Measuring Environmental Change in Forest Ecosystems by Repeated Soil Sampling: A North American Perspective

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Environmental change is monitored in North America through repeated measurements of weather, stream and river flow, air and water quality, and most recently, soil properties. Some skepticism remains, however, about whether repeated soil sampling can effectively distinguish between temporal and spatial variability, and efforts to document soil change in forest ecosystems through repeated measurements are largely nascent and uncoordinated. In eastern North America, repeated soil sampling has begun to provide valuable information on environmental problems such as air pollution. This review synthesizes the current state of the science to further the development and use of soil resampling as an integral method for recording and understanding environmental change in forested settings. The origins of soil resampling reach back to the 19th century in England and Russia. The concepts and methodologies involved in forest soil resampling are reviewed and evaluated through a discussion of how temporal and spatial variability can be addressed with a variety of sampling approaches. Key resampling studies demonstrate the type of results that can be obtained through differing approaches. Ongoing, large-scale issues such as recovery from acidification, long-term N deposition, C sequestration, effects of climate change, impacts from invasive species, and the increasing intensification of soil management all warrant the use of soil resampling as an essential tool for environmental monitoring and assessment. Furthermore, with better awareness of the value of soil resampling, studies can be designed with a long-term perspective so that information can be efficiently obtained well into the future to address problems that have not yet surfaced.

ENVIRONMENTAL CHANGE is monitored in North America through organized networks that make repeated measurements of the environment that include weather, stream and river flow, air and water quality, and forest growth. The time-series data generated by these networks represent the core of our predictive understanding of how human activities affect our air, water, and trees (Likens, 1989; Magnuson, 1990). Missing from these invaluable records, however, are data from soil remeasurement networks, particularly in forested settings. Despite the importance of soils as a controlling component of terrestrial and aquatic ecosystems, efforts to document changes in forest soils through repeated measurements are largely nascent and uncoordinated.

Traditionally, soil development has been viewed as a set of processes that takes place across centennial to millennial time scales (Walker and Syers, 1976). Important physical and chemical characteristics of soils, however, have been shown to change across time scales of less than a decade (Varallyay et al., 1990). This information has led to a growing recognition of the dynamic nature of soils and the need to better understand how soils change and how fast these changes occur (Tugel et al., 2005). As the rate and extent of human-caused soil change increases, so too does the value of repeated soil sampling. In the absence of direct measurements of soil change, alternative approaches have been used to infer soil change, such as chronosequences (space for time substitution), watershed mass balances, dendrochemistry, and short-term manipulations. These approaches provide coarse estimates of soil change, and all require assumptions regarding the link between the soil property of interest and the property

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Abbreviations: CRT, characteristic response time; LTSE, long-term soil experiment.

being measured. Errors inherent in these methods also limit certainty in inferring soil change. For example, the need to estimate mineral weathering fluxes in the watershed mass balance approach adds large uncertainty to estimated rates of soil change. Computer modeling is also used (Sullivan et al., 2011), but soils are complex systems controlled by high-order interactions of climate, geomorphology, parent materials, vegetation, and humans, which have not been well quantified. The accuracy of the information gained from inferential approaches requires verification by direct measurements of soil change, which is best accomplished by repeated soil measurements using standard methodologies with proper quality control.

The method of repeated soil sampling to detect change falls under the broad category of long-term soil experiments (LTSEs), which includes observational studies of soils subject to uncontrolled environmental changes, as well as treatments specifically designed to protect water quality and improve land management for the production of food, wood products, fiber, and other ecosystem services. To facilitate networking among the world's long-term soil experiments, a global inventory of LTSEs has been launched (Richter and Billings, 2008; Richter et al., 2007) with an advanced-format website (http://ltse.nicholas. duke.edu; accessed 31 May 2012).

Repeated soil sampling can be explicitly defined as the monitoring of soils to determine if changes occur either through natural or human-induced drivers that include climate change, changes in air quality, invasive plants and animals, land use activities, and other unforeseen factors that might alter ecosystems. In eastern North America, repeated soil sampling has begun to provide valuable information on the consequences of environmental issues such as air pollution through records of soil change in forested settings. In an effort to support and coordinate this work, the Northeastern Soil Monitoring Cooperative (NSMC) was formed in 2007 (Lawrence and Bailey, 2007). As a product of the NSMC, this review summarizes the methods and research highlights of repeated soil sampling in this region, in forested settings that have been subjected to changing levels of acidic deposition. Our objectives were to: (i) present the concepts and methodologies applied in soil resampling; and (ii) summarize key forest soil studies to demonstrate the application and value of various resampling approaches. The intent of this review is to further the development and use of soil resampling as an integral method for recording and understanding environmental change in forested settings. The review begins with the origins of soil monitoring to credit the foresight of early soil scientists and demonstrate how the value of a well-maintained soil collection grows with time.

Origins of Long-Term Soil Monitoring: Rothamsted and the Russian Soil Collections

The longest running soil experiments have been conducted in England at the Rothamsted Experimental Station, which dates back to the mid-1800s (Rasmussen et al., 1998). The ongoing research at Rothamsted has focused on long-term sustainability of agricultural management approaches but has also included the monitoring of agricultural sites that were allowed to regrow to forest more than a century ago. The combination of managed plots and untreated natural plots has provided the opportunity

to evaluate the interactions of various agricultural practices with long-term environmental drivers such as acidic deposition and climate change. In one study of forested soils, pronounced differences in soil acidification were observed between a site that had been heavily limed before forest regrowth and a site that had been unlimed since the 18th century (Blake et al., 1999). The previously limed plots showed minimal decreases in pH, but the previously unlimed plots exhibited substantial acidification. In the acidified plots, declines in soil pH and exchangeable Ca through the first half of the 20th century were attributed about equally to forest regrowth and acidic deposition, but from 1964 to 1991, proton sources from acidic deposition were estimated to be five to six times greater than internal sources. This result is unique in that it demonstrates differences in the sensitivity of soils to acidic deposition as a result of management history.

Other opportunities for long-term soil monitoring to define environmental change were provided by early work by Russian soil scientists, including Vasily Vasil'evich Dokuchaev (1846–1903), regarded by many as the founder of soil science. In response to a massive crop failure in the Russian steppe caused by drought in 1891, Dokuchaev was enlisted by the Russian Department of Agriculture to investigate the problem and recommend management solutions. As part of this work, he established research stations where he and his colleagues conducted a number of experiments. He also instituted the collection and archiving of intact soil profiles (monoliths) that have been retained by the Central Dokuchaev Soil Museum in St. Petersburg, Russia, now under the auspices of Saint Petersburg University and the Academy of Agricultural Sciences of Russia (Lapenis et al., 2008). Soil monoliths dating back to the 1890s are retained by the museum, which houses the world's oldest collection of intact profiles available for analysis.

One of the original research stations established by Dokuchaev in 1892 has been maintained as a part of the Dokuchaev Institute and now lies within the Kamennaya National Park, one of the few remaining sanctuaries of uncultivated steppe soil. Samples of the native Chernozem were collected within this preserve and archived in 1920, 1947, 1970, and 1998. These samples have enabled an assessment of climate change effects on steppe soils during the 20th century (Lapenis et al., 2008). Precipitation, previously less than evapotranspiration, increased to levels that exceeded evapotranspiration during the second half of the 20th century. Soils that were previously not subjected to leaching lost 17 to 28 kg m^{-2} of Ca in the form of carbonates from the upper profile from 1970 to 1998 as a result of a regionwide shift in the hydrologic balance. The archived soils were essential in demonstrating how the pool of pedogenic carbonates responded to the changing hydrologic regime resulting from climate change in this region.

A second example of the value of these early collections was shown in a study of acidic deposition effects on forest soils (podzoluvisols in the FAO classification) near St. Petersburg (Lawrence et al., 2005). Soil samples collected in 1926, 1964, and 2001 in the experimental forest of the St. Petersburg State Forestry Academy revealed a transition from base cation-exchange buffering to Al buffering as base cations were depleted by accelerated leaching due to acidic deposition. Neutralization of acidic deposition from 1926 to 1964 occurred through the release of base cations, but continued acidic deposition subsequently mobilized Al during

1964 to 2001, thereby demonstrating a shift from cation exchange to weathering of Al-bearing minerals as the primary mechanism of acid neutralization. These results were noteworthy because they provided direct support for the long-standing theory of soil acidification by acidic deposition postulated by Ulrich (1988) through his research on German soils.

Concepts and Methodologies of Soil Resampling

The concept of soil resampling to detect changes in soil properties has only recently been embraced by the scientific community because of a long-standing view that the rate of soil change was too small to detect in the presence of the high spatial variability (horizontal and vertical) typically expressed by forest soils. Our understanding of temporal and spatial variability in soils has improved, however, as have methods for addressing these factors. These methods incorporate a better understanding of sources of soil measurement instability, which can both mask real changes and create artifactual trends. Measurement instability in soil monitoring has been described by Desaules (2012) as a six-linked chain that extends from site selection to laboratory analysis; each link is a possible source of measurement instability that needs to be quantified and included with interpretation of the results.

