

# Assessment of Methods for Soil Monitoring in the Adirondack Region of New York State

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# **Assessment of Methods for Soil Monitoring in the Adirondack Region of New York State**

*Final Report*

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## Notice

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# Abstract

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Repeated sampling to detect changes in forest soils was rarely used before 1990, but the value of soil monitoring in understanding environmental change is becoming well established. The growing number of resampling studies has shown that sampling designs and procedures must be adapted to the objectives of the monitoring program and the soils monitored. In the Adirondack region, current priorities include the response of soils to large increases, and more recently, large decreases in acidic deposition, and changes driven by trending climate such as altered pools of soil organic carbon, as well as other unforeseen factors that will occur in the future.

To improve methods and assess the feasibility of long-term soil monitoring in the Adirondack region, the United States Geological Survey (USGS) conducted a pilot project to evaluate a new sampling method for characterizing soils on a watershed basis. Results obtained with this new approach, referred to as the ADK (abbreviation for Adirondack) sampling method, was compared to methods used in previous sampling conducted in 2004 as part of the Western Adirondack Stream Survey (WASS), and also to previous high-replication pit sampling in the North and South Tributary watersheds of Buck Creek (North Buck and South Buck). The number of sampling locations and spatial distribution of sampling points within watersheds differed among the methods, although pit excavation was used to obtain samples in all cases. In addition, this investigation evaluated the use of small diameter corers as a means to measure forest floor mass with greater accuracy and precision than commonly used methods such as pit excavation.

Sufficient statistical power to detect ecologically relevant changes in upper profile horizons (Oe, Oa and upper 10 cm of the B) were achieved with the ADK sampling method that utilized 18 pit excavations per watershed. The sampling locations were organized within each watershed into three study areas (six sampling locations per study area) that represented the primary types of landscape within the watershed. Sampling at 18 locations per watershed was found to be nearly as effective at detecting changes as sampling at 28 locations per watershed. Numerous significant changes ( $P < 0.10$ ) were detected with both 18 and 28 sampling locations at sampling intervals of 12 to 16 years. The relation between soil data obtained with the ADK method and stream chemistry at the base of the watershed suggested that this approach adequately characterized soil variability within the watershed for the purpose of studying soil-stream linkages. Significant changes in upper B horizon calcium ( $P < 0.10$ )

and Oa horizon aluminum ( $P < 0.01$ ) were detected when the data from the four WASS watersheds were combined with the two Buck Creek watersheds, which suggested that there would be value in resampling other WASS watersheds previously sampled in 2004 to support a regional assessment.

Study results support small diameter cores as a useful method to monitor changes in the organic matter mass of the forest floor. This method showed high reproducibility in repeated sampling tests and lower spatial variability in sample data than traditional approaches when compared on a watershed basis.

Soil coring is also faster and requires less equipment than pit excavation methods, which makes it more conducive to sampling over large areas. However, organic matter mass of the forest floor determined by coring was consistently less than the values obtained by the ADK sampling method that used pit sampling and vertical horizon measurements, and also literature values of a previous Adirondack study that utilized pit sampling in which the entire horizon was collected over a measured area. However, a high correlation ( $R^2 = 0.87$ ) occurred between organic matter content (expressed as  $\text{Mg ha}^{-1}$ ) determined by coring and the ADK sampling method. Differing methods with regard to where samples could be collected, and how organic matter was collected for chemical analysis were the likely reasons for differences in quantification of forest floor organic mass.

Collection of forest floor cores in conjunction with the ADK method provides improved sensitivity in detecting changes in the forest floor in proximity of where full analyses of the soil profile are performed. This dual sampling approach represents an optimized method for measuring and understanding how Adirondack soils will change in the future.

## Keywords

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acid rain, acidic deposition, soil monitoring, soil sampling methods, forest floor, soil organic matter, soil chemistry, soil aluminum, soil calcium depletion, Adirondack region

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# Executive Summary

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Repeated sampling to detect changes in forest soils was rarely used before 1990, but the value of soil monitoring in understanding environmental change is becoming well established. The growing number of resampling studies has shown that sampling designs and procedures must be adapted to the objectives of the monitoring program and the soils monitored. In the Adirondack region, current priorities include the response of soils to large increases, and more recently, large decreases in acidic deposition, and changes driven by trending climate such as altered pools of soil organic carbon, as well as other unforeseen factors that will occur in the future.

To improve methods and assess the feasibility of long-term soil monitoring in the Adirondack region, the United States Geological Survey (USGS) conducted a pilot project to evaluate a new sampling method for characterizing soils on a watershed basis. Results obtained with this new approach, referred to as the ADK (abbreviation for Adirondack) sampling method, was compared to methods used in previous sampling conducted in 2004 as part of the Western Adirondack Stream Survey (WASS), and also to previous high-replication pit sampling in the North and South Tributary watersheds of Buck Creek (North Buck and South Buck). The number of sampling locations and spatial distribution of sampling points within watersheds differed among the methods, although pit excavation was used to obtain samples in all cases. In addition, this investigation evaluated the use of small diameter corers as a means to measure forest floor mass with greater accuracy and precision than commonly used methods such as pit excavation.

Sufficient statistical power to detect ecologically relevant changes in upper profile horizons (Oe, Oa and upper 10 cm of the B) were achieved with the ADK sampling method that utilized 18 pit excavations per watershed. The sampling locations were organized within each watershed into three study areas (six sampling locations per study area) that represented the primary types of landscape within the watershed. Sampling at 18 locations per watershed was found to be nearly as effective at detecting changes as sampling at 28 locations per watershed. Numerous significant changes ( $P < 0.10$ ) were detected with both 18 and 28 sampling locations at sampling intervals of 12 to 16 years. The relation between soil data obtained with the ADK method and stream chemistry at the base of the watershed suggested that this approach adequately characterized soil variability within the watershed for the purpose of studying soil-stream linkages. Significant changes in upper B horizon calcium ( $P < 0.10$ )

and Oa horizon aluminum ( $P < 0.01$ ) were detected when the data from the four WASS watersheds were combined with the two Buck Creek watersheds, which suggested that there would be value in resampling other WASS watersheds previously sampled in 2004 to support a regional assessment.

Study results support small diameter cores as a useful method to monitor changes in the organic matter mass of the forest floor. This method showed high reproducibility in repeated sampling tests and lower spatial variability in sample data than traditional approaches when compared on a watershed basis.

Soil coring is also faster and requires less equipment than pit excavation methods, which makes it more conducive to sampling over large areas. However, organic matter mass of the forest floor determined by coring was consistently less than the values obtained by the ADK sampling method that used pit sampling and vertical horizon measurements, and also literature values of a previous Adirondack study that utilized pit sampling in which the entire horizon was collected over a measured area. However, a high correlation ( $R^2 = 0.87$ ) occurred between organic matter content (expressed as  $\text{Mg ha}^{-1}$ ) determined by coring and the ADK sampling method. Differing methods with regard to where samples could be collected, and how organic matter was collected for chemical analysis were the likely reasons for differences in quantification of forest floor organic mass.

Collection of forest floor cores in conjunction with the ADK method provides improved sensitivity in detecting changes in the forest floor in proximity of where full analyses of the soil profile are performed. This dual sampling approach represents an optimized method for measuring and understanding how Adirondack soils will change in the future.

# 1 Introduction

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Over the past 100 years, the Adirondack region has experienced pronounced increases and decreases in atmospheric deposition (Fakhraei et al. 2014), and trends in various measures of climate are expected to strengthen (Hayhoe et al. 2007). Furthermore, the age of the Adirondack forest is increasing as harvesting intensity and frequency continues to decrease from the peak achieved in the early 20th century. Existing monitoring of lake and stream chemistry provide valuable information on how these changes are affecting key ecosystem components to inform policymaking and develop effective forest and watershed management plans. However, there is currently no program to monitor soil change even though soils play a key role in the health of both the terrestrial and aquatic components of the Adirondack ecosystem.

Historically, soil chemistry was assumed to change slowly over centuries or longer, but numerous studies have shown that important changes in forest soils can occur in as little as a decade, possibly less (Lawrence et al. 2013). For example, calcium, the primary cation for buffering soil acidity, and an essential nutrient for plants and animals, became depleted in Adirondack soils during the 20th century as a result of acidic deposition (Johnson et al. 2008). Although acidic deposition levels have decreased substantially, when and to what extent availability of calcium (Ca) will improve is uncertain, as are the effects of changing climate and a maturing forest on Ca and other elements such as magnesium (Mg) and potassium (K) that are both nutrients and provide acid buffering as well as sodium (Na) that provides acid buffering.

Soil organic carbon (SOC) is especially important to forest ecosystems for the role it plays in nutrient and moisture retention, nutrient cycling, and water quality. Furthermore, because the global SOC pool exceeds the atmospheric pool of carbon by a factor of about two (Lal 2004), the need to understand and quantify the role of soils in the sequestration of carbon has become an important element of climate change mitigation research. The potential for increasing the total carbon stored in soil is unclear (Amundson and Biardeau 2018), but the potential for climate warming to decrease the amount of carbon stored in soils has been identified in several analyses (Lal 2005, Ponge et al. 2011, Zhao et al. 2019). The high SOC content of Adirondack soils makes them vulnerable to this effect, and the large area covered by the Adirondack ecosystem makes New York State an important contributor to the total soil carbon stores of the United States. Federal land accounts for over 20% of SOC in the conterminous

48 states (Tan et al. 2015), giving national relevance to the organic-rich soils of the Adirondack Park, which cover an area similar to the largest National Forest outside of Alaska.<sup>1</sup> These factors, and the tendency of organic carbon to be stored in the soil for periods ranging from a few years to multiple centuries highlight the need for accurate monitoring of the Adirondack SOC pool, as well as other biogeochemical properties of soil.

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<sup>1</sup> USDA Forest Service: Humboldt-Toiyabe National Forest: <https://www.fs.usda.gov/htnf/>; accessed 5-29-2019.

## 2 Project Objective

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The use of repeated sampling to detect changes in forest soils dates back to the 1800s, but as a common practice is relatively new. However, examples of successful soil monitoring programs are growing in number (Lawrence et al. 2013, Lawrence et al. 2015, Mobley et al. 2019), and the few that have been maintained for extended periods have produced highly valuable information. This includes the continuous soil monitoring program conducted in the United States Forest Service Calhoun Experiment Forest in South Carolina, which is nearing their 60th anniversary in 2020. As new soil monitoring programs are started, progress in the standardization of field and laboratory methods is ongoing (Ross et al. 2015, Lawrence et al. 2016). This work has shown that sampling designs and procedures must be adapted to the objectives of the monitoring program and the soils monitored. In the Adirondack region, current priorities include the response of soils to substantial decreases in acidic deposition and changes driven by trending climate such as altered SOC pools. Other factors, currently unforeseen, will also likely impact the Adirondack ecosystem through changes in soil processes in the near future.

