Published February 25, 2016

Method Comparison for Forest Soil Carbon and Nitrogen Estimates in the Delaware River Basin

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The accuracy of forest soil C and N estimates is hampered by forest soils that are rocky, inaccessible, and spatially heterogeneous. A composite coring technique is the standard method used in Forest Inventory and Analysis, but its accuracy has been questioned. Quantitative soil pits provide direct measurement of rock content and soil mass from a larger, more representative volume. In this study, the two sampling methods were used to estimate soil C and N stocks in forested plots in the Delaware River Basin. Mean stocks in the whole soil profile (organic and mineral layers, 0–40 cm, using pits) were 76.6 Mg C ha^{-1} and 4.45 Mg N ha^{-1}. In the surface mineral layer (0–20 cm), lower bulk density (BD), lower coarse fragment content (CF), and greater C concentration (%C) were measured using the core method compared with the pit method. However, because the three variables are not independent and can be counterbalancing, soil C stocks did not differ between sampling methods. Spatial variation in C stocks was mainly driven by the variance of %C and BD in both methods, while the relative contribution of CF was greater in the soil pit method. Our results suggest that the physical problems associated with the core method and the ability of the core method to capture spatial variation in soil C and N stocks are questionable compared with quantitative soil pits. While variability and covariance among the contributing variables resulted in similar stock estimates from both sampling methods, they might accumulate greater uncertainty in spatial extrapolation to regional estimates of forest soil C and N stocks.

Abbreviations: BD, bulk density; CF, coarse fragment content; DEWA, Delaware Water Gap Area; DRB, Delaware River Basin; FC, French Creek; FIA, Forest Inventory and Analysis; NS, Neversink River Basin.

Soils represent the largest C pool and a highly dynamic component of the terrestrial C cycle (Batjes, 1996). Quantifying forest soil C and N stocks is critical to understanding the ecological responses of forests to changes in climate, land use, and management and to improve global change models (Smith et al., 2012; Dib et al., 2014). However, the accuracy of forest soil C and N estimates is hampered by the difficulty in characterizing forest soil properties because forest soils can be rocky, inaccessible, and spatially heterogeneous (Qureshi et al., 2012; Lark et al., 2014). In addition to this inherent variability, there is large uncertainty in forest soil C and N estimates associated with the soil sampling methods used (Gifford and Roderick, 2003; Jandl et al., 2014).

Forest Inventory and Analysis (FIA) provides a national database of forest soil C and N that has been broadly used in large-scale soil C and N estimations (Conkling et al., 2002; O'Neill et al., 2005). A composite coring technique is the standard soil sampling method in FIA, as well as in the national Forest Health Monitoring program (Conkling et al., 2002; Palmer et al., 2002). However, the accuracy of the soil core method has been questioned by several studies (e.g., Throop et al., 2012; Chimner et al., 2014). Several reports have compared sampling meth-

Core Ideas:

- **Soil cores and quantitative pits each have their limitations in forest soils.**
- **Bulk density, coarse fragment, and C concentration variations are method dependent.**
- **Compensating errors may mask uncertainty in regional estimates of soil C and N.**

Soil Sci. Soc. Am. J. 80:227–237
doi:10.2136/sssaj2015.04.0167
Received 30 April 2015.
Accepted 4 Oct. 2015.
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ods and found significant differences in measured variables (e.g., bulk density, soil C stocks, etc.) among them when used in forest soils, which are typically rocky and uneven with high spatial variation in soil development, horizon depth, and rock content (Page-Dumroese et al., 1999; Harrison et al., 2003; Johnson et al., 2012).

In the soil core method, soil C and N stocks are calculated using bulk density (BD), coarse fragment content (CF), soil C (%C) and N concentrations, and soil depth. Several problems with the soil core method may result in under- or overestimation of these three variables and therefore induce errors in soil C and N stock estimations (Table 1). First, when the core is punched into the soil, the soil inside and beneath the core can be compacted. If the compaction happens only inside the core, soil in the bottom of the core will have a greater density and there will be an empty space at the top of the core. The values of BD, %C, and CF will not be affected because we consider the volume of the whole core as the volume of the soil (Lichter and Costello, 1994). If the soil beneath the core is compacted during sampling, however, part of the soil that should be sampled would be missed. This problem will lead to underestimation in BD and CF (Harrison et al., 2003). Because the missed soil is always deeper soil and %C typically decreases with depth, the soil sample will overemphasize the surface soil and the %C will be overestimated. Second, the soil core could be obstructed by rocks or coarse roots in the soil. If the coring stopped above the rock but there is still soil beneath the rock, the %C will be overestimated because subsoil is missed. The CF will be overestimated, although the BD will not be affected. However, if the impact force of the core extruded the rocks and deep soil, leading to an overestimation of the soil volume (Levine et al., 2012), then the volume-based BD and CF will be underestimated (Page-Dumroese et al., 1999; Harrison et al., 2003). Third, locations with fewer rocks within the sampling frame are more likely to be sampled in the core method. This will result in an underestimation of the CF (Kulmatiski et al., 2003; Jandl et al., 2014). These problems could combine, leading to a large uncertainty in the soil C and N stock estimates, or may be counterbalancing, with no observable effect on the estimate. In spite of the limitations of this method, a literature survey has shown that the soil core is still the predominant method in soil C estimation (Throop et al., 2012).