Temporal Variability

The time scales of soil change vary greatly among different soil properties. Because the gaseous, liquid, solid, and biotic phases of the soil respond to environmental change at different rates, these phases require different lengths of time to adjust to a new environmental condition. This adjustment time has been termed the *characteristic response time* (CRT) by Arnold et al. (1990). The CRT reflects the temporal dynamics of soil properties and increases in the following general order: gaseous phase > liquid phase > biotic phase > solid phase. Considering the multiphase nature of the soil system, it is impossible to determine soil change or temporal variability by only one characteristic. Each soil phase and property has its own response time (Table 1). The CRT of labile soil properties such as gaseous concentrations, moisture, and temperature can be nearly as short as for those same properties in the aboveground atmosphere. Soil properties such as mineralogy and particle density can have a CRT comparable to that of the lithosphere. The CRT can be linked to the reversibility of soil changes. Some soil properties that change quickly in response to an environmental change can revert quickly when conditions return to the previous state. Some soil properties with long CRTs, however, can be changed quickly by a disturbance but may require a greater length of time for complete reversal or may not be irreversible without additional disturbance events. For example, properties of the upper profile such as exchangeable cation concentrations that are dependent on the release of elements through mineral weathering may not revert to original conditions if the controlling mineral has become depleted.

Soil changes can be expressed as random variability that results from sporadic events such as extreme weather or fire (Varallyay et al., 1990). Intensification of weather extremes has been identified as an important characteristic of modern climate change (Jentsch et al., 2007) that will undoubtedly manifest in greater variability in soil properties. Although abrupt changes in some properties may be reversible in the short term (such as redevelopment of the O horizon after fire), abrupt changes in

Table 1. Characteristic response time (CRT) of various soil properties (modified from Varallyay et al., 1990).

CRT	Soil physical properties	Soil chemical properties	Soil biological properties
yr			
<0.1	moisture content temperature soil air composition bulk density total porosity infiltration rate hydraulic conductivity	NO_3 content total N	
0.1–1	field capacity rate of diffusion	nutrient status soil solution composition pH	microbial biomass microbial activity fine roots enzyme activity species spectra
1–10	soil structure aggregate stability permafrost slickensides heat capacity heat conductivity	soil acidity carbonate content cation exchange capacity exchangeable cations base saturation	annual roots mezofauna
10–100	specific surface area	organic matter content quality of humus clay mineral association	soil biota tree roots
100–1000	soil color soil depth	primary mineral composition mineral fraction chemical composition Fe concretions	
>1000	soil texture rock content particle density parent material horizons	clay mineralogy	

other soil properties may persist for decades or longer (such as erosion of the mineral soil from high-intensity rainfall). In each case, variability may appear random with respect to time, with unpredictable recurrence intervals. Properties linked to weather can also express year-to-year variability that appears random, despite climate forcing that imparts a warming trend for the overall data record (Easterling and Wehrner, 2009).

Stochastic or cyclic variability with time can also complicate detection of a change induced by an environmental perturbation. Cyclic changes in soil properties are usually related to seasonal or diurnal fluctuations that are commonly expressed in properties such as soil moisture and temperature (Davidson et al., 1998), depth to the saturated zone (Lapenis et al., 2008), and C inputs from fall leaf drop (Goodale et al., 2009). In contrast, directional trends in soil properties are generally viewed across multiple years or decades.

Spatial Variability

Detection of changes in soil properties with time can be obscured by spatial variability because it is not possible to collect soil from the same volume more than once. The magnitude of spatial variability is influenced by the size of the study area, physiographic or landscape heterogeneity within that study area, and the specific soil property of interest (Lin et al., 2005). As the size and heterogeneity of the study area increases, the magnitude of soil variability generally increases (Boone et al., 1999).

Nonagricultural soils, and in particular forest soils, can express substantial variations in soil properties across a horizontal scale as small as a few centimeters or less. Factors such as microtopography, the position of rocks and boulders, overstory and understory vegetation and root distribution, and animal activity create variability that often cannot be controlled by reducing the size of the study area. Numerous publications have provided evidence to support this conceptualization of forest soil variability (Burrough, 1993; Heuvelink and Webster, 2001; Lin et al., 2004; Wilding and Drees, 1983; Wilding et al., 1994).

Differences in the magnitude of spatial variability are also evident with soil depth. Surface soils in humid, temperate regions are more subject to changes in temperature, moisture, and especially biological processes than soils deeper in the profile and for these reasons can exhibit greater spatial variability, both vertically and horizontally; however, the magnitude of variability with depth can also depend on factors such as human activity, hydrology, geology, and landforms. For example, in acid forest soils developed in glacial till and glaciofluvial sands, Fournier et al. (1994) found greater coefficients of variation for exchangeable base concentrations in mineral soil than in organic horizons across a 20- by 20-m site.

The magnitude of spatial variability varies considerably with the soil property. Texture, mineralogy, soil thickness, and color are less variable than more dynamic properties such as moisture content, infiltration rate, hydraulic conductivity, redox state, weathering rate, biological activity, and organic matter content. In particular, the observed coefficients of variation for soil hydraulic properties are often >100% (Wilding and Drees, 1983; Wu et al., 1997). The spatial heterogeneity of many soil properties is greatly reduced by plowing, fertilization, and other practices associated with agricultural crop production, but successional forest growth on previously cultivated soils reestablishes the heterogeneity of soil properties within a few decades (Li et al., 2010).

Study Designs and Statistical Considerations for Detecting Soil Changes

Various methods have been used in North America for resampling soils to detect change (Table 2). Resampling is generally directed by the previous experimental design to maximize comparability and, whenever possible, apply statistical tests to paired observations. In most of these studies the forest floor and upper soil horizons were sampled because they represented the horizons where temporal changes were most expected from influences such as vegetation growth and air pollution. Changes in some measurements can be larger at deeper positions in the profile, however, such as observed for N concentrations by Billings and Richter (2006). The monitored soil properties in these resampling studies were typically pH, total organic C, total N, exchangeable cations, and base saturation. Most often, units of concentration are used to evaluate soil change, but changes in the total amount of an element within the soil can yield valuable information regarding ecosystem processes and functions. Conversion of concentration to mass per unit area for the full soil profile requires a number of additional measurements such as bulk density, coarse fraction volume, and thickness of the profile intervals being sampled. These factors can vary substantially in x and y space and therefore require a substantial effort to quantify. Inevitably, these additional measurements reduce the precision with which the property of interest is quantified and therefore reduces the ability to detect change with time. Further work is needed to: (i) improve methods of measurement needed to quantify whole-profile content; and (ii) better quantify the uncertainty of these measurements.

Detecting change with repeated sampling often involves comparing data at two points in time, which lends itself to the use of t-tests or rank sum tests for non-normal data. In some cases, however, the original sampling may have been done from a single pit. To enable statistical testing, the variance obtained from replicated pits in a repeated sampling can be assumed applicable to the original sampling, as in the studies of Bailey et al. (2005) and Hazlett et al. (2011). This enables a one-sample t-test to be used, or the original value can be compared with a confidence interval developed from the resampling data.

For detection of a given change, a well-designed resampling scheme to address spatial variability can greatly reduce the needed sampling intensity. For example, Yanai et al. (2003) simulated the number of experimental units (forest stands) needed to detect differences in forest floor organic C mass based on a regional study of 30 stands by Friedland et al. (1992). By adopting a paired sampling design, only 38 experimental units were needed to detect a 20% change in organic C mass with a statistical power of 75%. With an unpaired sampling design, >80 experimental units were required to detect a 20% change with the same statistical power. In a geostatistical simulation with modeled data, increased precision, and therefore improved ability to detect change, was also obtained with paired designs, particularly with smaller sample sizes (Papritz and Webster, 1995). If the standard error is expected to vary widely among samplings, a bootstrapping technique can be used on a

Table 2. Selected forest soil resampling studies in North America.