To improve methods and assess the feasibility of soil monitoring in the Adirondack region, the United States Geological Survey (USGS) conducted a pilot project that involved resampling of soils that had been originally sampled in 2004 as part of the Western Adirondack Stream Survey (WASS). The method of resampling, hereafter referred to as the ADK (abbreviation for Adirondack) sampling method was designed to improve detection of ecologically important soil changes over time intervals that are useful to land managers, policymakers and research scientists without excessive cost and effort. Data available through previous high-replication sampling in the north and south tributary watersheds of Buck Creek (North Buck and South Buck) were also utilized to test the ADK sampling method. In addition, this investigation evaluated sampling the forest floor from the surface with small diameter corers as a means to measure forest floor mass with greater accuracy and precision than commonly used methods such as pit excavation. Both the ADK sampling and forest floor coring were designed to provide soil data representative of the variability that occurs within small watersheds (less than approximately 50 ha), so that detected soil changes would provide information to help interpret changes in stream chemistry as well as vegetation. The overall goal of the pilot project was to determine if these two sampling methods would be appropriate for long-term soil monitoring of Adirondack soils. Results were evaluated by addressing the following specific questions related to the design used in this study.

## 2.1 Analysis Questions

1. What are the minimum detectable changes that can be determined with the sampling design of the ADK method, based on statistical power of 0.80 and  $P < 0.05$ , and how do these minimum values compare to those of the prior sampling design used in these watersheds?
2. How does minimum detectable change vary with sample size and paired versus unpaired sampling designs in these soils?
3. How effective is the ADK sampling replication of  $n=18$  in detecting real changes in Adirondack soils?
4. Does spatial variability in soil chemistry change with sampling depth?
5. Did the ADK sampling method provide soil data that could be related to stream chemistry?
6. Did resampling with the ADK sampling method reveal soil changes in watersheds previously sampled with the WASS sampling method?
7. How reproducible are the forest floor data determined with small diameter corers?
8. How does the variability and quantification of forest floor mass measurements determined by coring compare on a watershed basis to those determined through pit excavation?

## 3 Methods

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### 3.1 Pit Sampling Design

Four watersheds,<sup>2</sup> coded 27020, 28011, 28030, and 29012, were selected for pit sampling from the 10 watersheds in which soil was sampled in 2004 as part of the WASS. The four watersheds were selected to provide a range in acid buffering, with base saturation values that fell below and above the base saturation threshold for aluminum (Al) mobilization of 16.8% determined by Lawrence et al. (2018). The parent material of soil in watersheds 27020, 28011, and 28030 was glacial till, whereas the parent material of soil in watershed 29012 was deep, glacially deposited sand, which is common near the southwestern edge of the Adirondack ecoregion, but uncommon elsewhere in the region.

In 2004, soil samples were collected in five sampling areas that were distributed longitudinally within each of the four WASS watersheds to cover the full span of hillslope positions. Three soil pits were dug in each sampling area within a radius of approximately 10 meters (m). Samples of equal volume were collected from each pit from the Oa or A, and upper 10 centimeters (cm) of the B horizons. Samples were combined by horizon within each area yielding a watershed sample size of five for the two horizons. During sampling it was not possible to clearly determine whether the sampled horizon that occurred beneath the Oe and above either the E, or uppermost B (where E was not present), was an Oa or A horizon. More information on the soils component of the WASS Project is available online.<sup>3</sup>

Soil remaining after analysis from all 10 WASS watersheds sampled in 2004 was archived in an air-dried form to enable future reanalysis for quality assurance purposes. In this study, all the samples collected in 2004 from watersheds 27020, 28014, 28030 and 29012 were reanalyzed along with the analysis of soil from the recent sampling to ensure analytical consistency, as described in Lawrence et al. (2016). Reanalysis data were used for Ca, Al and carbon (C). However, original pH data were used because of the possibility of some change occurring in pH during long-term storage (Lawrence et al. 2012). For the purposes of this analysis, the horizon between the Oe and E (or upper B) was grouped and hereafter referred to as Oa/A because Oa and A horizons could not be consistently differentiated in the field.

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<sup>2</sup> The watershed locations are shown in Lawrence et al., 2020a.

<sup>3</sup> Results from the 2003-2005 Western Adirondack Stream Survey, Final Report 08-22: <https://www.nysed.gov/-/media/Files/Publications/Research/Environmental/EMEP/Western-Adirondack-Stream-Survey.pdf>; accessed May 30, 2019.

The four WASS watersheds were resampled using the ADK sampling method in either 2016 (27020 and 28014) or 2017 (28030 and 29012) with a design that differed from that used in the 2004 sampling. Three areas (study units) were located within each watershed to represent the three primary landscape types within the watershed. Within each study unit, one main pit and five satellite pits were sampled. Therefore, within each watershed three samples from the Oe, Oa/A and upper 10 cm of the B horizons were collected from the main pits and 15 samples were collected from the satellite pits. All samples were chemically analyzed individually, yielding a watershed replication of  $n = 18$ . Collection of soil samples was done according to a detailed sampling protocol as described in Lawrence et al. (2016), which was developed to provide an optimized sampling approach for continued future soil monitoring of these watersheds, as well as enable comparison to the 2004 study results.

Sampling below the upper 10 cm of the B horizon was done only in the main pits to enable full characterization of the soil profile, which included estimating total element mass for the entire profile. To minimize costs and site disturbance, deeper sampling was not done in the satellite pits. However, this decision will need to be reviewed in future applications depending on the environmental conditions encountered. The ADK sampling method, which is detailed in appendix A, was developed from prior experience of the USGS New York Water Science Center and the Northeastern Soil Monitoring Cooperative (Lawrence et al. 2016).

Total horizon mass of nitrogen (N) and C were calculated from concentration values, horizon thickness measurements, and bulk density estimates determined by measurements of loss-on-ignition and the model of Federer et al. (1993). Coarse fraction was determined for the mineral soil in each main pit by excavating a measured volume of the entire B horizon (top to bottom), and weighing all fragments not passing through a 1.0-cm screen. Rock density was assumed to be  $2600 \text{ kg m}^{-3}$ . This value represents an approximate value of rocks and minerals common in the Adirondack region (Olhoeft and Johnson 1984, Newton et al. 1987). Coarse fraction material was rare in the forest floor, and therefore, not estimated for this layer.

### **3.2 Buck Creek Soil Sampling**

To support the analysis of the ADK sampling method, this report includes soil data that were collected in North Buck in 1997 and 2009–2010, and South Buck in 1998 and 2014. Soil samples were collected in each watershed within 15 m of 28 permanent point locations roughly equidistant along seven transects approximately perpendicular to the slope line and distributed from upper to lower elevations. Soil samples from the Oe, Oa, and upper 10 cm of the B horizons were collected at all locations. At a few locations,

the A horizon could not be distinguished from Oa in the field. In this analysis, A horizon samples were excluded. Soil pits were excavated with shovels to expose a pit face that was used for measurement of horizon thickness and collection of soil. The soil resampling procedures used in this study are further detailed in Lawrence et al. (2016). Total horizon mass of C and N were determined with the same method used for both the ADK sampling method and the WASS sampling.

### **3.3 Forest Floor Core Sampling Design**

A primary goal of this study was to test a new method for measuring changes in forest floor mass, a possible consequence of climate change as well as other environmental changes such as recovery from acidic deposition effects. Measurement of changes in forest floor mass is often complicated by difficulties in determining the interface between the forest floor and mineral soil. Methods used for determining forest floor mass either rely on combining field measurements of forest floor thickness with bulk density estimates or collecting and weighing the full thickness over a known surface area. In both cases, irregularity of the boundary between the forest floor and mineral soil can create high variability in forest floor thickness that can be difficult to account for whether by measuring or collecting the full-forest floor thickness. This variability can substantially reduce the precision of mass estimates. Sampling or measuring the forest floor thickness can also be complicated by a diffuse boundary between the forest floor and underlying mineral soil, which is common in hardwood forest soils.

To improve the precision of forest floor mass estimates, this project tested the use of a 1.8-cm-diameter corer with a removable liner to collect the entire horizon thickness, plus some of the upper mineral soil to ensure that the full thickness of the forest floor was sampled. Upon collection, the sample was returned to the laboratory inside the plastic liner. After drying, the sample was slid out of the liner, intact, to enable close inspection of the interface between the forest floor and the mineral soil. Samples that are determined to be mineral soil based on color (assessed visually) and texture (assessed both visually and by touch) are excluded.

Adirondack soil profiles commonly possess a mineral E horizon directly beneath the forest floor that has a distinct bleached color that contrasts with the dark brown color of the forest floor. This color contrast aids in locating the interface between the forest floor and mineral soil. However, where the E horizon is not present (also common in Adirondack soil profiles), the horizon directly overlaying the uppermost B horizon will be an organic Oa horizon or mineral A horizon, which can be difficult to differentiate by

color or texture either in the field or the laboratory. Identification of Oa and A horizons can only be done definitively by chemical analysis. In the forest floor coring method, where E horizons are not present, the horizon immediately above the B horizon (whether Oa or A) was included as part of the forest floor mass. The interface between the B horizon and Oa or A horizons can generally be identified by color and texture.

In this manner, the forest floor (including the A horizon, if present) was precisely separated from the mineral soil over the 2.54-cm<sup>2</sup> area of the core cross section. Error in the estimate of organic matter mass was thereby reduced relative to commonly used methods because the organic-mineral interface was determined over the small cross section of the corer (2.54 cm<sup>2</sup>) under close inspection in the laboratory. This new approach contrasts traditional methods of forest floor sampling that depend on capturing the irregular organic-mineral horizon interface over areas of 100 cm<sup>2</sup> (Federer 1982) to 0.5 m<sup>2</sup> (Fernandez et al. 1993) and may require additional horizon thickness measurements or measurements of large quantities of soil in the field (Huntington 1988).

To better characterize the areal variability of forest floor mass throughout the watershed and provide sufficient sample for analysis, three cores were collected at each sampling location that were combined in the laboratory prior to analysis. After determining total mass of the three cores (after drying at 65 °C), samples were sieved (2 mm) and homogenized by mixing the combined samples in a plastic bag. Sub samples were used to determine the concentration of organic matter, organic C, and organic N. By knowing concentration and total mass of the core samples, mass per unit area of organic matter and the mass of C and N were determined for the forest floor.