The quantitative soil pit method purportedly provides more accurate estimations of soil C and N stocks (Lyford, 1964). This method provides direct measurement of rock content and soil mass from a larger, more representative volume of soil (Vadeboncoeur et al., 2012). There are fewer limitations for the location of a quantitative pit because it does not need to avoid the presence of rocks. In addition, deeper portions of the soil profile are more accessible in the pit method than the conventional coring method. Soil pit excavation was considered as the preferred soil sampling method in many studies evaluating methods in measurements of soil properties (e.g., Harrison et al., 2003; Kulmatiski et al., 2003; Levine et al., 2012). However, because the soil pit method is so labor intensive, fewer pits than cores are likely to be sampled across the same landscape area and therefore the spatial variability in soil properties may not be adequately characterized.

Table 1. Main problems associated with the soil core sampling method and their effect on the soil properties of bulk density (BD), C concentration (%C), coarse fragment content (CF), and C stock. Possible effects include no effect (–), overestimate (↑), underestimate (↓), or uncertain effect (?).

Problem		BD	%C	CF	C stock
Compaction	inside core	–	–	–	–
	beneath core	↓	↑	↓	?
Obstruction by rock	missing soil under rock	–	↑	↑	?
	compression by rock	↓	–	↓	?
Plot selection		–	–	↓	↓

Estimation of soil C and N stocks requires resolving the variables of BD, CF, soil C and N concentrations, and soil depth. The choice of sampling method may bias each variable in different ways. In addition, the variables may contribute differently to the spatial variation of soil C and N stocks in different sampling methods (Holmes et al., 2011; Schrumpf et al., 2011). Therefore, a comprehensive comparison between sampling methods, particularly the widely used soil core method and the standard quantitative soil pit method, considering all variables and their contributions to the spatial variation, is of fundamental importance for our prediction ability in estimating large-scale forest soil C and N stock changes.

The Delaware River is one of the major rivers in the Mid-Atlantic region of the United States, draining an area of about 33,000 km^2 in Pennsylvania, New Jersey, New York, Delaware, and Maryland. The Delaware River Basin (DRB) represents the major physiographic provinces, ecozones, and soil types of the Mid-Atlantic region. It contains several intensive monitoring and research sites maintained by the US Forest Service, with long-term research on ecological components and processes (Murdoch et al., 2008). The diverse geologic, topographic, and ecological properties of the DRB and ongoing monitoring projects provided a good opportunity to explore the metrological uncertainty of soil C and N content at a regional scale.

In this study, the FIA standard soil core method and the quantitative soil pit method were used to estimate soil C and N stocks in three research sites in the DRB forest. The major goals of this study were to: (i) quantify soil C and N stocks to 20 cm using the FIA soil core method and to 40 cm using the soil pit method in the DRB forest plots; (ii) compare soil properties measured by the soil core and soil pit methods in the 0- to 20-cm mineral soil layer; and (iii) compare the contribution of variables to the spatial variation of soil C and N stocks in the two sampling methods.

METHODS

Study Area and Plot Design

The DRB is characterized by a humid continental climate, with a mean annual temperature of 9 to 12°C and mean annual precipitation of 1143 mm (Thornton et al., 2014). The DRB is located in the ecozone of deciduous forests and is ecologically diverse, comprised of five physiographic provinces and multiple species assemblages that represent most of the major eastern US forest types (Murdoch et al., 2008).

Three sites in the DRB were selected as intensive research sites (Fig. 1; Table 2). They are the Neversink River Basin (NS) in the northern, mostly forested region of the Appalachian Plateau province, the Delaware Water Gap Area (DEWA) with three small watersheds (Adams Creek, Dingman's Falls, and Little Bushkill) lying in the central Appalachian Plateau province; and the French Creek (FC) watershed in the mid-basin Piedmont province. The dominant soil suborders are Ochrepts in NS and DEWA and Udults in FC (Soil Survey Staff, 2014).

In total, 60 forested plots were randomly located across the three research sites (14 in NS, 32 in DEWA, and 14 in FC). The plots were established in 2001 to 2003 by the US Forest Service and US Geologic Survey and designed according to FIA and Forest Health Management protocols (US Forest Service, 2004). Each plot had four round subplots, covering a total area of 672 m^2 (Fig. 2). In each of the plots, three soil cores (0–20 cm) and one quantitative soil pit (0–40 cm) were sampled at systematically located positions as described below. If the locations for sampling landed on unforested areas (e.g., road, water body, farmland, etc.), no sample was collected. All the soil samples in FC and DEWA and three plots in NS were