Location	Time interval yr	Sampling design	Studied variables	Results	References
Adirondack Mountains, New York	72–74	individual pits sampled by horizon down to the C in 1930–1932 and 1984 at 48 plots; Oa and Oe horizons sampled in 2004 at 36 of the 48 plots	pH and extractable Ca	decreases in extractable Ca in Oe, Oa, and E horizons; decreases in pH in moderately acid (pH > 4.0) horizons	Johnson et al. (1994, 2008a), Bedison and Johnson (2010)
Walker Branch watershed, Tennessee	32	forest floor sampled by horizon, mineral soil sampled by 15-cm increments to 60 cm with auger; samples from 8 plots	exchangeable Ca, Mg, K, and C, total N	decreases in exchangeable Ca and Mg, increases in K, no trends in C or N	Johnson et al. (2007, 2008b), Trettin et al. (1999)
Calhoun Experimental Forest, South Carolina	30	composited samples from 20+ subsamples taken with punch-tube sampler (2-cm diam.) from four soil layers within 0 to 60 cm	exchangeable cations, pH, organic C, N, and P, trace elements	decreased base saturation, pH, organic N; increased acidity	Markewitz et al. (1998), Richter et al. (1994)
Allegheny National Forest, Pennsylvania	30	samples from four plots; in the first sampling, one pit per plot was sampled by horizon; resampling comprised four pits located around the original pit, sampled by horizon	pH, total C, and exchangeable cations	decreases in exchangeable Ca and Mg and pH at all depths; increases of exchangeable Al in Oa/A and upper B horizons	Bailey et al. (2005)
Coweeta Hydrologic Lab., Watersheds 17 and 18, North Carolina	20	triplicate composite soil samples collected by horizon (Oi, Oe, Oa, A, and B or AB) from subsamples taken with a punch-tube sampler	pH, exchangeable cations, base saturation	decreases in pH, exchangeable bases	Knoepp and Swank (1994)
Oak Ridge National Environmental Research Park, Tennessee	18	forest floor sampled by horizon, mineral soil sampled by 20-cm increments with auger; soil bulk density sampled with core sampler	total C and N	7% increase in forest floor C; no change in C or N in mineral soil	Johnson et al. (2003)
U.S. Northeast, Direct Delayed Response Project (DDRP) watersheds	17	one soil pit excavated in 139 DDRP watersheds; samples collected from each horizon down to the C horizon	pH, exchangeable cations and acidity, base saturation, cation exchange capacity (CEC), total C and N	decreases in pH and exchangeable base cations (especially Ca), increases in exchangeable Al in organic horizons; no change in CEC, C, or N	Warby et al. (2009)
Plastic Lake watershed, Ontario	16	samples from 15 plots, one pit per plot at each sampling, sampled by horizon down to the C	pH and exchangeable cations	decreases in pH and exchangeable Ca conc. and content	Watmough and Dillon (2004)
Turkey Lakes watershed, Ontario	15–19	forest floor sampled by horizon in one subwatershed; mineral soil sampled by horizon at seven plots across the site	pH, organic C, total N and cations, exchangeable cations	forest floor: increased total N content, exchangeable Ca and Na, no change in organic C or total cations; mineral soil: decreased exchangeable Na, no change in pH or exchangeable cations	Hazlett et al. (2011), Morrison and Foster (2001)
Central White Mountains, New Hampshire	15	samples from 13 plots; surface soil sampled by horizon (Oi, Oe, Oa, and A) using a 10- by 10-cm template (10 subsamples per plot)	organic matter mass, exchangeable Ca and Al, and acid-extractable Ca and Al	no change within a given horizon	Yanai et al. (2005)
Temiscaming, Quebec	11	initial sampling of the first 10 cm of the B horizon at 60-cm intervals along a single 16.5-m trench; second sampling along two trenches on each side of the former one	pH, total C and N, exchangeable cations	for one of two lines resampled, decreases in pH, C, and exchangeable Ca and Mg; no differences with resampling a second line	Godbout and Brown (1995)
Hermine watershed, Quebec	9	yearly samples from nine plots, composited from 16 subsamples of the forest floor and top 10 cm of the B horizon, taken with an auger	total C, exchangeable cations, extractable SO_4	decreased conc. of exchangeable Ca and Mg in forest floor, and exchangeable Mg in mineral soil, increased exchangeable Al in forest floor	Courchesne et al. (2005)
Acid Rain National Early Warning System, Canada	5	F (Oe), A and B horizons, five stations sampled at each of 103 plots across the country	pH, exchangeable Ca, Mg, Na	no consistent change in pH, increases and decreases in exchangeable cations for forest floor and mineral soil in relation to deposition zone	Morrison et al. (1996)
Allegheny National Forest, Pennsylvania	3	samples from four subplots at four sites; 10 subsamples each year using a 10- by 10-cm pin block, sampling by horizon down to the B horizon	pH, total C, and exchangeable cations	no linear trend in the measured variables	Bailey et al. (2005)
Great Smoky Mountains National Park and Gatlinburg, Tennessee	0.6	samples from the same pit at each site taken in April, July, and October, sampled by horizon in triplicate	exchangeable P and base cations, extractable NO_3–N and NH_4–N	decreases in NO_3–N and NH_4–N in surface horizons; increases in exchangeable P, Ca, and Mg in surface A horizon, decreases in subsurface horizons	Kelly and Mays (1999)
Hermine watershed, Quebec	0.5	samples from nine plots every 2 mo May–October for 4 yr, composited from 16 subsamples of forest floor and top 10 cm of B horizon with auger	total C, exchangeable cations, extractable SO_4	decreasing conc. in exchangeable Ca, Mg, K, and Mn saturation; increases in exchangeable Al saturation in B horizon	Courchesne et al. (2005)

preliminary data set to develop 95% confidence intervals for the standard error. This information can then be used to determine a sample size that will be sufficient to detect a specified magnitude of change regardless of variation in the standard error (Johnson et al., 1990).

Paired sampling within an experimental unit enables replication to be reduced, but variability within this unit can still substantially reduce the chances of observing a statistically significant change. Therefore, experimental units should be selected to encompass areas that are as homogeneous as possible in terms of topographic features, landscape position (such as crest, backslope, footslope, or toeslope), and understory and overstory vegetation (Hazlett et al., 2011). In some landscapes, pit and mound microtopography can result in large variations in hydrologic conditions and litter accumulation that can affect soil chemical measurements on the scale of a few meters (Hazlett et al., 2011), whereas uniformly level topography can result in relatively small amounts of variability in soil chemical measurements across this scale (Lawrence et al., 2005).

The time interval between samplings is not a constraint as long as the CRT of the soil property falls within the sampling interval (Table 1) and the magnitude of change can be discerned from spatial variability with the selected sampling design. Interpreting the cause of a measured change, however, is dependent on the time scale across which the causal agent of interest varies. The sampling interval should therefore be designed to address the time scale across which that factor varies, particularly when comparing data between two points in time. A detailed theoretical analysis by Brus and de Gruijter (2011) demonstrated how various sampling designs affect both the status (mean value at a point in time) and trend detection; these two aims need to be prioritized for an efficient space–time design that achieves the goals of the study.

Studies reported in Table 2 show that changes in exchangeable cation concentrations were detected across intervals ranging from 0.5 to 74 yr. These studies included the resampling of Morrison et al. (1996) that reported changes in Ca^{2+}, Mg^{2+}, and K^+ concentrations after a 5-yr interval. The primary goal of the Morrison et al. (1996) study, however, was to relate the effects of acidic deposition to exchangeable base cation concentrations, and variations in acidic deposition during the 5 yr were not sufficiently consistent among sites to relate acidic deposition effects to the measured soil changes. It was concluded that repeated samplings across longer time intervals would be needed to evaluate the role of acidic deposition in changing soil properties. Nevertheless, where sampling is repeated with time, shortening time intervals will decrease the length of time needed to detect change (Helsel and Hirsch, 1992).

The Use of Archived Samples

Maintaining consistent analytical methodology is crucial in any long-term monitoring program that requires collection and analysis of samples. Any change in methods of sample handling or analysis can introduce bias that could be interpreted as a change with time. The processing and analysis of samples cannot be assumed to be the same for each collection even if the laboratory and personnel are the same. The longer the time interval between collections, the greater the likelihood that methodological changes will be introduced. The possibility of analytical bias can

be assessed if samples from the previous collection are available for reanalysis. By reanalyzing the original samples with the recently collected samples, analytical consistency can be maintained.

The possibility exists, however, that archived samples changed during storage. Comparison between results obtained from reanalysis of archived samples and results from the original analysis enables possible storage effects to be assessed, but only if sample handling and analytical procedures are well reproduced. Therefore, full documentation of original handling and analysis methods, as well as the resultant data, should accompany archived soil samples.

Results of the reanalysis of air-dried soil stored for up to three decades have shown little or no changes in exchangeable cations (Bailey et al., 2005; Hazlett et al., 2011; Johnson et al., 2008a; Lawrence et al., 2012), organic C (Hazlett et al., 2011; Lawrence et al., 2012), organic matter (Bedison et al., 2010; Lawrence et al., 2012), and total N (Lawrence et al., 2012). Remeasurement of pH on air-dried samples, however, has shown that storage for one to three decades can cause a decrease in pH of up to 0.3 pH units (Bailey et al., 2005; Johnson et al., 2008a; Lawrence et al., 2012).

Reanalysis of all archived samples provides the most data to evaluate changes in methods or possible storage effects, but reanalysis of a subset of samples may be sufficient to verify quality assurance issues with previous data. Archived samples should be preserved to as great an extent as possible to enable verification for future resampling and also to provide an opportunity to analyze for constituents not previously measured. Technological advancements are likely to enhance analytical capabilities in the future that will further the value of archived soil samples.

Soil Monitoring Networks

Inclusion of soil in forest inventory and monitoring programs in the United States and Canada is relatively new (Table 3). The first program of this type was the Canadian Forest Service Acid Rain National Early Warning System, established in 1984 (D'Eon and Power, 1989). As part of a program developed to monitor forest vegetation and soils for possible changes caused by air pollution, complete profile sampling was conducted at 103 permanent sample plots (0.04 ha) in 1985 across a range of atmospheric deposition levels (Hall and Addison, 1991). In 1990 and 1995, sampling of the F (Oe), A, and B horizons was repeated (D'Eon et al., 1994), but sampling was suspended after 1995. Only two other monitoring networks that incorporate soil sampling were begun before 2000: the Forest Ecosystem Study and Monitoring Network in Quebec (Gagnon et al., 1994) and the U.S. Forest Service Forest Inventory and Analysis Program, which has established permanent plots throughout the United States that are sampled on a 5-yr rotating schedule (O'Neill et al., 2005).

Other nationwide, regional, state, and provincial programs have been started since 2000, as soil monitoring for environmental assessment has grown in acceptance (Table 3). This includes the Critical Zone Exploration Network, a consortium of field observatories that have been established to study changes in the Earth's surface in response to anthropogenic perturbation (http://www.czen.org/; accessed 12 Dec 2012). Most recently, the International Soil Carbon Network (http://www.fluxdata.org/nscn/SitePages/Home.aspx; accessed 11 Dec 2012) was formed through a multi-institutional partnership

Table 3. North American forest monitoring programs that explicitly include soil monitoring.