Core sampling was done in the same four WASS watersheds where pit resampling was done, as well as in North and South Buck Creek watersheds, Maple and Balsam watersheds in the central Adirondacks, and watershed T24, the undisturbed reference watershed in the Honnedaga liming experiment (Lawrence et al. 2019). An additional eight watersheds were also selected for coring from WASS and the East-Central Adirondack Stream Survey (ECASS) watersheds where stream chemistry had been previously sampled. In each watershed, except T24, a two-person crew collected sets of three cores at 15 locations distributed along transects perpendicular to the approximate slope line over the full hillslope gradient (45 cores combined into 15 samples per watershed). In watershed T24, three cores were collected at five locations within each of five 20- by 50-m rectangular plots, yielding a total of 25 samples within the watershed. Watershed T24 was sampled in this manner in 2013, 2014, 2015 and 2016 to provide data to assess reproducibility of the method.

### 3.4 Chemical and Statistical Methods

Measurements of pH (0.01 CaCl<sub>2</sub> extraction), exchangeable acidity and Al (1.0 M KCl extraction), exchangeable Ca, Mg, Na, and K (1.0 M NH<sub>4</sub>Cl extraction), total C, total N, and loss-on-ignition (LOI) were made on all samples collected with WASS, ADK, and Buck Creek sampling methods. Total C, total N and LOI were determined on all soil cores. Because these soils do not contain carbonate, total C represents total organic C. Exchangeable hydrogen (H) was determined by subtracting exchangeable Al from exchangeable acidity. Exchangeable forms of Al, Ca, Mg, Na, K, and H are hereafter referred to simply as Al, Ca, Mg, Na, K, and H. All chemical analyses were done in the USGS New York Water Science Center Soil and Low-Ionic Strength Water Quality Laboratory (hereafter the USGS Troy Laboratory). Full documentation of analysis methods appear in Lawrence et al. (2020a). Cation exchange capacity (CEC) was determined from the laboratory measurements by summing exchangeable concentrations of the primary cations in these soils (Ca, Mg, Na, K, Al and H). Base saturation was determined by dividing the sum of base cations (Ca, Mg, Na, and K) by the CEC. Al saturation was determined by dividing Al by CEC. All data used for the analysis of this report are available in Lawrence et al. (2020a).

SigmaPlot 13.0 was used to run paired and unpaired T-tests and Mann-Whitney rank sum tests where normality was disproven to determine differences between samplings as well as power analysis with paired and unpaired T-tests to determine minimum detectable difference between samplings. All T-tests were two-tailed.

## 4 Results and Discussion

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**Question 1.** *What are the minimum detectable changes that can be determined with the ADK sampling method, based on statistical power of 0.80 and alpha of 0.05, and how do these minimum values compare to the prior sampling design used in these watersheds?*

The minimum detectable change (MDC) for the ADK sampling method differed markedly between horizons. Higher values of MDC were found for the Oa/A than the upper B for nearly all measurements evaluated in this analysis (Table 1). This result is in part related to the role of the Oa/A as a transition between the litter-derived Oe and the mineral-dominated E or upper B, which leads to large variations in organic C concentrations in the Oa/A. This high degree of variability reduces the power of detecting changes over time through resampling. Lower MDC values in the upper B than Oa/A are also related to lower concentrations of total C, which is largely associated with organic matter. In these soils, organic matter is the primary source of the negative charge of soil particles, which provide CEC. Lower CEC results in lower concentrations of exchangeable cations (such as Ca, Al, and H). The lower bulk density of the porous Oa/A than the upper mineral soil may also contribute to greater variation in these measurements in the Oa/A. Because MDC tends to decrease with concentration, lower MDC values do not necessarily equate to increased sensitivity for detecting changes. For example, in watershed 28030, with a mean B horizon Ca concentration of  $1.5 \text{ cmol}_c \text{ kg}^{-1}$ , the MDC was  $1.4 \text{ cmol}_c \text{ kg}^{-1}$  (Table 1), whereas in watershed 29012, with a mean Ca concentration of  $0.17 \text{ cmol}_c \text{ kg}^{-1}$ , the MDC was 0.13 (Table 1). Although the Ca concentration of watershed 28030 was nearly 10 times higher than that of watershed 29012, the MDC values were similar when expressed as a percent (96% and 78% for 28030 and 29012, respectively). Therefore, the opportunity for detecting change might be similar in each of these soils despite the much higher absolute value of MDC in watershed 28030.

Consistent differences in MDC also occurred among the four watersheds as a result of variations in soil characteristics such as texture, organic carbon content and acid-buffering capacity. These and other factors that vary across soils can affect the statistical power to detect change, as shown in Table 1. Regardless of differences in soils, however, the considerably larger sample size of 18 provided by the ADK sampling method relative to the sample size of five used in WASS sampling resulted in lower MDC values for most measurements in all watersheds and both horizons. Combining WASS samples before analysis had the potential for reducing variance by attenuating extreme values, but also reduced the sample size used in the MDC calculation. Had WASS samples not been combined before analysis, the sample size of 15 would have been similar to that of the ADK method. The effect of combining samples before analysis may have played a role in Ca measurements, for which MDC values were less

for the WASS method than the ADK method in half of the comparisons despite a much lower sample size. However, for Al measurements, the ADK method yielded lower MDC values in all comparisons and the ADK yielded lower MDC values for pH and total carbon in 12 of 16 comparisons (Table 1). Combining samples before analysis is often done to save laboratory costs, but this comparison indicates that the higher sample size obtained by not combining samples before analysis typically yields greater power to detect change, and therefore is the preferred approach.

**Table 1. Minimum Detectable Change from ADK and WASS Sampling Methods**

Red text indicates that the minimum detectable change for the ADK method was less than that for the WASS method. Units are  $\text{cmol}_c \text{ kg}^{-1}$  for exchangeable Al and Ca, and percent for total C.

		Minimum Detectable Change							
		pH		Al		Ca		Total C	
Watershed	Horizon	ADK	WASS	ADK	WASS	ADK	WASS	ADK	WASS
27020	Oa/A	0.51	0.49	3.5	9.3	6.3	7.0	13	13
28014	Oa/A	0.45	0.51	3.6	5.7	3.0	2.4	11	17
28030	Oa/A	0.49	0.55	1.5	2.2	10.6	11	12	21
29012	Oa/A	0.19	0.47	0.94	2.4	4.9	6.5	12	7.7
mean		0.41	0.51	2.4	4.9	6.2	6.7	12	14.7
27020	upper B	0.24	0.43	1.2	2.1	0.27	0.24	2.6	5.8
28014	upper B	0.29	0.36	2.5	2.6	0.54	0.18	2.4	3.8
28030	upper B	0.37	0.28	1.6	2.5	1.4	0.8	1.3	3.7
29012	upper B	0.24	0.32	1.1	1.6	0.13	0.14	1.8	4.2
mean		0.29	0.35	1.6	2.2	0.59	0.34	2.0	4.4

**Question 2.** *How does minimum detectable change vary with sample size and paired versus unpaired sampling designs in these soils?*

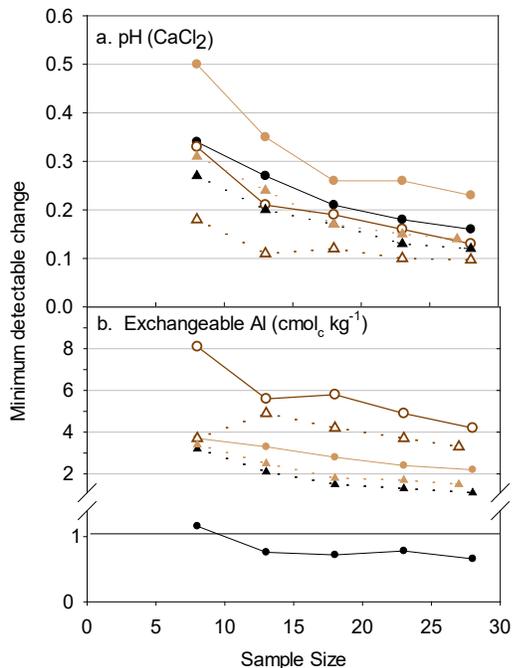
The MDC decreases as sample size increases, but the selection of sample size is always subject to practical constraints. The optimum sample size must provide the ability to detect ecologically relevant changes in soils without exceeding project budgets or destroying the area needed for future resampling within the study unit. To evaluate the sample size used in the ADK method, data from sampling/resampling in North Buck were used to calculate the MDC from sample sizes of eight, 13, 18, 23, and 28 (Lawrence et al. 2015). Values of MDC were based on unpaired T-tests that relied on data variability from sampling at 28 locations in 2009–2010. Values of MDC were also

determined based on paired T-tests that required data from the initial sampling in 1997, as well as the resampling in 2009–2010 to incorporate the variability in changes measured between the two samplings. Paired T-tests could be used in this comparison because the sampling and resampling in North Buck were done at the same 28 fixed locations within the watershed. For sample sizes of 8 through 23, samples were randomly chosen from the total of 28. Samples from the Oe, Oa and upper 10 cm of the B were used for both paired and unpaired T-tests.

Values of MDC for pH decreased sharply from a sample size of eight to a sample size of 18 (the sample size used in the ADK method), then more gradually to 23 and 28 (Figure 1a). This trend was consistent for all three soil horizons and unpaired and paired T-tests. Paired T-test results showed substantially lower values of MDC than unpaired T-tests in all horizons and all sample sizes, which has been noted previously in a soil resampling study (Yanai et al. 2003). Concentrations of Al showed patterns in MDC decrease with sample size that were similar to those for pH, although there were some inconsistencies in the Oa horizon (Figure 1b). Like pH, MDC values based on paired T-tests were less than those based on unpaired T-tests in all comparisons and all horizons.