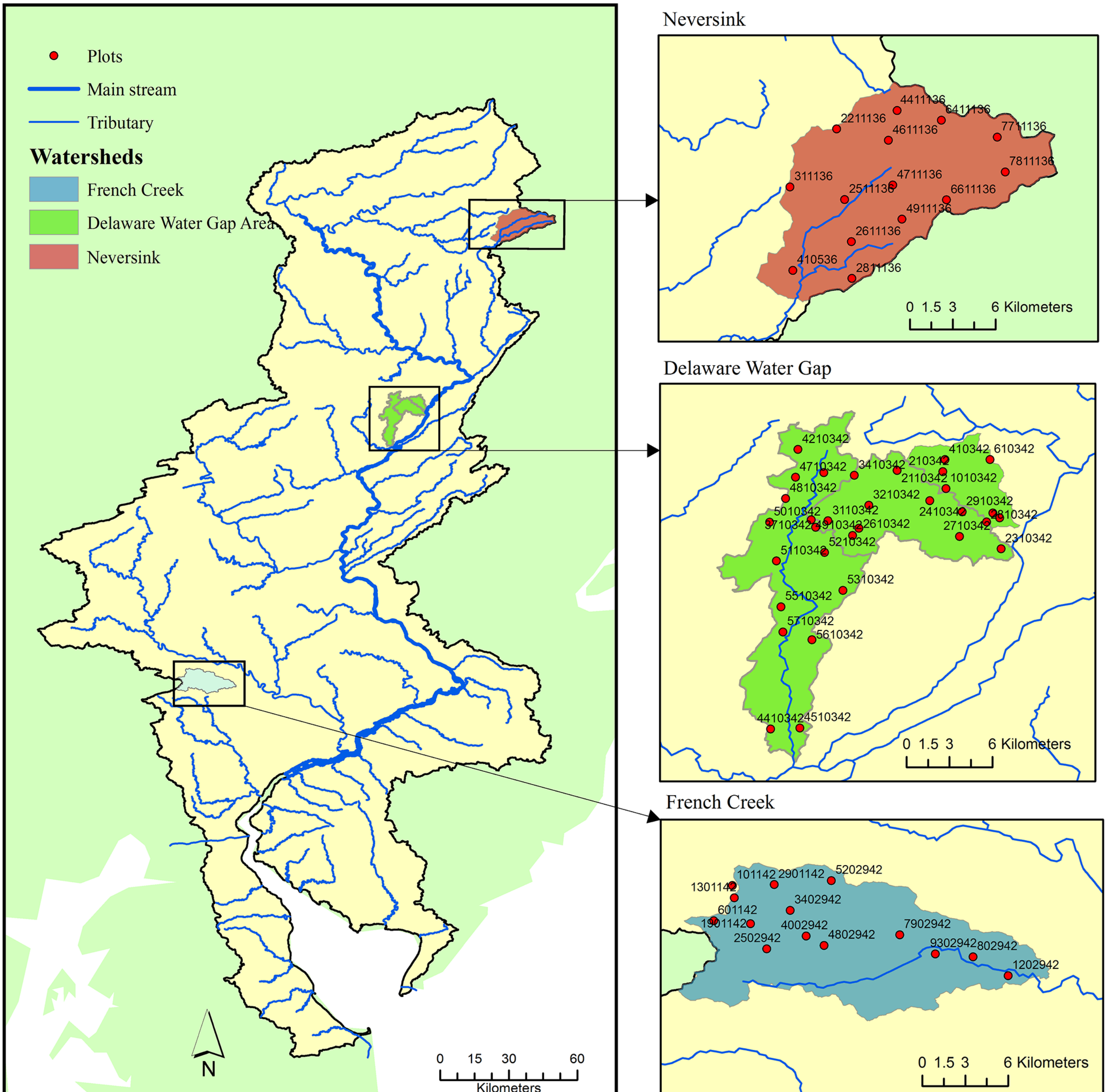

Fig. 1. The hydrological boundary of the Delaware River Basin and the main stream and tributaries of the Delaware River. The three research areas are shown in different colors. The red dots show the locations of soil sampling plots labeled by their plot ID.

Table 2. Environmental conditions at the three research sites in the Delaware River Basin. All data were extracted from a GIS database, and mean values for each site are shown. Annual temperature and precipitation are 30-yr means from 1981 to 2010 (Thornton et al., 2014). Wet deposition is inorganic N deposition from 1983 to 2007 (Grimm, 2008).

Site	Elevation	Mean annual temperature	Mean annual precipitation	Wet deposition	Dominant soil order	Dominant forest type
	m	°C	mm	kg N ha^{-1}		
Neversink River Basin	773	5.7	1503	6.4	Inceptisol	maple–beech– birch
Delaware Water Gap Area	360	8.5	1219	6.3	Inceptisol	oak–hickory
French Creek	166	11.2	1172	6.6	Ultisol	oak–hickory

collected in summer 2013, while the remaining 11 plots in NS were sampled in summer 2014.

Standard Forest Inventory and Analysis Soil Core Sampling Method

The standard FIA soil core sampling protocol was used to collect soil core samples (US Forest Service, 2004). Core samples were collected 9.14 m away from the center of Subplots 2, 3, and 4 (Fig. 2). In 2001 to 2003, soil samples had been collected at the location of Visit 1 in these DRB plots, so in this second measurement, the coring was located at Visit 2 of each subplot to avoid the disturbance of the previous survey. In the standard FIA protocol, only one mineral soil sample is collected at Subplot 2, while in this study, mineral soil samples were collected in each of the three subplots (Fig. 2) to get more representative samples (i.e., a larger area) to compare with the soil pit method.