Country	Program title	Primary management	Dates	Purpose	Reference
Canada	Acid Rain National Early Warning System	Natural Resources Canada	1984–1996	to monitor vegetation and soils to detect long-term changes attributable to air pollutants in representative forest ecosystems	D'Eon et al. (1994)
Quebec, Canada	Forest Ecosystem Study and Monitoring Network	Quebec Ministry of Natural Resources and Wildlife	1988–	to detect early changes in forest ecosystem components (atmosphere, vegetation, soils), their causes, and consequences for ecosystem functions	Gagnon et al. (1994)
United States	Forest Health Monitoring/Forest Inventory and Analysis Program	U.S. Forest Service	1990–	to determine the status, change, and trends in indicators of forest health	O'Neill et al. (2005)
Alberta, Canada	Long-Term Soil and Vegetation Plots Established in the Oil Sands Region	Syncrude Canada, Suncor Energy, Albian Sands, Cumulative Environmental Management Association	2000–	to refine understanding of natural ecosystems and evolution of reclaimed ecosystems	Cumulative Environmental Management Association (2006)
Vermont	The 200 Year Soil Monitoring Project	Vermont Monitoring Cooperative in collaboration with the NRCS and U.S. Forest Service and Univ. of Vermont	2001–	to establish long-term replicated plots in Vermont's unmanaged forests to detect environmental change	Villars et al. (2013)
United States	Vital Signs Inventory and Monitoring Program	National Park Service, organized into 32 ecoregional networks of parks	2002–	to inventory and monitor the natural resources under National Park Service stewardship to determine their nature and status; soils are one of 12 primary inventories	Fancy et al. (2009)
Canada	National Forest Inventory	Natural Resources Canada, Parks Canada, Ministries of Natural Resources of 12 provinces/territories	2004–	to assess and monitor the extent, state, and sustainable development of Canada's forests	Gillis et al. (2005)

to develop a publicly accessible soil database created through voluntary data contributions. The approaches used by these programs vary widely with respect to sampling design, collection and analysis methods, and sampling frequency. Some programs are developed around uniform protocols applied throughout a centrally managed program, while others are designed to capture data distributed among various programs and research groups to enhance data accessibility.

Soil Sampling Techniques

Soil Pit Excavation

Whenever possible, collection of soil samples should be done following procedures that will enable resampling in the future with consistent methods (Table 4). This includes selecting a sampling area within which slope, drainage, and vegetation are as homogeneous as possible and variations in microtopography are minimal (Hazlett et al., 2011). The area of sample collection should be generally representative of the ecosystem or landscape of interest, however, and large enough to be resampled multiple times. Note that there is no standard depth for sampling an individual soil because soil depth can be highly variable within and across landscapes. Sampling depth is therefore operationally defined by the protocols of a particular program or project-specific research objectives and is usually tailored to the soil being studied. Excavating pits to enable collection of soil from the profile face is a common approach used in resampling studies because soil characteristics (horizon limits, redox conditions, stoniness, rockiness, root abundance and depth, etc.) are best observed with a fully exposed pit face (Schoeneberger et al., 2002). Furthermore, it is often the only practical way to collect samples from very stony soils that are common in the northeastern United States, eastern Canada, and other glaciated forest regions of North America.

Table 4. Advantages and disadvantages of soil sampling techniques.

Sampling technique	Advantages	Disadvantages
From soil profiles		
Profile face	easy to precisely delimit soil horizons and to sample every genetic horizon or by depth intervals	labor intensive, difficult to make many replicates for controlling for spatial variability
Quantitative profile	best estimation of soil bulk density by horizon or depth intervals and of root biomass and stoniness	very labor intensive, difficult to make many replicates for controlling for spatial variability
Monolith	unaltered soil and horizon boundaries, suitable for long-term archiving as its future sampling can be adjusted to later needs	labor intensive, needs proper archiving and accompanying metadata, difficult to make many replicates for controlling for spatial variability
Coring samplers		
Auger	easy to manipulate and make composite samples from many subsamples, reproducible, to make composite sample from many subsamples, best suited for sampling a given diagnostic horizon or the mineral soil by depth intervals	difficult to sample by genetic horizons, not reliable for assessing soil bulk density and stoniness
Volumetric or punch-core sampler	easy to manipulate, reproducible, good estimation of soil bulk density	difficult to use in stony soils, not as efficient as the auger for making composite samples

Differences in how profiles are interpreted by investigators can introduce a sampling bias that suggests artifactual changes in soils (Lawrence et al., 2012). Therefore, in addition to thorough documentation of how samples are collected, a full description of the profile using methods such as those followed by the NRCS (Schoeneberger et al., 2002) should be included to help maintain consistency between repeated samplings.

Samples can be collected and analyzed from selected horizons or depth intervals or a combination of both as prescribed by clearly defined protocols. The volume of soil collected is determined by that required for the intended physical, chemical, or biological analyses, plus additional sample to be archived for analysis in the future. Variability that occurs three dimensionally within horizons or defined depth intervals results in sample variability that can be reduced by increasing the volume of soil collected and by homogenizing the sample before subsampling for analysis. Soil should be collected from the full vertical thickness of the horizon or depth interval if possible. If the horizon thickness is >20 cm, equal volumes of soil may need to be collected at multiple depth intervals within the horizon for an overall horizon characterization. Some horizons may be too thin to sample without including material from horizons above or below. This is of particular concern when sampling thin E, Bh, or Bt horizons where adjacent abrupt boundaries can represent transitions to dramatically different soil materials. Sampling can be further complicated if diffuse horizon boundaries reduce the thickness of the layer that can be sampled.

Combining samples from different depth intervals or horizons (by equal volume when possible) reduces the number of samples to be analyzed but also reduces information on profile variability and reduces sensitivity to change detection. Nevertheless, in vertical sections of the profile with highly variable horizonation, collection of a depth increment that includes multiple horizons is more readily duplicated in a repeated sampling and is therefore more likely to detect possible changes with time than a detailed sampling approach that is heavily dependent on how the profile is interpreted by the collector. This approach was used, for example, in resampling of the upper 10 cm of B horizons in northeastern red spruce (*Picea rubens* Sarg.) stands that included varying expressions of Bh, Bhs, and Bs horizons (Lawrence et al., 2012).

If measurements of the total mass of an element in the mineral soil are needed, quantitative pit excavation is the most direct approach (Fernandez et al., 1993, 2003; Hamburg, 1984; Huntington, 1988). To remove measured volumes of soil, a square frame (usually 0.5 m^2) is secured to the soil surface to provide a guide for excavating material (soil, coarse roots, and coarse rock fragments) by horizon or depth interval. By keeping the pit walls perpendicular to the frame, the total volume excavated can be measured. Soil, coarse rock, and coarse roots are weighed separately, and the volume of the rock fragments is calculated by assuming a constant density. Horizons or depth intervals of fine earth fractions are homogenized individually and subsampled to determine chemical concentrations. Fine earth is typically defined as mineral soil material passing through a 2-mm-diameter mesh sieve, but 2- to 6-mm sieve sizes are commonly used for organic horizons. This technique permits more precise measurement of rock volume, soil bulk density, and root biomass than estimates made from the pit face. Quantitative pit sampling is more likely to mix soil from layers (or horizons)

above or below the layer of interest, however, because the entire layer within the frame is collected while maintaining the irregular surface that defines the lower boundary of this layer. Face sampling can be done more discretely because the sample can be removed from areas of the face where collection is not impeded by rocks or roots or, in the case of horizon sampling, the horizon interval is sufficiently thick to avoid including material from horizons above or below. Quantitative pit sampling is time consuming and labor intensive, which tends to limit the number of replicated pits. Excavation of each pit disturbs an area of several square meters, so a relatively large homogeneous area needs to be available for future resampling. The number of pits needed to adequately characterize the variability within a plot or watershed may prove unfeasible.

Collection of intact soil profiles, referred to as monoliths, has been done to display soils for pedological study and educational purposes in Europe, North America, and elsewhere. A slice of the profile, several centimeters thick, is removed from the pit face down to a depth of approximately 1 m. Collection of monoliths can be complicated by high coarse fragment content, but by keeping the profile intact, the features are preserved for viewing. Recent work in Russia has also demonstrated the value of monoliths in repeated sampling studies (Lapenis et al., 2004; Lawrence et al., 2005). The opportunity to combine the viewing and sampling of an archived monolith collected years in the past with the current resampling of that same soil in the field helps to ensure consistency between the two samplings. To date, we are not aware of the use of monoliths for repeated soil sampling in North America.

Soil Coring

In many instances, sampling with punch tubes, cores, or augers provides an efficient means to collect individual samples and also to create composite samples from multiple cores to effectively address spatial variability. Samples are removed from the soil without mixing so that the horizons remain in their original positions and discrete horizons or depth increments can be separated for analysis. Multiple cores can be easily collected within a certain landscape unit to characterize the soil within that unit (Fernandez, 1988). Some samplers can be effective for sampling both the Oe and Oa horizons of the forest floor as well as the mineral soil, although sampling of some soil types can compress the collected soil, which makes them ineffective for bulk density measurements or defining the depth interval of the collected sample. Cores collected with vertical sampling enable horizons to be separated more easily than lateral coring into the pit face because the depth of horizon boundaries varies across the length of the corer. Lateral cores can intersect soil from additional horizons, resulting in longitudinal horizon boundaries within the core that can be difficult to separate. Vertical cores provide stacked horizons that can be easily segmented after extraction.