**Figure 1. Minimum Detectable Change versus Sample Size for pH and Al**

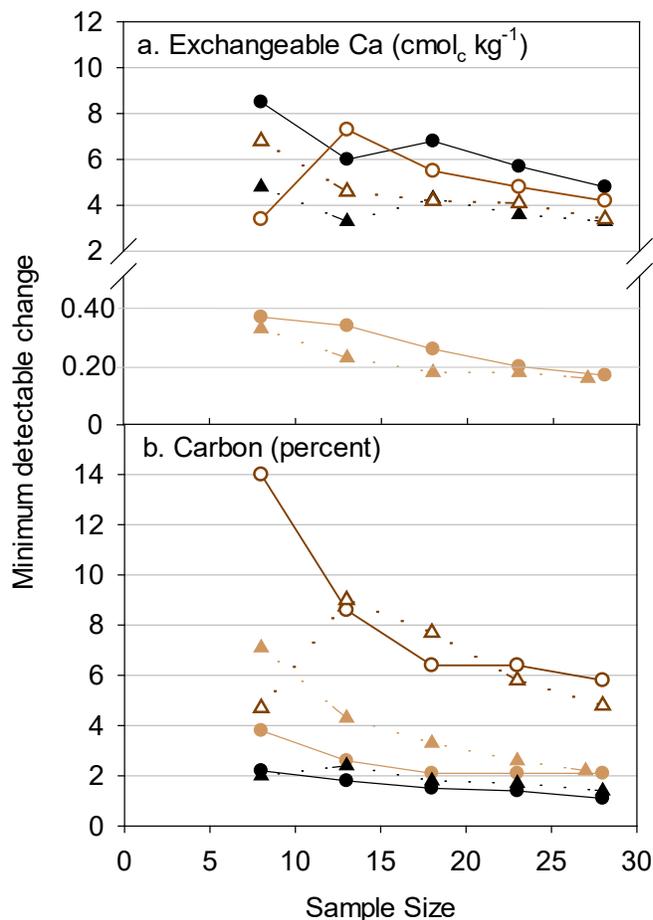
Data are based on soil samples collected from North Buck watershed. Black lines/symbols represent the Oe, horizon brown lines and unfilled symbols represent the Oa horizon, and tan lines/symbols represent the upper B horizon. Circles/solid lines are based on unpaired T-tests. Triangles/dashed lines are based on paired T-tests.



Trends in MDC with sample size for Ca were less consistent than pH or Al but showed the same general trend of a decreasing slope above a sample size of 18 (Figure 2a). Like Al, the values for Ca in the Oa horizon were somewhat erratic. Trends in MDC with sample size for total C showed distinct differences among horizons (Figure 2b). In the Oe horizon, the MDC was higher for the paired T-test than the unpaired T-test at most sample sizes, although values for both decreased similarly with increased sample size. Values of MDC in the Oa horizon were also higher for paired T-tests than unpaired T-tests for sample sizes of 13 and 18, and the pattern for the paired T-test differed from that for the unpaired T-test. The most unusual result was the higher MDC values for unpaired than paired T-tests in the B at all sample sizes. However, the MDC values for the B followed the typical pattern of a flattening slope beyond a sample size of 18.

**Figure 2. Minimum Detectable Change versus Sample Size for Ca and C**

Data are based on soil samples collected from North Buck watershed. Black lines/symbols represent the Oe horizon, brown lines and unfilled symbols represent the Oa horizon, and tan lines/symbols represent the upper B horizon. Circles/solid lines are based on unpaired T-tests. Triangles/dashed lines are based on paired T-tests.



The sampling replication for the ADK sampling method of  $n=18$  was chosen based on experience gained in prior soil resampling studies in similar soils (Lawrence et al. 2012, Lawrence et al. 2015, Lawrence et al. 2016), but had not been quantitatively evaluated. The results presented in Figure 1 and Figure 2 support the choice of a sample replication of  $n=18$  as a balance between detection sensitivity and practical considerations of increasing sample replication. Decreased MDC might be needed for other measurements depending on the question asked.

**Question 3.** *How effective is the ADK sampling replication of  $n=18$  in detecting real changes in Adirondack soils?*

The general decrease in slopes in relations between MDC and sample size beyond the ADK sample size of 18 provides support for this level of sampling in terms of a diminishing benefit for higher sampling replication. However, the results in Figure 2 do not relate this information to the actual magnitude of change that might realistically occur in Adirondack soils and does not consider the time frame over which the changes would occur. Therefore, detection of actual change was compared between the ADK sample size of 18 samples per watershed with a sample size of 28 samples per watershed using data from both North Buck (sampled in 1997 and 2009–2010) and South Buck (sampled in 1998 and 2014). In this analysis, results are presented from paired T-tests (or Mann-Kendall tests for non-normal data) with 18 samples randomly selected from the total of 28, as well as for all 28 samples, for the Oe, Oa and upper B horizon.

In the Oe horizon of North Buck, significant differences of  $P < 0.1$  were detected between the initial sampling and resampling 12–13 years later in 14 of 16 measurements with both  $n=28$  and  $n=18$  (Table 2). These data reflect substantial changes in soil properties over the sampling interval with little difference in detection between the two sampling sizes. For LOI and H, the uncertainty increased from  $P < 0.05$  to  $P < 0.10$  with the decrease in sample size, but for base saturation and C in  $\text{Mg ha}^{-1}$ , the reverse was true. The uncertainty also increased from  $P < 0.01$  to  $P < 0.05$  for pH and Ca with the decrease in sample size. In the Oe horizon of South Buck, the number of changes observed for  $P < 0.10$  were also similar between sample sizes; 12 with  $n=28$  and 11 with  $n=18$  (Table 2). However, shifts from  $P < 0.01$  to either  $P < 0.05$  or  $P < 0.10$  occurred in three measurements and the change in N as  $\text{Mg ha}^{-1}$  was highly significant ( $P < 0.01$ ) with  $n=28$  but not significant ( $P > 0.10$ ) with  $n=18$ . A change in N as  $\text{g kg}^{-1}$  was observed at  $P < 0.10$  with  $n=18$  but no change was seen with  $n=28$ .

**Table 2. Detected Changes in Buck Oe Horizons with n=28 and n=18**

Values in dated columns represent mean values of the 28 samples collected in those years. Base sat. denotes base saturation; Al sat. denotes Al saturation; H denotes exchangeable hydrogen. Symbols \*\*, \* and ~ represent P values of < 0.01, < 0.05 and < 0.10, respectively.

Oe Horizon	North Buck				South Buck			
	1997	2009-10	P (n = 28)	P (n = 18)	1998	2014	P (n = 28)	P (n = 18)
C (g kg <sup>-1</sup> )	460	513	**	**	427	421		
C (Mg ha <sup>-1</sup> )	29	32	~	*	23	16	**	~
LOI (%)	91	93	*	~	85	93	**	**
N (g kg <sup>-1</sup> )	22	25	**	**	22	23		~
N (Mg ha <sup>-1</sup> )	1.4	1.5			1.2	0.89	**	
C:N (g:g)	21	21			20	18	**	**
CEC (cmol <sub>c</sub> kg <sup>-1</sup> )	26	32	**	**	24	22		
Base sat. (%)	60	65	~	*	50	85	**	**
pH	3.02	3.16	**	*	3.35	3.36		
H (cmol <sub>c</sub> kg <sup>-1</sup> )	8.4	9.9	*	~	5.6	2.4	**	*
Al (cmol <sub>c</sub> kg <sup>-1</sup> )	2.4	0.8	**	**	6.8	0.95	**	**
Al sat. (%)	9.7	2.6	**	**	27	3.4	**	**
Ca (cmol <sub>c</sub> kg <sup>-1</sup> )	12	17	**	*	9.5	14.8	**	**
Mg (cmol <sub>c</sub> kg <sup>-1</sup> )	2.1	2.7	**	**	1.3	2.2	**	**
Na (cmol <sub>c</sub> kg <sup>-1</sup> )	0.11	0.06	**	**	0.14	0.08	**	*
K (cmol <sub>c</sub> kg <sup>-1</sup> )	1.3	1.5	*	*	1.3	1.5	~	

In the Oa horizon of North Buck, significant differences of  $P < 0.10$  were detected in nine measurements with n=28, but only six measurements with n=18 (Table 3). For measurements where change was detected with both sample sizes, results also showed an increase in uncertainty in three measurements if the sample size was decreased from 28 to 18. In the Oa horizon of South Buck, significant differences ( $P < 0.10$ ) were observed in six and five measurements with n=28 and n=18, respectively, and an increase in uncertainty from decreased sample size was seen in only one measurement where change was detected for both sample sizes (Table 3). Unlike the Oe horizon, no measurements in the Oa horizon showed stronger change detection in either watershed for n=18 than n=28.

**Table 3. Detected Changes in Buck Oa Horizons with n=28 and n=18**

Values in dated columns represent mean values of the 28 samples collected in those years. Base sat. denotes base saturation; Al sat. denotes Al saturation; H denotes exchangeable hydrogen. Symbols \*\*, \* and ~ represent P values of < 0.01, < 0.05 and < 0.10, respectively.

Oa Horizon	North Buck				South Buck			
	1997	2009-10	P (n = 28)	P (n = 18)	1998	2014	P (n = 28)	P (n = 18)
C (g kg <sup>-1</sup> )	416	466	**	*	378	366		
C (Mg ha <sup>-1</sup> )	82	92			24	15		
LOI (%)	83	83			74	84	**	*
N (g kg <sup>-1</sup> )	17	20	**	**	18	18		
N (Mg ha <sup>-1</sup> )	3.2	3.7	*	~	1.2	0.74		
C:N (g:g)	25	23	*	*	20	20		
CEC (cmol <sub>c</sub> kg <sup>-1</sup> )	31	31			20	15	**	**
Base sat. (%)	30	31			43	49		
pH	2.69	2.74	*		3.15	3.17		
H (cmol <sub>c</sub> kg <sup>-1</sup> )	8.4	9.9	*		5.6	2.4	**	
Al (cmol <sub>c</sub> kg <sup>-1</sup> )	10.4	7.8	*	~	6.1	3.1	**	*
Al sat. (%)	35	26	*		29	21		
Ca (cmol <sub>c</sub> kg <sup>-1</sup> )	7.6	8.0			8.6	6.0	~	~
Mg (cmol <sub>c</sub> kg <sup>-1</sup> )	1.4	1.3			0.90	0.90		
Na (cmol <sub>c</sub> kg <sup>-1</sup> )	0.14	0.04	**	**	0.13	0.05	**	**
K (cmol <sub>c</sub> kg <sup>-1</sup> )	0.49	0.43			0.69	0.61		

In the upper B horizon of North Buck, change detection differed between sample sizes for only two measurements (Table 4). Uncertainty increased in Na measurements from  $P < 0.05$  to  $P < 0.10$  with decreased sample size from size n=28 to n=18, however a significant change was recorded in C (g kg<sup>-1</sup>) for n=18 ( $P < 0.05$ ), but not for n=28 ( $P > 0.10$ ). Similarly, in South Buck there was little difference in change detection between a sample size of n=28 and n=18 (Table 4). Ten changes were detected with n=28 and 11 changes were detected with n=18. The only differences between sample sizes were a significant change in Ca ( $P < 0.10$ ) with n=28 but no change with n=18 ( $P > 0.10$ ), and a significant change in N ( $P < 0.10$ ) with n=18 but not with n=28.