The organic layers within the frame of a bicycle tire (30.5 cm in diameter) were cut by saw and collected into multiple sample bags. Mineral soils were sampled using an impact-driven soil corer (5 cm in diameter, 20 cm in length) with two internal steel liners (10-cm length each). The core was driven into the ground until the liner was even with the soil. The core was slowly rotated and carefully removed from the ground. The two liners were removed from the top of the core, and the soil in the liners was removed and collected into separate sample bags. If the soil core was obstructed by a rock or large root, the actual depths of the two layers were recorded and the space beneath was assumed to be occupied by rocks. If the mineral soil horizons were shallower than 3 cm, no mineral soil sample was collected for that subplot, and samples from the remaining two subplots were used. Although the method can be used to sample deeper soils, the coring was performed only to 20 cm to comply with FIA standard methods.

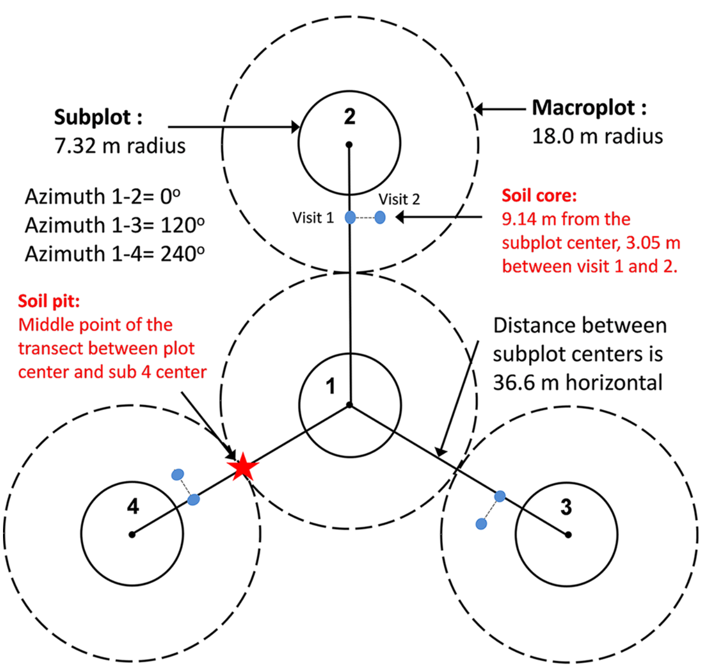

Fig. 2. Plot design of forest measurement and soil sampling (revised from US Forest Service, 2002). The red star represents the location of the quantitative soil pit. The Forest Inventory and Analysis protocol used Visit 1 and Visit 2 (represented by blue dots) as the sampling locations in each of the subplots. The survey in 2012–2013 was the second measurement of these plots, so the Visit 2 location of each subplot was selected for the soil core.

Quantitative Soil Pit Sampling Method

The middle point between the plot center and Subplot 4 was selected as the location for the quantitative soil pit in each plot (Fig. 2). If this location was not forested (e.g., located on agricultural land or on a road) or had a tree or rock outcrop within the sampling area, the same location in Subplot 2 or Subplot 3 was selected.

A 50- by 50-cm square frame was secured to the ground by four nails on the outer edge to control the area of the pit. All living vegetation within the sampling area was removed. The entire layer of loose leaf litter (Oi horizon) and the underlying organic matter (Oe + Oa horizons) were removed and weighed separately in the field. A subsample of ~0.3 kg was collected and returned to the laboratory for subsequent analyses. The mineral soil was excavated to depths of 0 to 20 and 20 to 40 cm. For each of the soil layers, the actual depths were measured at the middle points of the four pit edges. Soil, rocks, and roots were separated, then weighed. A subsample of well-mixed soil (~1 kg) from each layer was collected and returned to the laboratory for subsequent analyses. To measure the volume of the soil, known volumes of perlite (measured in a graduated cylinder) were poured into the pit until the pit was filled to the depth of the upper horizon. The density of perlite in the cylinder was found to be similar to its density when poured into the pit. All rocks and roots that had been excavated were placed back into the pit during this process and submerged in the perlite. The volume of the perlite therefore represented the actual volume of the excavated soil. When the volume of rocks was too large, or there were too many small rocks in the pit, the portion of rocks that could not be submerged into the perlite was not placed back into the pit but was instead weighed. The volume of the rocks was then calculated using a standard rock density of 2.65 g cm^{-3} (Telford et al., 1990) and was subtracted from the volume of the perlite.

Soil Carbon and Nitrogen Analyses and Carbon and Nitrogen Stock Calculations

Organic layer samples were oven dried at 105°C for >12 h, then ground to <2 mm. Mineral soil samples were air dried and sieved through a 2-mm screen. Twenty mineral samples representing the two soil layers distributed among the three research sites and collected by both methods were oven dried, and a correction curve between air-dry and oven-dry soil weights was generated (oven dry = 0.9845 × air dry, R^2 = 0.999). Oven-dry weights of the mineral samples were estimated using this curve. An aliquot from each sample was ground by mortar and pestle to pass a 250-μm sieve to increase sample homogeneity. Ground samples (15 mg for mineral soil and 5 mg for organic soil) were analyzed for total C and N concentrations (g kg^{-1}) using an ECS4010 elemental analyzer (Costech). Approximately 11% of the samples (n = 71) were measured twice to verify the precision of the C and N analyses. These analytical replicates showed a precision in line with instrumental error (i.e., a coefficient of variance of ∼5% for both C and N). Soil survey data for these regions indicates a lack of carbonates and soil pH values of 4.0 to 7.5 (Soil Survey Staff, 2014). We therefore equated total C concentrations to organic C. The C/N ratios were calculated as the mass ratio between C and N concentrations.