Soil coring is generally best suited for fine-textured, non-stony soils or surficial organic horizons; however, coring in rocky soils can also be accomplished with a diamond-tipped coring bit driven by a rotary two-cycle engine as described by Rau et al. (2011). This corer can provide an estimate of coarse fragments in the soil because it can drill through rocks and was found to provide suitable estimates of soil C and N content (Rau et al., 2011); however, the dust resulting from rock drilling could potentially contaminate

measurements of exchangeable cations (Levine et al., 2012) or other soil properties.

Influential Soil Remeasurement Studies

Interest in soil change in nonagricultural systems developed in large part from concerns about the effects of acidic deposition on soil pools of exchangeable bases (Cowling and Dochinger, 1980). Related concerns also arose about the effects of forest harvesting on Ca availability in soil (Federer et al., 1989) and the effects of heavy metal accumulations in forest soils (Kaste et al., 2006). The utility of soil resampling in a forested setting was first shown by a long-term soil experiment designed to assess changes in soils resulting from aggradation of a pine forest planted on soil with a long agricultural history. The following summarizes this experiment plus three resampling studies that measured soil changes related to acidic deposition effects, atmospheric deposition of heavy metals, and two studies in which changes were not detected. These studies all played important roles in defining the value of soil resampling in the study of environmental change and provided important information on methods that can be used for measuring change.

The Calhoun Long-Term Soil Experiment

At the Calhoun Experimental Forest in the Southern Piedmont of South Carolina, Dr. Lou Metz and Dr. Carol G. Wells of the U.S. Forest Service initiated a LTSE in the 1950s (Metz, 1958) to learn how reforestation altered soil and ecosystem biogeochemistry on long-cultivated lands. Loblolly pine (*Pinus taeda* L.) seedlings were planted on the coarse-textured Piedmont soils in 1956 to 1957 following a 2-yr fallow that was preceded by long-term cotton (*Gossypium hirsutum* L.) cultivation. The sampling protocols initiated at the experiment's establishment have been followed for >50 yr and have proven instrumental to the success of the study. Chief among these are the resampling and archiving about every 5 yr of mineral soils to the 60-cm depth in four layers (0–7.5, 7.5–15, 15–35, and 35–60 cm) on 16, 0.1-ha plots.

During the 1980s, the ecological impacts of acidic deposition were of topical interest and controversial, with much of the uncertainty attributable to the notable absence of the long-term research needed to provide direct observations of soil acidification. Computer simulations of soil acidity were useful in identifying key processes but were no substitute for direct observation (Binkley et al., 1989; Richter et al., 1994). The Calhoun LTSE's direct evidence for significant acidification (updated in Fig. 1a) came to be featured in national assessments of acid rain and air pollution (Richter, 1991; Richter and Markewitz, 1995).
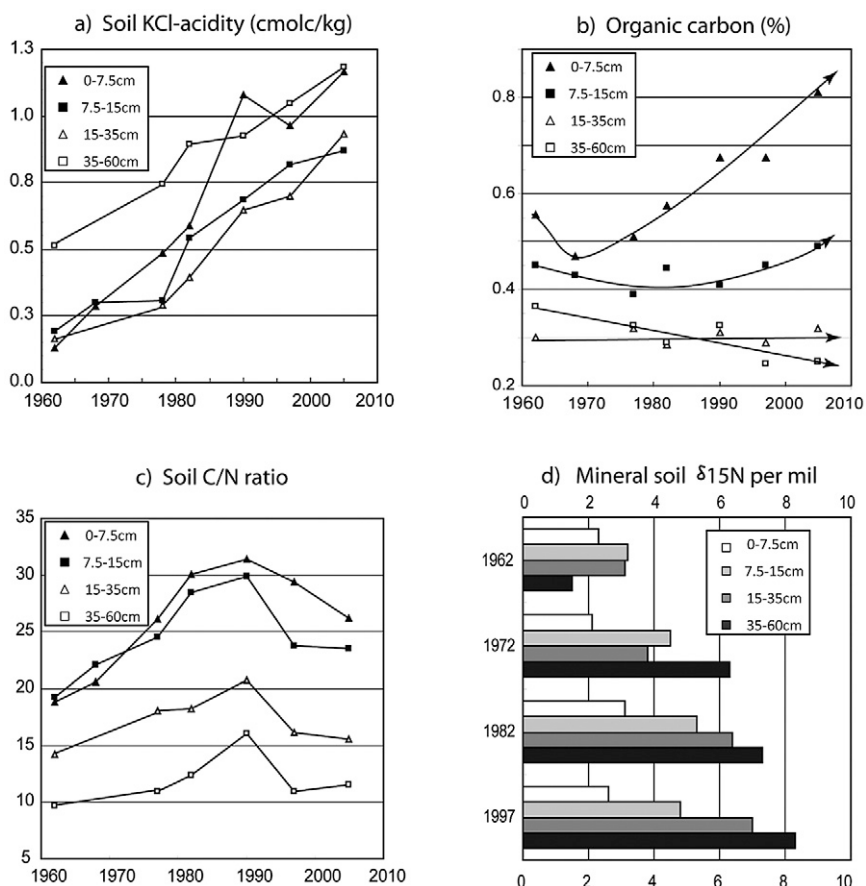

Fig. 1. Samples from the Calhoun Experimental Forest's permanent plots quantify (a) acidification, (b) C accrual and loss, and (c,d,) changes in the quality of organic matter (Richter et al., 1994, 1999, 2000; Billings and Richter, 2006).

The results indicated that Calhoun soils had acidified significantly since the 1960s (Fig. 1a), decreasing more than a full pH unit in surficial soil layers. Up to 40% of the directly observed increases in soil acidification were attributable to air pollution's acid influx (Markewitz et al., 1998). The Calhoun LTSE continues to be one of the few ecosystem experiments that can make such estimates. In addition to showing how and why forest growth acidifies soil, the experiment also quantified how the growing forest altered soil C and N over a period of decades (Harrison et al., 1995; Richter et al., 1999).

National and international demands for data on C gains and losses from ecosystems have made research sites such as the Calhoun LTSE invaluable for scientists, policy analysts, and the general public. The Calhoun study provides direct observations across five decades of C sequestration in above- and belowground ecosystem components: plant biomass, forest floor, and mineral soil. The depletion of soil organic matter from decades of cotton cultivation has begun to reverse through C inputs from the aggrading forest (Fig. 1b). The Calhoun forest as a whole has been a strong sink for atmospheric CO_2, sequestering C during the first four decades at rates in excess of $400 \ g \ m^{-2} \ yr^{-1}$ (Richter et al., 1999). Tree biomass accounted for 80% of the ecosystem C sink, followed by the forest floor. Belowground, gains in mineral soil C have accounted for small fractions of the ecosystem's total C sequestration. These data clearly demonstrate that it would take many more decades for the forest to recover the soil C lost during cultivation.

The availability of N also progressively decreased to levels of acute deficiency as the forest rapidly grew during its first three decades (Richter et al., 2000). During four decades, tree biomass and the forest floor accumulated >800 kg ha^{-1} of N, nearly all of which has been transferred from the mineral soil into these aboveground components. This large transfer of N caused substantial depth-dependent changes in the quality of soil organic matter, a pattern reflected by trends in the C/N ratio and δ^{15}N shown in Fig. 1c and 1d (Billings and Richter, 2006).

This experiment has been groundbreaking in demonstrating both the dynamic nature of soils on a decadal time scale and the value of soil resampling to provide direct information about rates of change driven by forest regrowth in an era of air pollution. Much of the success of this experiment can be attributed to the foresight of the original investigators, who recognized the importance of a repeatable long-term sampling design, well-documented sampling methods that could be readily duplicated, and the archiving of samples to ensure analytical consistency.

Fig. 2. Concentrations of 0.2 mol L^{-1} HCl extractable Ca in 1930 to 1932, 1984, and 2004 for organic horizons sampled at the Heimburger plots (Johnson et al., 2008a). Values identified by the same letter are not significantly different ($P < 0.05$); ODW = oven-dry weight.

Resampling of the Heimburger Plots

Carl C. Heimburger, as a Ph.D. student at Cornell University, conducted some of the first research on forest soils in North America through his sampling of soils in the Adirondack region of New York in the early 1930s. This work also provided some of the few data on forest soil chemistry in North America to predate the onset of acidic deposition. He dug soil pits, measured horizons, and used the standard analytical methods of his era to measure organic matter content (by loss-on-ignition), pH (by quinhydrone electrode), and 0.2 mol L^{-1} HCl extractable Ca (measured using a Ca-oxalate titration) in organic and mineral horizons.

In 1984, Andersen (1988) established 48 permanent plots in areas that matched the site descriptions of Heimburger, plus 16 plots in an experimental forest that had been sampled by Heimburger (1933). The set of 48 plots were located in high-elevation stands dominated by red spruce and balsam fir [*Abies balsamea* (L.) Mill.] and lower elevation stands dominated by white pine (*Pinus strobus* L.) or red pine (*Pinus resinosa* Ait.) or by northern hardwoods, principally sugar maple (*Acer saccharum* Marshall), American beech (*Fagus grandifolia* Ehrh.), and yellow birch (*Betula alleghaniensis* Britton). The 16 plots near Newcomb, NY, resampled in 1986, were all mixed stands containing northern hardwoods, spruce, and eastern hemlock [*Tsuga canadensis* (L.) Carrière].