**Table 4. Detected Changes in Buck Upper B Horizons with n=28 and n=18**

Values in dated columns represent mean values of the 28 samples collected in those years. Base sat. denotes base saturation; Al sat. denotes Al saturation; H denotes exchangeable hydrogen. Symbols \*\*, \* and ~ represent P values of < 0.01, < 0.05 and < 0.10, respectively.

Upper B Horizon	North Buck				South Buck			
	1997	2009-10	P (n = 28)	P (n = 18)	1998	2014	P (n = 28)	P (n = 18)
C (g kg <sup>-1</sup> )	80	91		*	57	60		
C (Mg ha <sup>-1</sup> )	35	35			27	20	**	**
LOI (%)	14	18	*	*	13	22	**	**
N (g kg <sup>-1</sup> )	3.4	3.6			2.7	2.6		~
N (Mg ha <sup>-1</sup> )	1.5	1.3			1.3	0.85	**	**
C:N (g:g)	26	26			21	24	**	**
CEC (cmol <sub>c</sub> kg <sup>-1</sup> )	5.2	11	**	**	3.4	4.6	**	*
Base sat. (%)	12	6.1	**	**	15	9.6	**	**
pH	3.35	3.53	**	**	3.79	3.74		*
H (cmol <sub>c</sub> kg <sup>-1</sup> )	1.4	1.4			0.51	0.42		
Al (cmol <sub>c</sub> kg <sup>-1</sup> )	3.3	8.6	**	**	2.4	3.9	**	**
Al sat. (%)	64	81	**	**	71	85	**	**
Ca (cmol <sub>c</sub> kg <sup>-1</sup> )	0.38	0.41			0.36	0.26	~	
Mg (cmol <sub>c</sub> kg <sup>-1</sup> )	0.11	0.10			0.07	0.06		
Na (cmol <sub>c</sub> kg <sup>-1</sup> )	0.03	0.02	*	~	0.05	0.02	**	**
K (cmol <sub>c</sub> kg <sup>-1</sup> )	0.07	0.09	~	~	0.06	0.07		

Overall, differences in detection between n=28 and n=18 were modest in the Oe horizon of both North and South Buck. Lower sample size in North Buck did have a stronger effect on change detection in the Oa than the Oe horizon, but in South Buck lower sample size had similarly small effects on both the Oa and Oe horizon. Overall changes in detection between n=28 and n=18 were minimal in the upper B of both watersheds. With the possible exception of the North Buck Oa horizon, reducing the sample size from 28 to 18 had relatively minor effects on the uncertainty of results. These data indicated that there was some variability in the relation between sample size and uncertainty in detecting change among watersheds and horizons, but that a sample size of 18 provided nearly the same amount of information as the sample size of 28.

In addition to showing that numerous changes in Adirondack soils can be detected on a small-watershed basis with a sample size of 18, this analysis also demonstrated that substantial changes occurred over periods of 12 to 16 years, and possibly less. A large decline in acidic deposition that occurred between sampling intervals (Driscoll et al. 2016) was likely to have played an important role in these changes. However, the decreased acidic deposition did not translate directly to increases in soil pH. Although a clear increase in soil pH occurred in North Buck, pH values were nearly the same in South Buck in 1998 and 2014, even though the close proximity of the watersheds suggested that they received highly similar levels of deposition.

The decrease of sulfate ion ( $\text{SO}_4^{2-}$ ), and to a lesser extent nitrate ion ( $\text{NO}_3^-$ ), concentrations in acidic deposition lowered the ionic strength of precipitation, making it more dilute as well as less acidic, and the effects of these two factors differed between the watersheds. In North Buck, the soil remained extremely acidic despite an increase in pH, due largely to the high-organic matter content related to effects of coniferous vegetation (Ross et al. 1996), which composes about half the basal area of the watershed. Organic acids associated with soil organic matter provided a high capacity to buffer the increase in pH as acidic deposition rates decreased. The abundant soil organic matter also provided a high CEC that reduced the dilution effect of the changing deposition.

In South Buck, where soil organic matter was considerably lower than in North Buck due to dominant hardwood vegetation, pH was higher, and CEC was lower than in North Buck. The decreased inputs of acidity and more dilute precipitation in South Buck was likely to have increased the solubility of soil organic matter to a greater degree in South Buck than North Buck, which likely led to an increase in decomposition in the Oe and Oa of South Buck. This interpretation is supported by large decreases in C ( $\text{Mg ha}^{-1}$ ) in the Oe and possibly the Oa in South Buck (although the Oa difference was not significant at  $P > 0.10$ ), whereas in North Buck relatively small increases in C ( $\text{Mg ha}^{-1}$ ) were measured that were only significant in the Oe ( $P > 0.10$ ). Additional interpretive details on soil changes related to SOC and differences between these watersheds is available elsewhere (Lawrence et al. 2020b).

Increases in pH were considered the primary factor in decreases of Al in O horizons in a regional study of sites in the northeastern United States and eastern Canada (Lawrence et al. 2015). However, increases in pH occurred in North Buck, but not South Buck and North Buck pH remained lower than South Buck. Increased solubility of soil organic matter from decreased ionic strength of acidic deposition was a more likely cause of the pronounced decrease in Al concentration and saturation in the Oe and Oa horizon in both watersheds. Decreased ionic strength was found to be the primary cause of increased organic carbon

solubility in a Czech watershed recovering from acidic deposition (Hruska et al. 2009). The decrease in Oe and Oa Al concentrations, which was more strongly expressed in South Buck than North Buck, represented a reversal of the increase in Al in the Oa horizon that was documented in the Adirondacks between the mid-1980s and about 2000 (Warby et al. 2009). Much larger increases in Oe base saturation in South Buck than North Buck were in part caused by the larger decrease in Al. However, Ca concentrations also increased to a greater degree in the Oe horizon in South Buck than North Buck, which also contributed to the larger increase in base saturation in South Buck. Because Al suppresses decomposition rates (Kunito et al. 2016), the large decreases in South Buck Al concentrations were likely to have furthered increases in decomposition along with the increase in base saturation. The Al decrease in the Oe and Oa horizons was contrasted by an increase in Al in the upper B horizon, which suggested that Al had been moving downward from the Oe and Oa to be deposited in the upper B (Lawrence et al. 2015).

**Question 4.** *Does spatial variability in soil chemistry change with sampling depth?*

The ADK sampling method focuses on the identification of soil change in the upper soil profile, where atmospheric and vegetation effects are expressed more directly than deeper in the profile where there is more opportunity for attenuation of effects that originate from above. Collection of soil deeper in the profile also requires a larger area for excavation than shallow pits, which precludes more of the study unit from future resampling. Nevertheless, important information has been identified from sampling and resampling lower B soil (Bailey et al. 2005, Gross and Harrison 2019, Hamburg et al. 2019). The full profile sampling of three pits in each watershed was included in the ADK sampling to provide quantitative information on pool sizes of key elements such as C and to aid in understanding processes of soil change that involve interaction among horizons.

Full-profile sampling of three pits per watershed would likely be insufficient to statistically detect soil changes in most circumstances. Therefore, the ADK sampling method would need to be altered to monitor deep soil horizons for change over time. As the need for additional information on soil change develops, the additional cost and sampling area needed for monitoring deep soil horizons may become warranted, but the replication needed for Adirondack soils is uncertain.

To begin evaluation of the number of samples that would be needed to identify soil changes in deep horizons on a watershed basis, the USGS determined the coefficient of variation (CV) for upper, mid, and lower B horizons using the ADK data (three pits per watershed). If the CV values were consistently lower for mid or lower B horizons than the upper B horizon, then adequate statistical power could be achieved with fewer soil pits than needed for the upper soil horizons, although the opposite would be true if lower soil CV values were higher. Comparison of the CV for upper B horizons with mid and lower B horizons did not reveal a pattern in variability with depth for pH, Al, Ca or total C across the four watersheds, although pH did exhibit a trend of decreasing CV with increasing depth in three of four watersheds (Table 5). These results suggest that similar sampling replication would be needed throughout the B horizon to obtain similar power to detect changes among these horizons.

**Table 5. Values of Coefficient of Variation for Three Depths of the B Horizon**

All values of coefficient of variation (CV) are based on a sample size of three.

<b>Watershed</b>	<b>Horizon</b>	<b>pH</b>	<b>Exchangeable Al</b>	<b>Exchangeable Ca</b>	<b>Total C</b>
27020	Upper B	5.3	65	32	51
	Mid B	3.5	91	51	102
	Lower B	2.9	90	43	85
28014	Upper B	12.7	128	35	67
	Mid B	6.5	94	9.5	93
	Lower B	2.2	48	39	46
28030	Upper B	13.5	49	53	1.2
	Mid B	4.6	63	68	45
	Lower B	3.8	34	88	22
29012	Upper B	1.7	22	21	15
	Mid B	0.9	41	46	19
	Lower B	2.2	86	87	85

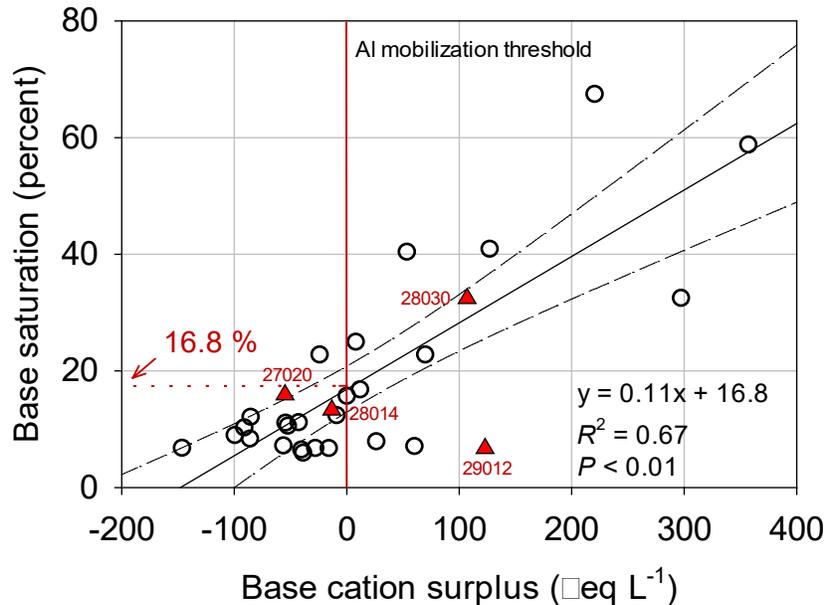
**Question 5.** *Did the ADK sampling method characterize soil variability on a watershed basis?*

Previous studies have shown a strong link between soil chemistry and stream chemistry during high-stream flows in small watersheds (Lawrence et al. 1999). Because stream chemistry at the base of a watershed integrates the effects of soil variability throughout that watershed, stream samples can be used to evaluate the effectiveness of soil sampling in characterizing soil for the watershed as a whole. If the sampling design reasonably represents the variability of soils in the watershed, then a relation between soil chemistry and stream chemistry should be apparent if a substantial fraction of streamflow were derived from subsurface flow paths that discharge water directly from the soil into stream channels. This situation most commonly occurs in headwater streams during elevated streamflow. Low-flow conditions, or other factors that result in flow paths below the soil, as well as strong influences from riparian areas, can weaken soil-stream chemistry relations.