For organic layers, soil masses were determined as the oven-dry weight divided by the sampling area. If all three subplots were sampled, the sampling areas were 731 cm^2 × 3 subplots for a total of 2193 cm^2 in the soil core method and 2500 cm^2 in the soil pit method. Carbon and N stocks (kg m^{-2}) were calculated by multiplying the soil mass (kg m^{-2}) by the C and N concentrations (g kg^{-1}).

For mineral soil layers, the soil BD was defined as the density of the soil after subtracting the weight and volume of rocks and roots. It was calculated as the oven-dry weight of the 2-mm sieved soil divided by the volume of the soil. In the soil core method, the volume of the soil was taken as the volume of the core minus the volume of rocks collected through sieving (>2 mm). In the soil pit method, the volume of the soil was taken as the volume measured by perlite minus the volume proportion of rocks measured in the sample during sieving. Because the soil mass (kg m^{-2}) can be measured directly using the quantitative pit method, the determination of soil BD is not strictly necessary. However, as noted below, we sought to compare how multiple variables such as BD contributed to the variability in both methods and thus needed the values. By definition, the coarse fragment content (CF) soil fraction was the volume percentage of rock and roots >2 mm in diameter. It was equal to one minus the volume percentage of soil in the soil core or pit. In the soil pit method, the actual depths measured in the field were used to calculate the total volume. Because coarse roots accounted for very little volume in the soil, CF mainly consisted of the soil rock content. Using the BD, depth, CF, and C and N concentration data for each mineral soil layer, the soil C stock was calculated as

$$\text{C stock} = \text{BD} \times \text{depth} \times (1 - \text{CF}) \times \%\text{C} \qquad [1]$$

N stocks were calculated using the same equation with N concentration.

Because the major objective of the study was to compare estimates between the core and pit methods in the 0- to 20-cm depth increments, BD, CF, and %C for the two depth increments (0–10 and 10–20 cm) in the soil core method were combined using the actual depth as their weighting. Soil mass and C and N stocks (0–20 cm, g m^{-2}) were calculated as the sum of the two depth increments.

Data Analysis

We performed three statistical tests on the experimental data. First, soil pit C stock (Mg C ha^{-1}) and C/N ratio data for each layer (Oi, Oe + Oa, 0–20 cm, 20–40 cm, and total) were tested for differences among the three research sites (NS, DEWA, and FC) using one-way ANOVA, followed by post-hoc comparisons between sites when the main effect was found to be statistically significant using the nonparametric Wilcoxon Each Pair test in JMP Pro Version 12 (SAS Institute). Second, to test for differences between the two sampling methods, BD, CF, soil mass, C and N concentration, C and N stock, and C/N ratio data for the calculated 0- to 20-cm layer from the three soil cores in each of the plots were averaged, and mean values were compared with the single pit values for each plot. As the data were not normally distributed, the nonparametric Wilcoxon signed-rank test was used to test the difference of the paired values of the two methods. Third, Type II regression analysis was used to test for correlations among these soil properties measured using the two sampling methods.

To evaluate the spatial variability in the assessment of soil C stock, an error propagation approach based on the linear Taylor series expansion (Goidts et al., 2009) was used to separate and quantify the contribution of variance from each input variable. In Eq. [1], soil C stock was calculated as the product of BD, depth, soil volume (1 − CF) and C concentration (%C). Each of these variables has its own associated measurement error as well as covariance, since the variables are not necessarily independent from each other. Soil depth was fixed in this study so it was not included as a source of error. The total variance of C stocks in the 0- to 20-cm mineral soil for each sampling method and research site can then be apportioned into the following terms including variances of single factors and covariances between factors:

$$\begin{aligned}\text{Var}(\text{C stock}) = {} & (\text{C stock})^2 \Bigg[\frac{\sigma^2_{\text{BD}}}{\text{BD}^2} + \frac{\sigma^2_{\%\text{C}}}{\%\text{C}^2} + \frac{\sigma^2_{1-\text{CF}}}{(1-\text{CF})^2} \\ & + 2\frac{\sigma_{\text{BD}\times\%\text{C}}}{\text{BD}\times\%\text{C}} + 2\frac{\sigma_{\text{BD}(1-\text{CF})}}{\text{BD}(1-\text{CF})} \\ & + 2\frac{\sigma_{\%\text{C}(1-\text{CF})}}{\%\text{C}(1-\text{CF})} \Bigg] \end{aligned} \qquad [2]$$

where σ_{BD}, $\sigma_{\%C}$ and $\sigma_{(1-CF)}$ are standard deviations of BD, %C and (1 − CF); $\sigma_{BD\times\%C}$, $\sigma_{BD(1-CF)}$ and $\sigma_{\%C(1-CF)}$ are their covariances, some of which could have negative values. The denominators are the means of the variables. Standard deviations and covariances were directly calculated from the measured data sets for each method and for each site as well as all sites combined. To calculate the relative contribution of each variable, each term in the brackets was divided by the sum of absolute values of each term in the brackets in Eq. [2] and expressed as a percentage. For instance, the relative contribution of BD is $\sigma_{BD}{}^2/BD^2$ divided by $\{\sigma_{BD}{}^2/BD^2 + \sigma_{\%C}{}^2/\%C^2 + \sigma_{(1-CF)}{}^2/(1-CF)^2 + |2(\sigma_{BD.\%C}/BD{\cdot}\%C)| + |2[\sigma_{BD(1-CF)}/BD(1-CF)]| + 2[\sigma_{\%C(1-CF)}/\%C(1-CF]|$. Positive values in the numerator result in positive relative contributions, while negative values in the numerator (e.g., covariance) result in negative relative contributions.