Organic and mineral horizons were sampled at those sites in 1984, processed and analyzed following Heimburger's original methods, and archived. Andersen (1988) also showed that the results obtained using Heimburger's analytical methods were the same as those obtained using mid-1980s analytical techniques (electrometric pH and Ca in dilute HCl extracts measured by atomic absorpti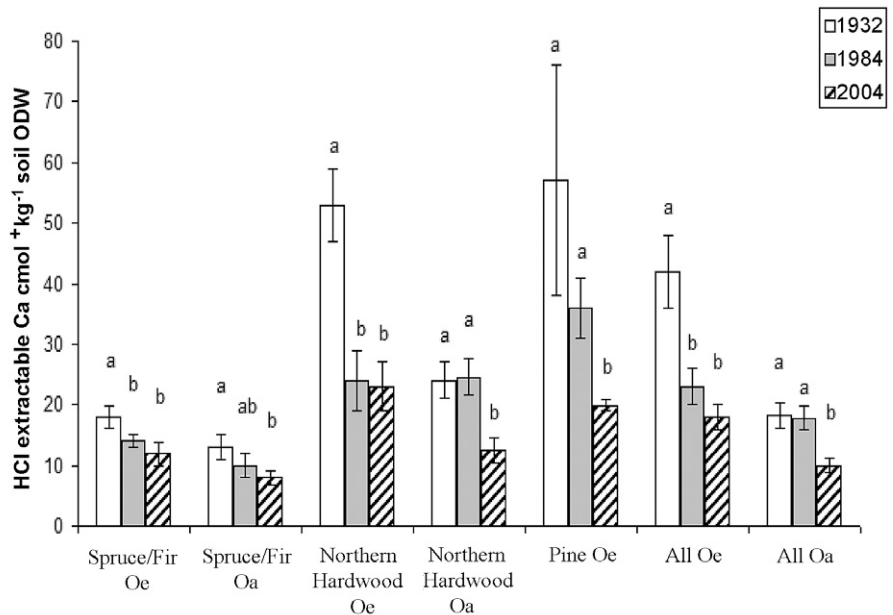on spectroscopy). Differences in methods and a lack of archived soil added to the challenge of this analysis, but the principal findings showed that (i) pH and dilute-acid extractable Ca concentrations decreased ($P < 0.05$) in Oe and Oa horizons, (ii) extractable Ca concentrations decreased ($P < 0.01$) in E horizons but not in B horizons, and (iii) extractable Ca loss from the Newcomb soils was statistically significant ($P < 0.05$) and approximately equal to the Ca in wood added by the living trees between 1932 and 1986 (Andersen, 1988; Johnson et al., 1994).

Additional sampling of the forest floor and mineral soil from 2004 to 2006 enabled dilute-acid extractable Ca concentrations to be compared on three dates spanning seven decades (1930–1932, 1984, and 2004–2006) and 1 mol L^{-1} NH$_4$Cl extractable Ca and Al concentrations to be compared for samples collected in 1984 and 2004 to 2006 (Bedison and Johnson, 2010; Johnson et al., 2008a). Over the >70-yr interval, HCl-extractable Ca concentrations in the Oe and Oa horizons decreased by approximately 50% in northern hardwood and high-elevation spruce–fir forests and 60% in pine-dominated stands ($P < 0. 05$; Fig. 2). Between 1984 and 2004 to 2006, HCl-extractable Ca concentrations decreased significantly in organic horizons when the three forest types were grouped ($P < 0.05$), indicating continued Ca loss from these soils in spite of declines in acidic deposition during this period. In addition, Ca concentrations and content decreased significantly in the upper 20 cm of the mineral soil while showing no change at depths >20 cm.

Data measured by NH$_4$Cl extraction and grouped by forest type also showed decreases in Ca concentrations in the forest floor between 1984 and 2004 to 2006, although the differences were only significant ($P < 0. 05$) for the Oa horizon in spruce–fir stands and the Oe horizon in the pine stands. Significant increases ($P < 0. 05$) in exchangeable Al were also observed in all forest types in the Oe horizon. Estimates of the forest floor Ca pool indicated annual losses averaging 0.5 to 2.0% per year depending on vegetation type, time period (1932–1984 or 1984–2004) and Ca fraction. High-elevation spruce–fir plots

had the highest estimated Ca loss rate between 1984 and 2004, ranging from 7.6 to 9.8 kg ha^{-1} yr^{-1}.

This study remains unique in North America because it provides the only data on soil change that predates the acid deposition era, considered to have begun after the economic depression of the 1930s. This was also the first study to show substantial depletion of soil Ca in a forest setting that had no previous history of agricultural manipulation and liming. Furthermore, comparisons between 1984 and 2004 indicated decreases in NH$_4$Cl-extractable Ca despite decreases in acidic deposition in the study region. The results of this study played a key role in identifying the problem of soil Ca depletion that resulted from acidic deposition.

Resampling in the Allegheny National Forest after 30 Years

In 1967, complete soil profiles were sampled at a number of locations in Warren County in northwestern Pennsylvania as a routine part of county soil surveys conducted by the Soil Conservation Service (now the NRCS). Western Pennsylvania received some of the highest levels of acidic deposition in the country during the 1980s and 1990s (Driscoll et al., 2001). These samples were chemically analyzed and archived in the Pennsylvania State University Soil Characterization Laboratory, which provided an opportunity for resampling to evaluate soil change and storage effects. These types of county soil surveys by the NRCS conducted routinely, nationwide, may provide a large, and largely untapped, opportunity for evaluating soil change, particularly in cases such as this where subsamples are archived (Ciolkosz et al., 1970). In 1997, Bailey et al. (2005) accessed subsamples of these soils and relocated six of the original sampling sites on forested land using aerial photographs also stored in the archives. Four sites in the Allegheny National Forest had been undisturbed in the intervening 30 yr and were resampled following the original field protocols with the assistance of one of the original samplers, Dr. William Cunningham. Reanalysis of the archived subsamples showed little or no bias between laboratories, indicating that new analyses could be compared with the original analyses published by Ciolkosz et al. (1970).

To evaluate whether any differences between the 1967 and 1997 sampling pits were due to temporal change or spatial variability, four pits were dug and sampled in 1997, 10 m away from the original 1967 pit in each of the cardinal directions. Because horizon sequence and depth varied from pit to pit, temporal change in soil properties was assessed using index horizons, including the Oa/A, the uppermost subdivision of the B, and the horizon sample straddling the 50- and 100-cm depths, generally a lower B or BC horizon. To determine whether the difference between 1967 and 1997 results represented a long-term shift or interannual variability, surface horizon mass and chemistry were evaluated in 1997, 1998, and 1999 using quantitative pin-block samples.

Significant decreases ($P < 0.001$) were observed over the 30-yr interval in exchangeable Ca and Mg concentrations and pH at all depths when the four sites were grouped (Fig. 3). Exchangeable Al concentrations were higher in 1997 than in 1967 at all depths at all sites, although the differences were only significant ($P < 0.05$) in the Oa/A and upper B horizons. Short-term temporal changes, estimated by sampling the Oa/A horizon annually for 3 yr, were insignificant. This result suggested that the differences between 1967 and 1997 were part of a long-term trend rather than interannual variability. The 1967 exchangeable Ca and Mg levels were above the threshold values necessary for sugar maple health, as determined empirically by Bailey et al. (2004). In comparison, 1997 exchangeable Ca and Mg levels were below those threshold values. These results suggested a change in site suitability for sugar maple and were consistent with the widespread occurrence of sugar maple decline disease in the region during the 1980s and 1990s (Horsley et al., 2000). This was the first study to link changes in soils to changes in forest health, and the results were consistent with the research at the Heimburger plots (Bedison and Johnson, 2010; Johnson et al., 2008a) in showing a link between acidic deposition and depletion of soil Ca.

Resampling the Direct Delayed Response Project Sites across the U.S. Northeast after 17 Years

The Direct Delayed Response Project (DDRP), started in 1984 under the National Acid Precipitation Assessment Program, was designed to assess the effects of acidic deposition on surface waters in the eastern United States (Lee et al., 1989). In total, the DDRP sampled approximately 280 pedons and 1400 horizons within 145 watersheds (Adams et al., 1992) in three northeastern regions (Adirondack, Catskill/Pocono, and central New England and Maine). In 2001 and 2002, Warby et al. (2009) sampled the Oa horizon in 139 of these watersheds located in the northeastern United States. The data sets evaluated in that study were comprised of 75 samples from 47 watersheds for the 1984 survey and 55 samples from 55 watersheds for the 2001 to 2002 survey. The 2001 to 2002 survey did not sample at the same locations as the 1984 survey, although samples were collected in 24 watersheds in both 1984 and 2001 to 2002.

During the 17-yr interval, no changes were observed in regionally grouped data for effective cation exchange capacity, C, and N ($P > 0.05$); however, median exchangeable concentrations of Ca declined from 23.5 to 10.6 cmol$_c$ kg^{-1}, and median exchangeable Al increased from 8.8 to 21.3 cmol$_c$ kg^{-1} (Fig. 4). Changes in median exchangeable Mg and K were not significant, but median exchangeable Na decreased ($P < 0.05$). Median soil pH (in 0.01 mol L^{-1} CaCl$_2$) also decreased ($P < 0.05$) from 3.14 to 2.98 (Fig. 4).