The four watersheds sampled in this study did not provide enough data to develop a soil-stream relation to evaluate the sampling design on their own. However, these data were compared to a relation developed previously from 26 other Adirondack watersheds (Lawrence et al. 2018), which related the base-cation surplus (BCS) of stream water to the base saturation of the upper B horizon (Figure 3). This relation provided a useful method for evaluating the overall relation of stream chemistry to soil chemistry because the BCS is derived from seven water measurements (calcium ion– $\text{Ca}^{2+}$ , magnesium ion– $\text{Mg}^{2+}$ , sodium ion– $\text{Na}^+$ , potassium ion– $\text{K}^+$ ,  $\text{SO}_4^{2-}$ ,  $\text{NO}_3^-$ , chloride ion– $\text{Cl}^-$ , and dissolved organic carbon) and base saturation is derived from six soil measurements (described in Methods). Full details on the derivation and use of the BCS is available in (Lawrence et al. 2007). The means of base saturation values measured within each of the four watersheds in the pilot study ( $n = 18$ ) in either 2016 or 2017 were plotted against the mean BCS values determined by collecting a stream sample in each of these watersheds during snowmelt in 2014 and again in 2015 ( $n = 2$ ).

**Figure 3. Soil Base Saturation as a Function of Base Cation Surplus (BCS in Streams)**

The value of 16.8% represents the threshold of base saturation below which mobilization of Al occurs. Base saturation was measured in the upper B horizon. Negative BCS values indicate Al mobilization. Confidence interval ( $P < 0.05$ ) is indicated by dashed lines. Watersheds where soil samples were collected in this study from excavated pits are shown by red triangles.



Three of the four watersheds plotted within or very close to the 95% confidence interval of the best-fit line determined from the 26 other watersheds (Figure 3), which suggested that the soil sampling design characterized soil variability reasonably well for these watersheds. Watershed 29012, however, did not fit the relation, which was not an unexpected result. The highly permeable deep sand deposits underlying this watershed enable nearly all precipitation to infiltrate and move far below the sampled soil layer before eventually discharging to the stream channel, even during periods of high precipitation and snowmelt. As a result, the chemistry of the stream water was strongly affected by deep deposits that differed biogeochemically from the upper B horizon presented in Figure 3.

**Question 6.** *Did resampling with ADK sampling method reveal soil changes in watersheds previously sampled with the WASS sampling method?*

The primary objective of the WASS soil sampling design was to characterize soil variability throughout the sampled watersheds. Combining samples from the same sampling area helped to incorporate spatial variability into the measurements and reduced the costs of laboratory analyses, but this approach substantially reduced the statistical power when these data were compared to the ADK sampling to determine if changes over time had occurred. The loss of statistical power resulted from the small WASS sample size of five, but also from the unbalanced comparison of  $n=5$  versus  $n=18$  samples that increased the chances of unequal variances. Nevertheless, differences of  $P < 0.05$  were observed for Al and C in the Oa/A horizon of two sites, and pH in the upper B of one site (Table 6). Differences of  $P < 0.10$  were also observed for pH in the Oa/A horizon of three sites (Table 6).

**Table 6. Detecting Change with Western Adirondack Stream Survey Data**

Data were collected in 2004 with Western Adirondack Stream Survey (WASS) sampling method ( $n=5$ ); data collected in 2016/2017 were collected with ADK sampling method ( $n=18$ ). Values in blue indicate a significant change at  $P < 0.10$ ; values in red indicate a significant change at  $P < 0.05$ .

		2004	2016/2017	2004	2016/2017
Watershed	Horizon	pH		Al (cmol <sub>c</sub> kg <sup>-1</sup> )	
27020	Oa/A	3.07	3.59	11.8	6.8
28014	Oa/A	3.55	3.46	9.4	7.0
28030	Oa/A	3.02	3.31	2.2	1.7
29012	Oa/A	3.06	2.93	2.8	2.4
27020	upper B	3.75	3.95	3.3	3.2
28014	upper B	3.84	3.99	3.5	3.0
28030	upper B	3.78	4.09	3.1	2.6
29012	upper B	3.88	3.92	1.8	2.7
		Ca (cmol <sub>c</sub> kg <sup>-1</sup> )		Total C (percent)	
27020	Oa/A	7.5	5.4	26.0	27.8
28014	Oa/A	1.8	2.8	18.4	30.4
28030	Oa/A	11.4	12.0	20.4	22.6
29012	Oa/A	5.8	4.3	10.7	17.6
27020	upper B	0.37	0.49	5.5	5.4
28014	upper B	0.26	0.41	6.4	5.6
28030	upper B	0.67	1.45	4.7	5.1
29012	upper B	0.12	0.17	3.4	4.5

The results from this analysis indicate that changes occurred between sampling intervals that were quite pronounced in some watersheds and suggest that potentially valuable information would be gained if other watersheds sampled with the WASS method in 2004 were resampled using the ADK method. This includes resampling of other WASS watersheds that had been sampled in 2004 to enable grouping of watersheds for the purpose of regional assessment. In the analysis shown in Table 6, each of the four watersheds showed a decrease in Al in the Oa/A horizon and an increase in Ca in the upper B horizon. However, with a sample size of  $n=4$ , the power of the paired T-test to detect a change was extremely low for this group of watersheds. By adding data from North Buck and South Buck, the sample size increased to six and the representativeness of the group for the southwestern Adirondack region also improved somewhat and increased detection power. Accordingly, the paired T-test run on the group of six watersheds did show a decrease in Oa/A horizon Al ( $P < 0.01$ ) and an increase in upper B horizon Ca ( $P < 0.10$ ). These are important changes that suggest some recovery from acidic deposition effects, and are particularly noteworthy with regard to Ca, because no other studies have yet detected a regional Ca increase. Increasing availability of Ca in the soil is an essential step in the recovery of ecosystems damaged by acidic deposition. Nevertheless, the statistical power of 0.37 obtained with six watersheds in the Ca analysis is still low relative to the desired power of at least 0.80. The overall results of this analysis therefore suggest that resampling of other WASS watersheds sampled in 2004 would be of significant value for increasing the power to evaluate the recovery response of the western Adirondack region.

**Question 7.** *How reproducible are the forest floor data determined with small diameter corers?*

The forest floor core samples collected yearly for 4 years in the reference watershed (T24) as part of the Honnedaga liming experiment provided a useful opportunity to evaluate the reproducibility of core sampling, because the forest floor mass was considered unlikely to change in this undisturbed watershed within the three-year period that samples were collected. Therefore, variation in replicated sampling among years is of use in assessing sampling error. Year-to-year variability of the three measurements did not exhibit any trends and there were no significant differences between any of the years ( $P > 0.10$ ). The maximum difference in annual mean values ranged from 5.4% for organic matter content (percent based on  $\text{Mg ha}^{-1}$ ) to 13.3% for nitrogen content (percent based on  $\text{Mg ha}^{-1}$ ), and the values of CV averaged for the 4 years ranged from 28% for organic matter to 34.7% for nitrogen (Table 7). This variability includes the error associated with the chemical analysis, and is actually similar to the error that typically results from the chemical analysis alone (Ross et al. 2015). This level of reproducibility suggests high sensitivity in detecting changes if they occur in forest floor measurements with this method of sampling.

**Table 7. Variation of Repeated Sampling with Soil Cores**

Cores were collected in the same 20- by 50-m plots within an undisturbed Adirondack watershed in each of four years.

	Organic Matter		Organic Carbon		Nitrogen	
	Mean	CV	Mean	CV	Mean	CV
Year	Mg ha <sup>-1</sup>	%	Mg ha <sup>-1</sup>	%	Mg ha <sup>-1</sup>	%
2013	48.2	31.8	23.6	40.7	1.1	40.0
2014	45.6	28.6	21.9	33.5	1.0	34.8
2015	47.0	30.4	23.7	38.0	1.1	38.5
2016	46.3	22.1	22.8	25.8	1.0	25.6
mean	46.8	28.2	23.0	34.5	1.1	34.7
maximum difference (%)	5.4	-	7.2	-	13.3	-

**Question 8.** *How does the variability and quantification of forest floor mass measurements determined by coring compare on a watershed basis to those determined through pit excavation?*

The mass of organic matter retained within the forest floor has long been recognized as a key element of forest ecosystems because the forest floor controls retention and release of water and nutrients as well as controlling a variety of biogeochemical processes (Likens et al. 1977). The mass of organic matter stored in the forest floor depends on rates of inputs and losses, which are controlled by factors such as forest productivity, the biochemical composition of organic matter, and soil moisture and temperature. Ecological studies of the forest floor have therefore become increasingly focused on what effects changing climate will have on aspects of the forest floor including organic matter mass. Methods for measuring organic matter mass of the forest floor have generally involved collecting an entire block of the forest floor extending down to the interface with the mineral soil or using a pit face to measure the thickness of the forest floor along with the collection of a sample from the pit face. Each of these approaches provide a useful quantification of forest floor organic matter. However, substantial error is introduced into the quantification with both approaches by factors such as high spatial variability in forest floor thickness over horizontal distances on a centimeter scale, heterogeneity caused by extensive root development, and diffuse boundaries at the organic-mineral interface that are commonly encountered in soils of hardwood forests (Figure 4).

#### Figure 4. Photograph of the Forest Floor and Upper Mineral Soil

Yellow lines demonstrate different thickness of the Oe horizon across the pit face. Measuring tape shows inches and centimeters. White markers indicate horizon boundaries.

*Photograph taken by G.T. McPherson*



Measurement imprecision in estimates of organic matter mass limits the ability to detect change that may occur from climate change and other factors (Yanai et al. 2003). Therefore, the results obtained by sampling with the small diameter corer were compared with the results from pit excavation. In this comparison, forest floor samples (Oe plus Oa/A horizons) were collected with pit excavation and coring at 15 locations per watershed. Because forest floor samples had been collected with pit excavation at 18 locations, 15 of these measurements were randomly selected so that the sample sizes of pit excavation and coring were equal. For the 18 measurement comparisons presented in Table 8, values of CV were lower for core data than pit data in all cases except organic matter in South Buck, and C in South Buck and watershed 29012. The CV for core measurements averaged 9.0%, 3.8% and 5.6% less than pit measurements for organic matter, C and N, respectively.

