Y., H.L. Logan, D.H. Glueck, and K.E. Muller. 2013. Selecting a sample size for studies with repeated measures. *BMC Med. Res. Methodol.* 13:100. doi:10.1186/1471-2288-13-100
- Heinze, S., B. Ludwig, H.-P. Piepho, R. Mikutta, A. Don, P. Wordell-Dietrich, M. Helfrich, D. Hertel, C. Leuschner, K. Kirfel, E. Kandeler, S. Preusser, G. Guggenberger, T. Leinemann, and B. Marschner. 2018. Factors controlling the variability of organic matter in the top- and subsoil of a sandy Dystric Cambisol under beech forest. *Geoderma* 311:37–44. doi:10.1016/j.geoderma.2017.09.028
- Holub, S.M., and J.A. Hatten. 2019. Soil carbon storage in Douglas-fir forests of western Oregon and Washington before and after modern timber harvesting practices. *Soil Sci. Soc. Am. J.* (this issue) doi:10.2136/sssaj2018.09.0354
- Homann, P.S., B.T. Bormann, J.R. Boyle, R.L. Darbyshire, and R. Bigley. 2008. Soil C and N minimum detectable changes and treatment differences in a multi-treatment forest experiment. *For. Ecol. Manage.* 255:1724–1734. doi:10.1016/j.foreco.2007.11.037
- James, J., and R. Harrison. 2016. The effect of harvest on forest soil carbon: A meta-analysis. *Forests* 7:308. doi:10.3390/f7120308
- Johnson, C.E., A.H. Johnson, and T.G. Huntington. 1990. Sample size requirements for the determination of changes in soil nutrient pools. *Soil Sci.* 150:637–644. doi:10.1097/00010694-1990090000-00010
- Knebl, L., G. Leithold, and C. Brock. 2015. Improving minimum detectable differences in the assessment of soil organic matter change in short-term field experiments. *J. Plant Nutr. Soil Sci.* 178:35–42. doi:10.1002/jpln.201400409
- Levine, C.R., R.D. Yanai, G.G. Lampman, D.A. Burns, C.T. Driscoll, G.B. Lawrence, J.A. Lynch, and N. Schoch. 2014. Evaluating the efficiency of environmental monitoring programs. *Ecol. Indic.* 39:94–101. doi:10.1016/j.ecolind.2013.12.010
- Li, J., D.D. Richter, A. Mendoza, and P. Heine. 2010. Effects of land-use history on soil spatial heterogeneity of macro- and trace elements in the Southern Piedmont USA. *Geoderma* 156:60–73. doi:10.1016/j.geoderma.2010.01.008
- Markewitz, D., D.D. Richter, H.L. Allen, and J.B. Urrego. 1998. Three decades of observed soil acidification in the Calhoun experimental forest: Has acid rain made a difference? *Soil Sci. Soc. Am. J.* 62:1428. doi:10.2136/sssaj1998.03615995006200050040x
- McKnight, T.L., and D. Hess. 2008. Climatic zones and types. In: T.L. McKnight and D. Hess, editors, *Physical geography: A landscape appreciation*. 9th ed. Pearson Education, London.
- Mobley, M.L. 2011. An ecosystem approach to dead plant carbon over 50 years of old-field forest development. PhD Diss. Duke Univ., Durham, NC. doi:10.1611/4992
- Mobley, M.L., K. Lajtha, M.G. Kramer, A.R. Bacon, P.R. Heine, and D.D. Richter. 2015. Surficial gains and subsoil losses of soil carbon and nitrogen during secondary forest development. *Glob. Change Biol.* 21:986–996. doi:10.1111/gcb.12715
- Mobley, M.L., D.D. Richter, and P.R. Heine. 2013. Accumulation and decay of woody detritus in a humid subtropical secondary pine forest. *Can. J. For. Res.* 43:109–118. doi:10.1139/cjfr-2012-0222
- National Climatic Data Center. 2008. Station Union 8S, Asheville, NC. Coop ID 388786. National Climatic Data Center, Washington, DC. <https://www.ncdc.noaa.gov/cdo-web/datasets/GHCND/stations/GHCND:USC00388786/detail> (accessed 29 Oct. 2008).