The largest changes in soil acid–base properties occurred in central New England and Maine. Given that this region historically received lower acidic deposition rates than the Adirondack or Catskill/Pocono study regions (Ollinger et al., 1993), this finding was not expected. On the basis that emissions of SO$_2$ peaked in the United States in 1973 (Driscoll et al., 2001), the researchers hypothesized that the Adirondack and Catskill/Pocono regions had been substantially acidified before the 1984 DDRP study and therefore were less likely to show further changes, whereas the previously less impacted central New England and Maine region had more capacity for change between 1984 and 2001.

These results were highly significant because they were the first to show soil acidification throughout much of the northeastern United States. They also provided a large data set to
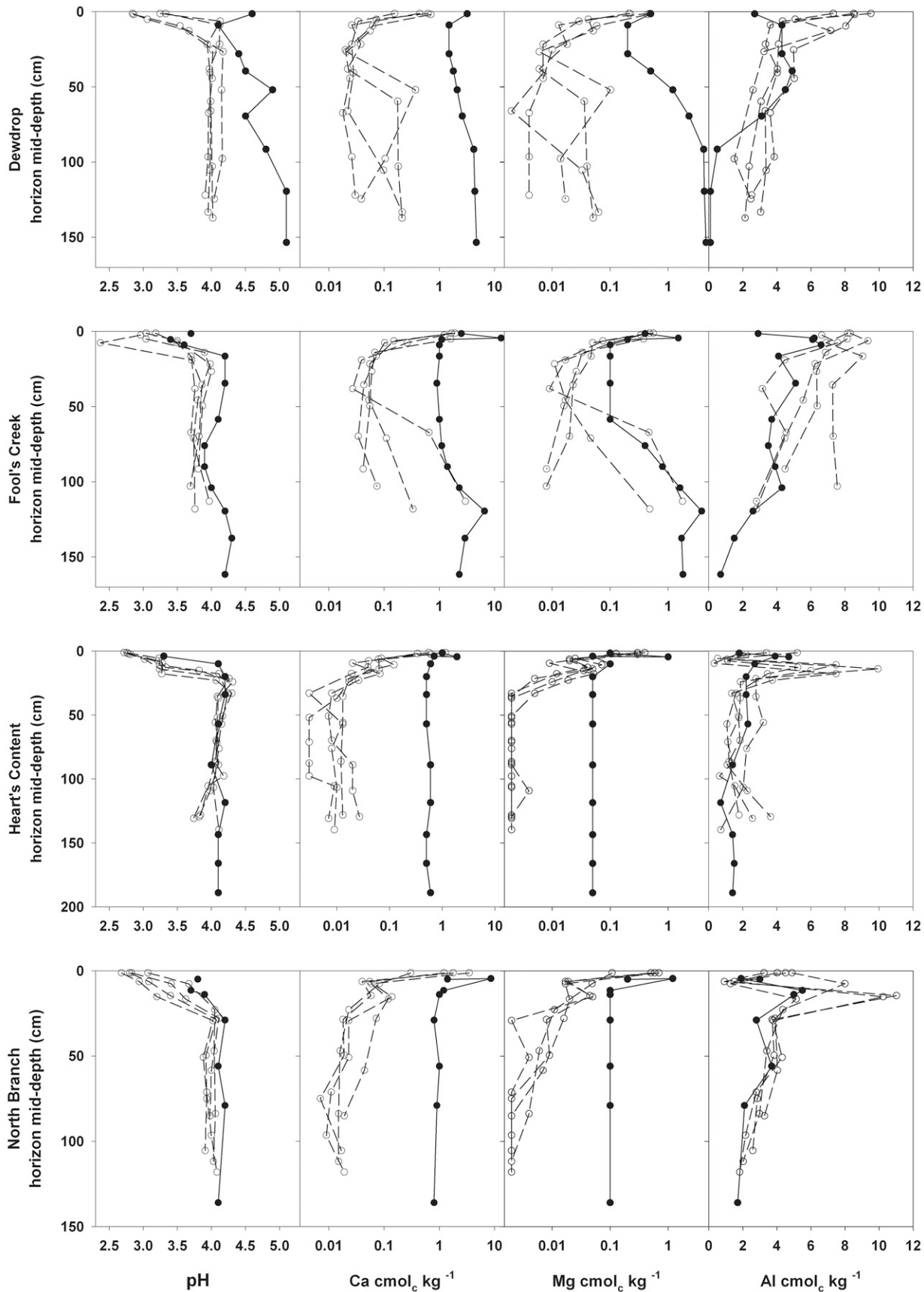
Fig. 3. Depth profiles of pH and exchangeable Ca, Mg, and Al at each of four study sites in western Pennsylvania (Bailey et al., 2005). The closed circles represent the data collected from the original pit dug in 1967. The open circles represent the data collected from four pits dug in 1997, located 10 m away from the original pit in each of the four cardinal directions. The dotted horizontal line shows the average depth of the top of the fragipan, where present. A fragipan was found at three of the five pits at Dewdrop, all five pits at Fool's Creek, and one of five pits at Heart's Content.
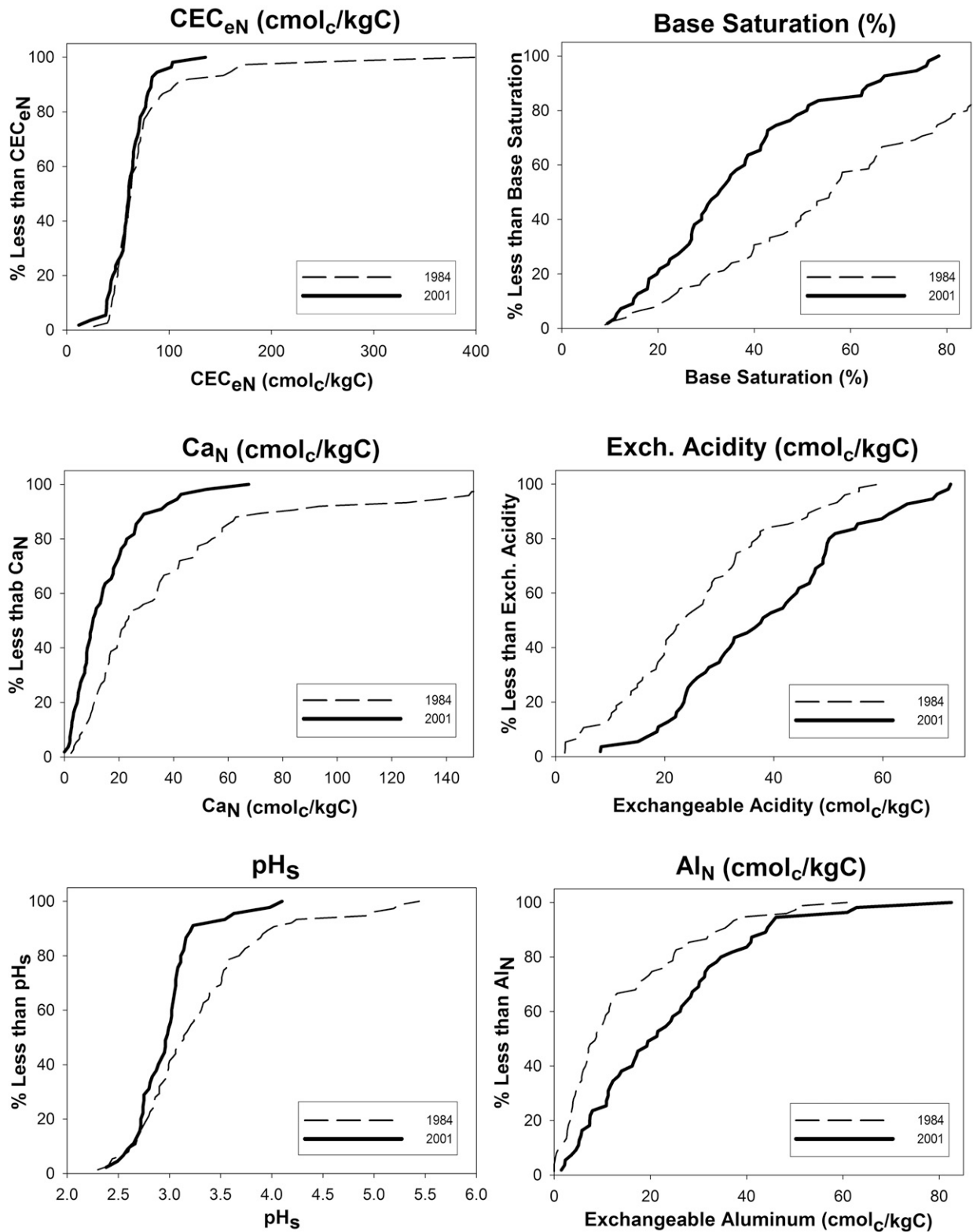
Fig. 4. Cumulative frequency diagrams for selected acid–base properties of organic horizons from the Direct Delayed Response Project (DDRP) across the northeastern United States in 1984 and 2001 (Warby et al., 2009); CEC_{eN} is effective cation-exchange capacity normalized for organic C concentration, Ca_N is Ca concentration normalized for organic C concentration, Exch. acidity is H plus Al, pH_s is pH measured in 0.01 mol L^{-1} $CaCl_2$, Al_N is Al concentration normalized for organic C concentration.

explain why increases in the acid-neutralizing capacity of surface waters were less than expected in response to the substantial declines in acidic deposition that occurred during the 1980s and 1990s. The results of Warby et al. (2009) also provided strong evidence to support the early identification by Lawrence et al.