**Table 8. Forest Floor Coefficient of Variation and Mean Values**

Measurements determined by pit and core sampling are presented by watershed for OM (organic matter), C (organic carbon) and N (nitrogen). Coefficient of variation (CV) and mean values were based on individual samples collected at 15 locations within each watershed.

	<b>CV OM (%)</b>		<b>Mean OM (Mg ha<sup>-1</sup>)</b>	
<b>Watershed</b>	<b>pit</b>	<b>core</b>	<b>pit</b>	<b>core</b>
27020	43.5	31.9	161.7	45.9
28014	26.9	14.9	115.2	46.7
28030	31.1	23.2	81.2	37.3
29012	31.3	27.3	85.8	35.3
North Buck	49.6	30.5	222.0	53.8
South Buck	31.6	32.3	68.7	34.7
mean	35.7	26.7	122.4	42.3
	<b>CV C (%)</b>		<b>Mean C (Mg ha<sup>-1</sup>)</b>	
<b>Watershed</b>	<b>pit</b>	<b>core</b>	<b>pit</b>	<b>core</b>
27020	37.7	34.0	72.1	23.7
28014	30.1	21.1	60.9	21.0
28030	37.9	28.6	38.3	19.7
29012	31.1	38.1	42.9	16.8
North Buck	49.9	37.9	124.0	26.0
South Buck	36.0	40.2	30.9	15.6
mean	37.1	33.3	61.5	20.5
	<b>CV N (%)</b>		<b>Mean N (Mg ha<sup>-1</sup>)</b>	
<b>Watershed</b>	<b>pit</b>	<b>core</b>	<b>pit</b>	<b>core</b>
27020	38.0	28.7	3.42	1.02
28014	32.8	18.9	2.81	0.87
28030	39.5	36.0	1.77	0.84
29012	32.1	30.5	2.08	0.74
North Buck	36.9	33.7	5.20	0.84
South Buck	37.0	35.2	1.64	0.61
mean	36.0	30.5	2.8	0.8

Organic matter mass of the forest floor was previously determined in 2005/2006 by Bedison et al. (2010) at 54 sites distributed throughout the Adirondack ecoregion (Bedison and Johnson 2009) by excavating the forest floor (Oe plus Oa horizon) down to the mineral soil within a 0.5 m<sup>2</sup> frame attached to the soil surface. The mean value of organic matter mass determined by Bedison et al. (2010) equaled 88.3 Mg ha<sup>-1</sup>, which was somewhat lower than the average of 122 Mg ha<sup>-1</sup> listed in Table 8. In addition to the differing methods of soil collection, the difference in estimates may be related to the small sample size of four available in this study, which would not effectively capture random variability across the Adirondack region, and possibly also the 10- to 12-year time difference between sampling in the two studies.

An even larger difference was observed when the organic matter mass determined by the ADK pit sampling was compared to the core sampling. The ADK sampling method resulted in estimates that exceeded those obtained from coring by a factor of approximately three (Table 8). The mean value reported by Bedison et al. (2010) was greater than the average from coring by a factor of approximately two (Table 8). The lower values determined by the coring method rather than the pit method is likely to be related, at least in part, to how sampling locations were selected in the two approaches. To enable pit excavation, locations for pit sampling were selected that avoided (1) areas that included rock exposed at the surface, (2) areas near the boles of trees to avoid large anchor roots, (3) excessively steep slopes, and (4) excessively thick understory. These restrictions did not apply to coring. This bias in selecting sampling locations may have led to lower estimates of forest floor organic matter from coring because the four areas not suitable for pit excavation will tend to have thinner forest floors. The coring data may therefore better reflect forest floor organic matter on a watershed basis.

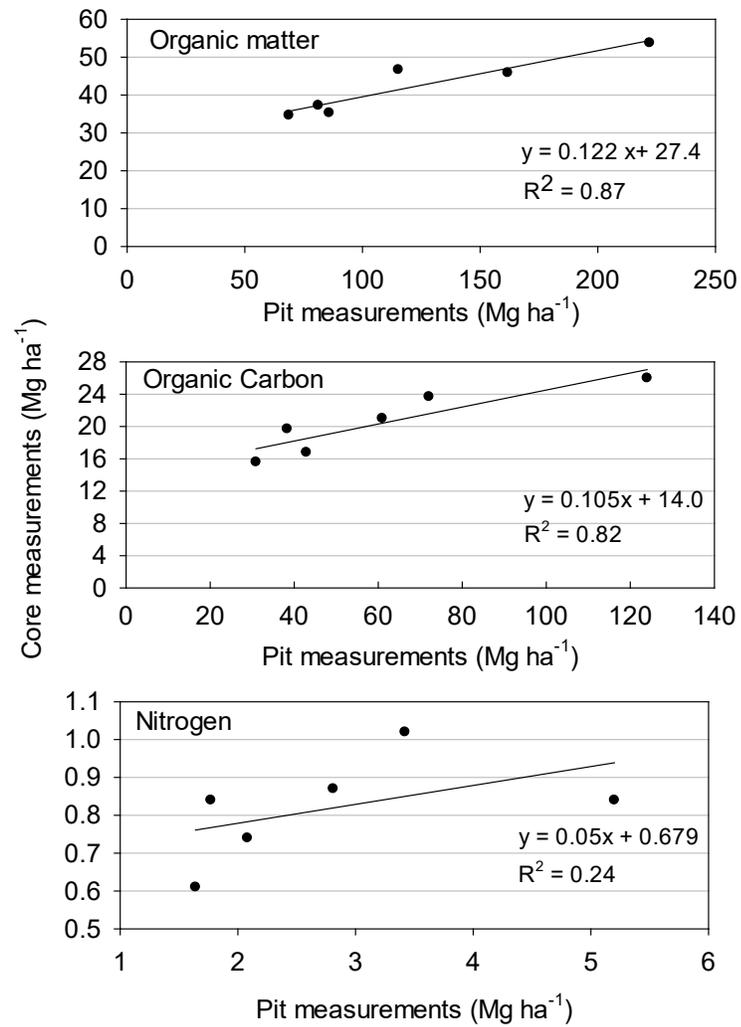
Differences in measurement methods may also contribute to lower estimates from coring than pit excavation. Measurements of the thickness of an intact forest floor includes all the materials within the forest floor. Thick clusters of fine roots often compose a large fraction of the forest floor volume and therefore contribute to the horizon thickness measurement required to estimate forest floor mass with the ADK sampling approach. With the coring method, forest floor thickness and bulk density values are not needed. This factor may contribute to the difference in the estimates of forest floor mass obtained by coring and by pit sampling.

Differences in how forest floor is collected may also contribute to differing estimates of organic content of the forest floor. Ideally, in the ADK approach the Oe and Oa/A horizon samples needed for chemical analysis are collected from the pit face where the thickness measurement is taken. However, commonly in Adirondack soils, these horizons are too thin to collect enough sample for chemical analyses from just the pit face. Therefore, additional areas near the pit are sampled from the surface. The additional sample is collected where the forest floor is thick enough to avoid mixing with mineral soil, thereby imparting a bias. Areas with thicker forest floor depth are likely to have a higher concentration of organic matter than at the pit face. The result is the collection of forest floor samples with concentrations biased high, which can lead to an overestimation of forest floor mass on both a plot and watershed basis. This sampling bias that occurs in pit sampling but not core sampling represents a factor that likely contributed to the differing estimates of forest floor mass between the two methods.

As discussed in the preceding paragraphs, coring and pit sampling yielded estimates that differed considerably, but these differences were found to be proportional between methods for the six watersheds where both methods were used. The organic matter estimates from coring and pits were significantly related ( $P < 0.01$ ) and highly correlated when watershed values were compared (Figure 5). Relations for C were also significant ( $P < 0.05$ ) with a similarly high correlation but were not significant for N ( $P > 0.10$ ). The lack of correlation for N measurements appears to reflect differences in C to N ratios among the watersheds.

**Figure 5. Forest Floor Mass Measurements Estimated by Coring and Pit Excavation**

Symbols represent watershed averages.



## 5 Summary and Recommendations

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Results of this analysis support the use of the ADK pit sampling design for long-term monitoring of changes in chemical concentrations in Adirondack soils. The sample size of 18 per watershed (six per study area) area was shown to provide sufficient statistical power to detect ecologically relevant changes in Adirondack soils. The rate of increase in power diminished with sample sizes greater than about 18. Therefore, a sample size of 18 provides a practical balance between the need for sensitive detection capability and costs of increasing sample sizes. Combining individual samples collected from pit excavation to decrease laboratory costs substantially lowered the statistical power for detecting changes. Because the primary goal of soil monitoring is to track changes in soils, combining samples collected with pit sampling is not recommended. However, the ease of sample collection with the small diameter corer makes it feasible to collect replicate samples that can be combined before analysis while maintaining a sample size that provides sufficient statistical power.

The power analysis suggested that a sample size of 18 would provide adequate statistical power for detecting concentration changes, but this analysis did not incorporate the time interval of resampling. North Buck and South Buck soil data were useful for comparing actual changes in Adirondack soils detected with sample sizes of 18 and 28 over sampling intervals of 12 to 16 years. By including three horizons from two watersheds with distinctly different soils this analysis provided a robust test of sample size on Adirondack soils. In general, a sample size of 18 yielded results highly similar to a sample size of 28, both in terms of change detection (the number of detected changes based on  $P > 0.10$ ) and uncertainty in detecting change (the value of  $P$ ). The only possible exception to this would be the North Buck Oa horizon where the number of changes detected were six with  $n = 18$ , and nine with  $n = 9$ . However, in South Buck the number of changes detected in this horizon with  $n = 18$  was only one less than with  $n = 28$ . The difference in results between the two sample sizes was not great enough to warrant the sample size of 28. However, the sampling design in the Buck Creek watersheds was based on transects throughout the watershed rather than sampling within three designated study areas, as was done in ADK sampling. Because the landscape variability in the Buck watersheds was largely defined by hillslope position, sampling upper, mid, and lower hillslope study areas would have likely detected similar changes and related to stream chemistry in a manner similar to the transect sampling design. On the basis of this analysis, future resampling of the two Buck watersheds could

be conducted with the smaller sample size of 18 without impairing the overall record or significantly weakening the sensitivity for detecting change. If watersheds contain more than three distinct landscape types, sampling of additional study areas (six additional samples per study area) is recommended for full watershed characterization.