RESULTS

Soil Carbon and Nitrogen Content in Different Horizons

Using the FIA standard soil core method, the mean C stock in the organic layer was 12.8 Mg C ha^{-1} and the N stock was 0.52 Mg N ha^{-1}, with 40.8 Mg C ha^{-1} and 2.28 Mg N ha^{-1} in the 0- to 20-cm mineral layer (Table 3). Using the quantitative soil pit method, the mean C stock in the organic layer across all plots was 14.6 Mg C ha^{-1} and the mean N stock was 1.38 Mg N ha^{-1}. The C and N stocks in the 0- to 20-cm mineral layer were 44.7 Mg C ha^{-1} and 2.59 Mg N ha^{-1}, with 17.3 Mg C ha^{-1} and 1.26 Mg N ha^{-1} in the 20- to 40-cm mineral layer (Table 3). Thus, the mean total (0–40 cm) C and N stocks as measured using soil pits were 76.6 Mg C ha^{-1} and 4.45 Mg N ha^{-1}. The mean C/N ratio decreased from 31.1 in the surface Oi horizon to 14.0 in the deeper mineral horizon.

Among the three research sites, the NS site had the largest total C stock (99.8 Mg C ha^{-1}, $p < 0.01$) and N stock (6.04 Mg N ha^{-1}, $p < 0.01$) based on the soil pit method (Table 3). The C stock at the FC site was the smallest, mainly because of a thinner Oe + Oa horizon. The C/N ratio in the organic layer was relatively constant among the three research sites. At DEWA, the C/N ratio in the surface mineral soil was much greater than in the deeper mineral soil. However, at the NS site, the two mineral soil layers had very similar C/N ratios.

Table 3. Soil C and N stocks and C/N ratio sampled using soil core and soil pit methods at the three research sites in the Delaware River Basin. Sample size (*n*) represents number of plots sampled in each study site. In the soil core method, the organic layer was defined as the sum of Oi and Oe + Oa layers, and the C and N stocks measured in the two mineral layers (0–10 and 10–20 cm) were summed together. Significant level of ANOVA testing for the effect of sites on C and C/N of each layer in the pit method are labeled as "*", $p < 0.1$; "", $p < 0.05$.**

Soil layer	Horizon or depth	Soil cores: N stock	Soil cores: C stock	Soil cores: C/N ratio	Soil pits: N stock	Soil pits: C stock	Soil pits: C/N ratio
	cm	Mg N ha^{-1}	Mg C ha^{-1}		Mg N ha^{-1}	Mg C ha^{-1}	
All sites (n = 59)							
Organic layer	Oi	0.53	12.8	26.5	0.86	2.61†	31.1
	Oe + Oa				0.52	12.0	23.0
Mineral soil	0–20	2.28	40.8	18.9	2.59	44.7†	18.4*
	20–40				1.26	17.3*	14.0
Total		2.80	53.6	20.2	4.45	76.6*	18.0†
Neversink River Basin (n = 14)							
Organic layer	Oi	0.61	13.6	24.3	0.09	2.5 ab‡	30.6
	Oe + Oa				0.60	12.7	21.4
Mineral soil	0–20	2.89	48.3	17.1	3.50	55.5 a	16.0 a
	20–40				1.85	29.1 a	15.9
Total		3.50	61.9	18.1	6.04	99.8 a	16.9 a
Delaware Water Gap Area (n= 31)							
Organic layer	Oi	0.46	12.0	27.5	0.08	2.4 a	31.1
	Oe + Oa				0.55	13.4	23.6
Mineral soil	0–20	1.95	38.3	20.6	2.21	41.8 b	20.1 b
	20–40				1.01	13.6 b	13.9
Total		2.41	50.3	22.1	3.85	71.2 b	19.3 b
French Creek (n = 14)							
Organic layer	Oi	0.55	13.7	26.4	0.11	3.3 b	31.6
	Oe + Oa				0.32	6.9	23.2
Mineral soil	0–20	2.43	39.3	16.7	2.56	41.1 b	16.9 a
	20–40				1.32	15.9 b	12.6
Total		2.99	53.0	18.0	4.30	67.2 b	16.1 a

* Site effect significant at $p < 0.05$ according to ANOVA.
† Site effect significant at $p < 0.1$ according to ANOVA.
‡ Means followed by different letters are significantly different ($p < 0.05$) among each pair of sites.