- Necpálová, M., R.P. Anex, A.N. Kravchenko, L.J. Abendroth, S.J. Del Grosso, W.A. Dick, M.J. Helmers, D. Herzmann, J.G. Lauer, E.D. Nafziger, J.E. Sawyer, P.C. Scharf, J.S. Strock, and M.B. Villamil. 2014. What does it take to detect a change in soil carbon stock? A regional comparison of minimum detectable difference and experiment duration in the north central United States. *J. Soil Water Conserv.* 69:517–531. doi:10.2489/jswc.69.6.517
- Peet, R.K., and N.L. Christensen. 1987. Competition and tree death. *Bioscience* 37:586–595. doi:10.2307/1310669
- Richter, D.D.J., and D. Markewitz. 2001. *Understanding soil change: Soil sustainability over millennia, centuries, and decades*. Cambridge Univ. Press, UK.
- Richter, D.D., and D. Markewitz. 1995. How deep is soil? *Bioscience* 45:600–609. doi:10.2307/1312764
- Rumpel, C., and I. Kögel-Knabner. 2011. Deep soil organic matter—a key but poorly understood component of terrestrial C cycle. *Plant Soil* 338:143–158. doi:10.1007/s11104-010-0391-5
- Saby, N.P.A., P.H. Bellamy, X. Morvan, D. Arrouays, R.J.A. Jones, F.G.A. Verheijen, M.G. Kibblewhite, A. Verdoodt, J.B. Uveges, A. Freudenschuß, and C. Simota. 2008. Will European soil-monitoring networks be able to detect changes in topsoil organic carbon content? *Glob. Change Biol.* 14:2432–2442. doi:10.1111/j.1365-2486.2008.01658.x
- Smith, P. 2004. How long before a change in soil organic carbon can be detected? *Glob. Change Biol.* 10:1878–1883. doi:10.1111/j.1365-2486.2004.00854.x
- Spooner, J., S.A. Dressing, and D.W. Meals. 2011. Minimum detectable change analysis. Tech notes 7, December 2011. Prepared for US EPA by Tetra Tech, Inc., Fairfax, VA. <https://www.epa.gov/polluted-runoff-nonpoint-source-pollution/nonpoint-source-monitoring-technical-notes> (accessed 1 October 2015).
- Spooner, J., C.J. Jamieson, R.P. Maas, and M.D. Smolen. 1987. Determining statistically significant changes in water pollutant concentrations. *Lake Reservoir Manage.* 3:195–201. doi:10.1080/07438148709354775
- Suarez, D.L. 1996. Beryllium, magnesium, calcium, strontium, and barium. In: D.L. Sparks, A.L. Page, P.A. Helmke, R.H. Loeppert, P.N. Soltanpour, M.A. Tabatabai, C.T. Johnson, and M.E. Sumner, editors, *Methods of soil analysis Part 3: Chemical methods*. SSSA, Madison, WI, p. 575–603.
- Yan, X., and Z. Cai. 2008. Number of soil profiles needed to give a reliable overall estimate of soil organic carbon storage using profile carbon density data. *Soil Sci. Plant Nutr.* 54:819–825. doi:10.1111/j.1747-0765.2008.00305.x
- Yanai, R.D., M.A. Arthur, M. Acker, C.R. Levine, and B.B. Park. 2012. Variation in mass and nutrient concentration of leaf litter across years and sites in a northern hardwood forest. *Can. J. For. Res.* 42:1597–1610. doi:10.1139/x2012-084
- Yanai, R.D., T.G. Siccama, M.A. Arthur, C.A. Federer, and A.J. Friedland. 1999. Accumulation and depletion of base cations in forest floors in the northeastern United States. *Ecology* 80:2774–2787. doi:10.1890/0012-9658(1999)080[2774:AAD0BC]2.0.CO;2
- Yanai, R.D., S.V. Stehman, M.A. Arthur, C.E. Prescott, A.J. Friedland, T.G. Siccama, and D. Binkley. 2003. Detecting change in forest floor carbon. *Soil Sci. Soc. Am. J.* 67:1583–1593. doi:10.2136/sssaj2003.1583
- Yanai, R.D., M.A. Vadeboncoeur, S.P. Hamburg, M.A. Arthur, C.B. Fuss, P.M. Groffman, T.G. Siccama, and C.T. Driscoll. 2013. From missing source to missing sink: Long-term changes in the nitrogen budget of a northern hardwood forest. *Environ. Sci. Technol.* 47:11440–11448. doi:10.1021/es4025723
- Yang, X.M., C.F. Drury, M.M. Wander, and B.D. Kay. 2008. Evaluating the effect of tillage on carbon sequestration using the minimum detectable difference concept. *Pedosphere* 18:421–430. doi:10.1016/S1002-0160(08)60033-8
- Yang, Y., R.D. Yanai, C.R. See, and M.A. Arthur. 2017. Sampling effort and uncertainty in leaf litterfall mass and nutrient flux in northern hardwood forests. *Ecosphere* 8:e01999. doi:10.1002/ecs2.1999