(1995) of a mechanism for Oa-horizon Ca depletion by acidic deposition, and the Adirondack results of Johnson et al. (1994, 2008a) and Bedison et al. (2010) thereby played a significant role in establishing the significance of Ca depletion as an impact of acidic deposition.

Resampling Forested Soils in the Northeast to Detect Changes in Heavy Metal Content

Soils of the northeastern United States received considerable burdens of pollutant metals during the 20th century. Repeated quantitative measurements of soils for metal concentration (parts metal per mass soil) and amount (mass metal per area soil) has been a very useful approach for characterizing the accumulation, storage, and loss of metals from various soil horizons over time in this region. Because many metals bind strongly to organic matter, the metal content of the organic (O) horizon is a function of air quality, atmospheric scavenging and deposition, and the residence time of the metal in the layer. In the last half century, researchers have utilized repeated soil sampling across time intervals of 5 to 10 yr to quantify changes in atmospheric metal deposition rates and to model the transport and biogeochemistry of pollutant metals in soils in the northeastern United States.

Lead is perhaps the most widely dispersed trace metal in the environment (Galloway et al., 1982), and consequently, this metal has received the most attention in repeated soil sampling studies in the northeastern United States. Moreover, Pb is not significantly cycled by vegetation, so in the absence of pollution, O horizons would typically have low Pb and can thus be very sensitive indicators of deposition (Johnson et al., 1982). In 1978, Siccama and Smith (1978) resampled the O horizon from a stand of white pines in Massachusetts that had been previously sampled in 1962. By measuring the metal content of the soils collected at each time, they calculated a net increase of 30 mg Pb m^{-2} yr^{-1} in the O horizon during the 1962 to 1978 period and attributed this to the deposition of gasoline-derived Pb to the region. The amount of Cu and Zn in the forest floor did not change significantly during the same time interval. Since then, a number of repeated-measurement studies have shown that the Pb content and amounts in the O horizons of the northeastern United States reached a concentration maximum during the 1975 to 1985 time period and, in some cases, have decreased since the mid-1980s in response to changing atmospheric inputs and migration (Friedland et al., 1992; Johnson et al., 1995; Kaste et al., 2003; Miller and Friedland, 1994; Yanai et al., 2004). Other works have shown that levels of Cd, Hg, and Pb in the litter layer in forests of New England and Quebec have declined between 1979 and 1996, a trend that probably reflects improvements in industrial emission controls and air quality (Evans et al., 2005).

Whereas earlier works that repeatedly measured the metal content of soils from a particular location were focused on quantifying contaminant deposition, more recent research has used repeat soil sampling to quantify the residence time of metals in the soil profile (Johnson and Richter, 2010; Kaste et al., 2006) and to ultimately characterize the biogeochemical behavior of metals in the soil environment (Johnson et al., 1995; Stankwitz et al., 2012; Wang and Benoit, 1997). Miller and Friedland (1994) coupled a model of atmospheric Pb deposition to repeated measurements made in 1980 and 1990 of Pb in soils on Camels Hump Mountain in Vermont. Using this approach, they concluded that the residence time of Pb in the O horizon was 17 yr in the northern hardwood forest zone and 77 yr in the spruce–fir forest zone. They suggested that the Pb was migrating from the O horizon to the mineral soil bound to organic colloids and that the transport of Pb out of the O horizon was controlled by the turnover of organic matter. More recent measurements of Hg and Pb at Camels Hump Mountain and nearby areas have confirmed that metal concentrations and residence time in the O horizon is dependent on elevation and vegetation type (Kaste et al., 2006; Stankwitz et al., 2012).

Continued soil resampling at forested sites around North America will allow the further development and calibration of biogeochemical models that will strengthen our ability to predict the fate of pollutant metals in ecosystems.

Studies in which Soil Changes Were Not Detected

Archived samples were analyzed to evaluate changes in Ca and Mg concentrations and content of the forest floor of northeastern hardwood and softwood stands by resampling at time intervals ranging from 10 to 21 yr (Yanai et al., 1999). The general conclusion of this study was that little or no change in concentrations of the forest floor (combined Oi, Oe, and Oa horizons) occurred, although a decrease in exchangeable Ca concentrations in the Oa horizon at the Hubbard Brook Experimental Forest was observed from 1978 to 1997.

The Ca mass-balance study of Likens et al. (1996) suggested that peak Ca depletion occurred in the 1960s and early 1970s in this region, which would not have been shown in the Yanai results. The Yanai resampling was complicated, however, by high spatial variability and differences in field sampling techniques between the original collection and the resampling (Yanai et al., 1999). In soils of hardwood stands in New Hampshire, little difference was also found during 15 yr in exchangeable and extractable Ca, Mg, and Al concentrations in Oa horizons at six sites and in O horizons (combined Oe and Oa horizons) at 13 sites (Yanai et al., 1999). This study also estimated that a difference >50% would be needed to be statistically significant due to the large degree of within-site spatial variability. The results of Yanai et al. (1999) indicated that decadal changes in soil chemistry may not occur at all sites and may be difficult to detect in some soils due to natural variability and the difficulty of separating horizons during sampling.

In 2003 and 2005, Hazlett et al. (2011) resampled mineral soils initially sampled in 1986 as part of nutrient cycling research at the Turkey Lakes watershed (TLW) in northeastern Ontario. Genetic horizons were sampled from the total depth of the soil profile at seven plots with varied topographic conditions across the 10.5-km^2 watershed. Archived samples previously collected at these sites were available to assess the comparability of analytical results. Watershed-level analyses showed no significant changes during the 17- to 19-yr period in concentrations of exchangeable Ca, Mg, or K, whether expressed per soil mass or per organic matter mass. There was a decline in exchangeable Na concentrations in deeper horizons of the soil profile. There were also no changes observed in pH.

The lack of change in mineral soil Ca found in this study contrasts with the declines documented in the studies of Johnson et al. (2008b) and Bedison and Johnson (2010) during similar resampling periods, despite the high cation export rates of the TLW compared with other forested sites (Watmough et al., 2005). A confounding factor with the statistical analysis of this study was that the range of topographic positions and initial soil properties resulted in a large variation in paired plot differences across the site. Calculated minimum detectable differences

were large, indicating a need for greater sample size or better stratification of sampling locations to detect soil change with time. Notwithstanding these considerations, the TLW has much greater total soil base cation content than sites where cation declines have occurred. These results suggest that mineral weathering buffered the exchangeable base concentrations despite high leaching losses. These results reinforce the observation of Yanai et al. (1999) that decadal change may not occur at all sites and, furthermore, that inherent site characteristics can influence the potential for change.

Future Relevance of Soil Resampling

One of the most significant effects of human activities during what is now being referred to as the Anthropocene Era (Crutzen and Steffen, 2003) may be an acceleration of soil change and its accompanying challenge to sustainability. Repeated soil sampling therefore provides a key method being used by a growing number of scientists in North America for documenting environmental change. The viability of this approach has been demonstrated by studies that have measured statistically significant changes in soils during time intervals of <1 yr to approaching a century. Most of the repeated soil sampling studies conducted to date have relied on earlier soil sampling that had not been designed with future resampling in mind, the Calhoun LTSE and the Canadian Forest Service Acid Rain National Early Warning System being notable exceptions. With better awareness of the value of soil resampling, studies can be designed with a long-term perspective so that information can be efficiently obtained well into the future to address problems that have not yet surfaced. For example, whole-watershed manipulations were conducted to simulate the effects of acidic deposition to determine, among other things, if base cations were being depleted from the soil (Fernandez et al., 2003). Had soil monitoring programs been in place before the onset of high deposition levels, this question would have been answered decades sooner, with far less expense. Future changes in soils from unforeseen effects of environmental drivers such as deeper rooting under elevated atmospheric CO_2 concentrations (Iversen, 2010) will need to be documented.

The potential of repeated sampling can be limited by inadequate documentation of the sampling location and conditions at the time of sampling, insufficient replication, inconsistencies in the identification and collection of horizons, analytical inconsistencies among laboratories, and lack of sample archiving. These complications can be avoided in the future with informed research designs that utilize the appropriate methods. It is also important to note, however, that despite the limitations that can result from methods used in historical soil sampling, new opportunities to gain valuable information through repeated soil sampling continue to be identified. These include potential opportunities that can arise from national programs, such as the NRCS soil surveys, in situations where archived samples have been saved and methods can be replicated on sites with known land use histories.

As the value of repeated soil sampling continues to gain recognition, sampling designs are being developed that anticipate future resampling. Organizational efforts such as the Northeastern Soil Monitoring Cooperative need to be expanded to improve standardization and coordination for the development of spatial assessments of soil change. Ongoing, large-scale issues such as recovery from acidification, long-term N deposition, the effects of climate change, impacts from invasive species, and the increasing intensification of soil management all warrant the use of soil resampling as an essential tool for environmental monitoring and assessment.

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