Soil Properties Measured by the Two Sampling Methods

Comparisons of the two sampling methods were focused on the surface mineral soil layer (0–20 cm) because the deeper (20–40 cm) layer was not sampled using the core method and because CF is not a relevant issue in the organic layer. The results show that using the quantitative soil pit method, the mean soil BD was 0.93 kg L^{-1} in all the DRB plots combined. The mean BD measured from the FIA standard soil core method was 0.57 kg L^{-1}, a value approximately 40% smaller than the mean BD measured using the soil pit method (Table 4; Fig. 3a). However, the volume percentage of CF measured by the soil core method was also significantly less than the CF measured by the soil pit method (Wilcoxon test, $p < 0.01$). Therefore, the soil mass, which was calculated based on BD and CF, was still significantly greater for the soil pit method ($p = 0.002$). The difference in soil mass between the two methods was smaller than the difference in BD (Fig. 3a and 3c). In some plots, C and N concentrations were very large for the soil core method but not the soil pit method (i.e., the cluster of data points above the 1:1 line in Fig. 3d). The resulting soil C stock (0–20 cm) was estimated to be 40.8 ± 4 Mg C ha^{-1} for the core method compared with 44.7 ± 4 Mg C ha^{-1} for the pit method. No significant difference between methods was detected in C and N stocks (C stocks: $p = 0.12$; N stocks: $p = 0.14$), and the correlations between C and N stocks measured by the two methods were not significant (C stock: $r = 0.22$, $p = 0.096$; N stocks: $r = 0.39$, $p = 0.15$). The C/N ratios measured by the two methods were highly correlated ($r = 0.72$, $p < 0.01$) and the mean C/N ratio using the soil core method was slightly greater than that using the soil pit method ($p = 0.047$). Smaller BDs but greater C and N concentrations and smaller CF contents for the soil core method appear to have offset each

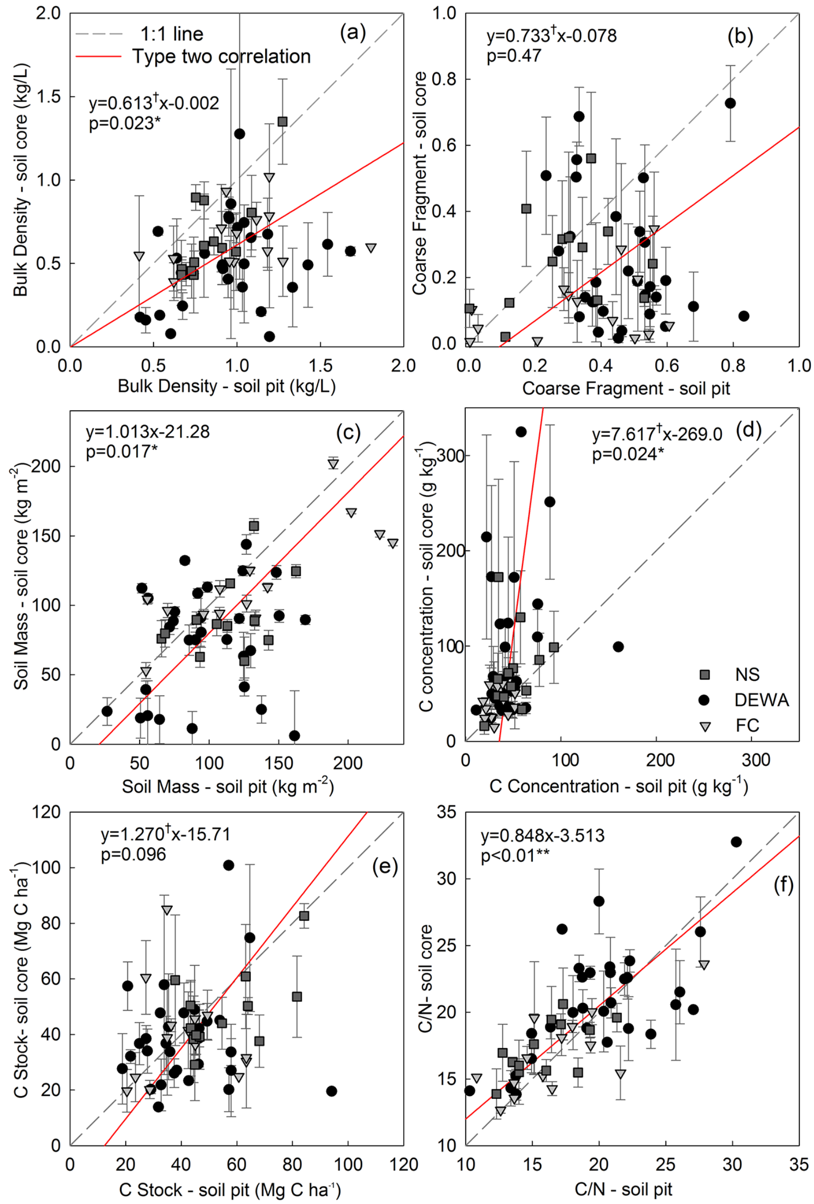

Fig. 3. Correlations of (a) bulk density, (b) coarse fragment, (c) soil mass, (d) C concentration, (e) C stock, and (f) C/N mass ratio measured by the soil core and soil pit methods at three sites: Neversink River Basin (■), Delaware Water Gap (●), and French Creek (▼). The 1:1 line and Type II (major axis) linear regression are shown. †Slopes are statistically different from 1. Vertical error bars represent the standard error among the cores (n = 3) vs. only one pit (i.e., no horizontal error bars).