The sampling approach of combining individually collected samples before analysis, which was used in the original sampling of WASS watersheds, was found to substantially reduce statistical power and is not recommended for soil monitoring. However, some significant changes ( $P < 0.10$  to  $P < 0.05$ ) in individual watersheds were detected when data collected with the WASS method were related to resampling data obtained with the ADK method. Further value would be gained by resampling of other WASS watersheds to enable grouping of watersheds for the purpose of regional assessment. Significant changes in upper B horizon Ca ( $P < 0.10$ ) and Oa horizon Al ( $P < 0.01$ ) were detected when the data from the four WASS watersheds were combined with the two Buck watersheds.

Limited data were available to evaluate relations between depth in the soil profile and spatial variability. Data from the 12 soil pits in which the ADK full-soil profile sampling method was used showed no difference in variability among the upper, mid, and lower B horizons. Because important information including pool sizes and changes over time may occur deeper in the profile, inclusion of deep profile samples in soil monitoring is recommended. However, based on the additional cost and disturbance to the study area that occurs with deep profile sampling, the use of greater replication for deep profile measurements may not be feasible in all situations.

Limited data were also available to assess the effectiveness of ADK sampling in relating to stream chemistry at the base of the watershed. However, three of the four watersheds sampled with the ADK method did fit the Adirondack relation between soil base saturation and BCS of stream water, and in the one watershed that did not exhibit that relation, extremely deep sand deposits provided a clear reason. The data available support the ADK sampling method as a method of characterizing the upper profile soil variability of the overall watershed.

Study results support small diameter cores as a useful method to monitor changes in the organic matter mass of the forest floor. This method showed high reproducibility in repeated sampling tests and lower spatial variability in sample data than obtained with pit excavation when compared on a watershed basis. For this reason, the sample size of 15 collection locations per watershed is considered adequate for small watershed characterization. Forest floor sampling with coring is also faster and requires less equipment

than pit excavation methods, which makes it more conducive to sampling over large areas. Furthermore, this approach can more effectively characterize an entire watershed than pit sampling because sample collection with cores can be done with little restriction, whereas pit sampling requires a location where pit excavation is feasible. However, organic matter mass of the forest floor determined by coring was consistently less than the values obtained by ADK sampling and literature values of a previous Adirondack study. The differences are attributable to where samples could be collected in the ADK method versus coring. Therefore, the organic matter content of the forest floor determined by coring should not be considered directly comparable to estimates determined by pit excavation. However, the consistency with which the two measures relate enable values to be adjusted for comparison, with some error introduced.

Based on the overall results of this study, combining the collection of forest floor cores with ADK pit excavation can provide better sensitivity in detecting changes in the forest floor at the same locations where full analyses of the upper soil profile are performed. This dual approach will document soil changes with direct measurements and help to understand the processes through which soils are changing.

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# Appendix A: ADK Sampling Field Protocol

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## A.1 Study Unit Selection and Description

1. Select three areas (study units) that are roughly homogenous individually, and as a group, include the primary landscapes and or hillslope positions within the watershed. These will be numbered 1–3. The study unit should be representative of a single landscape type with regard to slope, hillslope position, aspect, vegetation, parent material, drainage and exposed rock. Choose a central point within the study area, bury a survey magnet one foot beneath the surface, then record the coordinates of that location on the field sheet. One main pit (labeled as "M#", with # as study unit number) and five satellite pits (labelled as "S#-&", with # as study unit number, and & as satellite pit number) will be excavated within the sampling area. The areal extent of the study unit should be sufficient to enable resampling with this design at least four more times. In the selection of sampling pit locations, avoid land surfaces that are of minor importance within the study unit, and therefore not representative of the study unit. Also avoid locations where sampling methods are not possible because of perennial wetness, excessive rocks at or near the surface or excessive density of trees.
2. Record the slope by hanging flagging or some other marker at eye level approximately 10 m directly downslope of the pit and measure the slope with a clinometer from approximately 10 m upslope from the planned pit location, sighting on the flagging. Record the compass reading (aspect) along the predominant downslope direction. Record the hillslope profile position (using terms in Schoeneberger et al. [2012], under Surface Morphology) and upon returning to the office, record the elevation of the main pit using the topographic geographic information system (GIS) coverage.
3. Identify the dominant common overstory trees and describe the understory vegetation (to the best of your knowledge) surrounding the pit. Take a digital photo of the understory from about 10 m downslope from the planned pit location, looking directly upslope.

## A.2 Excavation and Profile Description of the Main Pit

1. Select a location for the main pit and record its location on the field form with respect to the survey magnet location (distance and aspect).
2. Lay out a tarp (approximately 10 ft by 12 ft or 3.1 m by 3.7 m) adjacent to the location where the main pit is to be excavated. Place the screen over the tarp.
3. Choose one side of the planned pit (upslope side if possible) to protect from trampling and contamination during pit digging by covering with plastic garbage bag or something similar. This side will then be used for the profile description and sampling.

4. Begin excavating the pit by removing the forest floor (O horizon) with the shovel. If possible, keep the forest floor intact and place where it will not be mixed with mineral soil that is removed from the pit. Excavate the pit with the smallest footprint possible (usually about 0.5 to 1 m<sup>2</sup>).
5. Once you begin removing the mineral soil, shovel it onto the screen (1.27 cm square openings). Sieve the soil either with the shovel or by shaking the screen. Once the coarse fraction builds up on the screen, dump it onto the weighing tarp, then weigh and record on the field form in the “Rock mass” column. Repeat until pit excavation is finished, shaping the pit as rectangularly as possible for measuring the excavated volume. Measure and record dimensions of the pit as indicated on the field form. Take additional measurements if needed to enable the pit volume to be accurately estimated. Where large rocks extend into the pit, take three measurements of the rock extension (length, height, width) to estimate its volume then record next to the pit diagram.
6. Prepare a vertical pit face for description and sampling by lightly scraping downward with a hand trowel to remove any loose soil resulting from the excavation. Prune roots with hand snippers where necessary. If excessive rocks or roots preclude the clearing of a pit face for description and sampling, or reaching the desired depth, the pit may need to be expanded somewhat.
7. Record in the notes box on the field form any observations of water seeping into the pit from a pit face or the bottom of the pit.
8. Visually evaluate the pit face from top to bottom for differences in color, texture, and structure. Remove small amounts of the differing soil and place side by side on a white piece of paper (such as the back side of the field form) to assist in identifying horizon boundaries, as shown in Figure 3.
9. Record the horizon designations following pages 2-2 to 2-5 of the NRCS Field Book (Schoeneberger et al, 2012).
10. Mark horizon boundaries with T shaped pins or similar objects. Take a digital photo of the profile with horizon markers.
11. Measure and record the depth of the top and bottom of each horizon with a metric tape relative to the interface between the air and soil surface.
12. Record the color of each horizon using the Munsell Soil Color Book following pages 2-8 to 2-11 of the NRCS Field Book (Schoeneberger et al, 2012).
13. For each horizon, record the texture class (pages 2-36 to 2-37) and structure type (pages 2-52 to 2-54), following the instructions in the NRCS Field Book (Schoeneberger et al, 2012). Also, for each horizon, indicate whether fine roots (<2 mm diameter) are abundant, common, few, or none.

### A.3 Sample Collection from Main Pit and Satellite Pits

1. Select the horizon from which soil will be collected. This will normally include the following:
  - Oe
  - Oa or A
  - the uppermost 10 cm of the B (this may include more than one subhorizon)
  - a 10 cm section that occurs at approximate mid depth of the B
  - a 10 cm section from the lowermost 10 cm of the B

The entire thickness of the Oe and Oa or A should be collected. If the Oa or A is greater than 20 cm thick, collect equal volumes from the uppermost 10 cm and the lowermost 10 cm. Where present, the E and C horizon may be also be collected depending on objectives. For each horizon to be sampled, insert pins across the pit face to define the layer from which soil will be collected.

Fill in the label on the sample bag with (1) watershed name or code, (2) pit identification, (3) date, (4) sampler name, (5) sampled horizon, (6) sampled depth interval (cm) as measured from the surface of the Oe, and (7) project code. Leave SSN blank.

1. Start by collecting the deepest sample and working upward.
2. Collect soil across the breadth of the pit face where sampling can occur (no rocks or roots in the way). To remove the sample from the pit face, insert the gardening trowel near the bottom of the sampling layer. Then insert a flat trowel above the gardening trowel to loosen the soil so that it can be removed with the bottom trowel. Collect approximately half a bag of mineral soil, two-thirds of a bag, with Oa or A, and a full bag with Oe. Double-bag samples if soils are stony.
3. Once sampling is completed, backfill the pit with the mineral soil and coarse fragments. Place forest floor on top of the mineral soil, keeping the organic material as intact as possible. Record the location of the pit on the field form with respect to the study unit monument (distance and aspect).
4. Repeat main pit sampling in the remaining study units.
5. Excavate five satellite pits within each of the three study units. Collect only Oe, Oa and upper 10 cm of the B horizon from each pit using the same procedures as used for the main pit. Record depths of all horizons present down to the upper 10 cm of the B. If there is more than 10 cm of B, indicate as > bottom depth of the upper 10 cm. Record the Lat/Long of each pit on the field sheet.

# Appendix B: Forest Floor Core Sampling Field Protocol

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1. Before collecting the soil core, the following information should be recorded on label tape placed on the sample tube:
  - Watershed
  - Plot\_Transect number
  - Subplot number
  - Replicate letter (*a*, *b*, or *c*)
  - Date
2. Place a tube in the corer.
3. Select any location to sample where there is not evidence that water at the surface occurs for sustained periods. Overly wet areas can be identified by (1) understory vegetation that differs from adjacent well-drained areas, (2) depressions that allow for collection of surface water, and/or (3) channelization from surface flow has developed. Also avoid sampling close (less than 1 m) to where rock is exposed at the surface.
4. Insert the corer into the ground, pushing it down as deep as possible to collect the sample. Start over if you think you are hitting a large root. Do not start over when you hit a subsurface rock because in most cases, there will not be organic soil below the rock. This will be replicate core *a*.
5. Push the sample out of the corer tip into the sample tube using the wooden dowel. If no soil is collected (this happens rarely) move a few m away and try again.
6. Remove the liner (without spilling soil) then place caps on the top and bottom. Tape caps if they are loose.
7. Use the brush to clean out the corer.
8. Repeat steps two through seven to collect two more cores (replicates *b* and *c*) within 1-meter radius of the first core.



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