Table 4. Comparison of soil core and soil pit sampling methods: means, standard deviations, and results of a Wilcoxon signed-rank test for all soil properties in surface mineral soil layers (0–20-cm depth). Paired data from 57 plots using both soil sampling methods were used for the Wilcoxon signed-rank test.

	Soil cores		Soil pits		Wilcoxon signed-rank test		
Property	Mean	SD	Mean	SD	W	z ratio	p
Bulk density, kg L^{-1}	0.57	0.25	0.93	0.29	1549	6.15	<0.01*
Coarse fragment content, %	21.4	17.5	39.5	18.0	1167	4.63	<0.01*
Soil mass, kg m^{-2}	92.2	46.4	112	45.7	793	3.15	<0.01*
C concentration, g kg^{-1}	74.2	60.3	45.2	22.9	−969	−3.85	<0.01*
N concentration, g kg^{-1}	3.83	2.93	2.55	1.4	−937	−3.72	<0.01*
C stock, Mg C ha^{-1}	40.8	17.1	44.7	16.1	393	1.56	0.12
N stock, Mg N ha^{-1}	2.3	1.02	2.6	1.16	369	1.46	0.14
C/N ratio	19.1	4.0	18.3	4.5	−501	−1.99	0.046†

* Site effect significant at $p < 0.05$ according to ANOVA.

† Site effect significant at $p < 0.1$ according to ANOVA.

other in this case, resulting in no significant differences in C and N stocks between sampling methods.

Contributions to Variation in Soil Carbon Stock

The sources of variance in soil C stock propagated from different variables were plotted for each research site and sampling method (Fig. 4). For the soil core method, the main source of variance in C stock was the soil %C, representing 43% of the total C stock variance. The contribution of %C was smaller for the soil pit method, where the contribution of %C was 36% in all plots combined, although at the FC site, BD and %C contributed equally (both 21%). The CF accounted for only 3% of the variance for the soil core method and 12% of the variance for the soil pit method.

Part of the variance in %C, BD, and CF was counterbalanced by covariance among these variables. The covariance between %C and BD contributed a greater proportion of the total variance of soil C stock for the soil core method (26%) than the soil pit method (20%). The covariance between %C and (1 − CF) decreased the C stock variability by 12% using the soil core method and 15% using soil pit method. The covariance between BD and (1 − CF) was positive for the soil core method but negative for the soil pit method except at the NS site. These largely negative covariance contributions demonstrate that the individual parameters are not independent and may result in reducing potential discrepancies in soil C and N stocks between sampling methods.

DISCUSSION

Sampling Method Comparison: Soil Pit vs. Soil Core

The organic layer C and N stocks were similar between the two sampling methods at all three research sites. This may be attributable to the fact that the sampling areas for the two methods were comparable: 2193 cm^2 (3 samples × 731 cm^2 per samples) using a bicycle tire in the soil core method vs. 2500 cm^2 in the soil pit method. Comparable sampling areas were not the case in the mineral soil layers, where the soil core method sampled 58.9 cm^2 (3 cores × 19.6 cm^2 per core) vs. 2500 cm^2 in the pit method.

The standard FIA soil core method samples only the surface 0 to 20 cm of the mineral soil. Conversely, the pit method more easily samples deeper surface layers. In this study, the deeper mineral layer (20–40 cm) accounted for 23 ± 10% of the measured C stock and 28 ± 9% of the measured N stock. The contribution of the deeper soil horizon varied among the three research sites (29 ± 8% at NS, 19 ± 9% at DEWA, and 23 ± 12% at FC). The FIA soil core method was therefore unable to capture large and variable C and N pools in the soil sampling process (Harrison et al., 2011; Jandl et al., 2014). For the soil pit method, C stocks in the deeper mineral layer were not correlated with the surface mineral soil at each site. This indicated the difficulty of predicting deeper soil C stocks on the basis of surface soil measurement. Therefore, the ability of using FIA soil core data in regional soil C stock estimation is limited due to the inability to sample the deep soil horizon.

For the soil core method, the problems identified in Table 1 could combine and lead to a large uncertainty of the soil C and N stock measurement. However, in previous studies, only one or two of the possible problems of the core method were identified to explain the metrological divergence. For instance, Page-Dumroese et al. (1999) and Levine et al. (2012) separately reported significantly lower rock fragment content in small-diameter cores, which they attributed to the obstruction problem in rocky forest soils from Montana, California, Nevada, New York, and New Hampshire. Soil compaction varying with soil texture was considered to be responsible for underestimation of BD and CF in a very gravelly sandy loam soil but not in a loamy sand soil in the state of Washington (Harrison et al., 2003). In 18 forest plots in southern New England, the core method produced lower BD and CF than the pit method, such that C and N stocks for the two methods were nearly identical (Kulmatiski et al., 2003), but only the problem of location selection was considered to explain the difference between the methods.

In the current study, significantly smaller BD and CF and greater %C were measured using the soil core method compared with the soil pit method (Table 4; Fig. 3). This is consistent with the effect of soil compaction problems beneath the soil core.

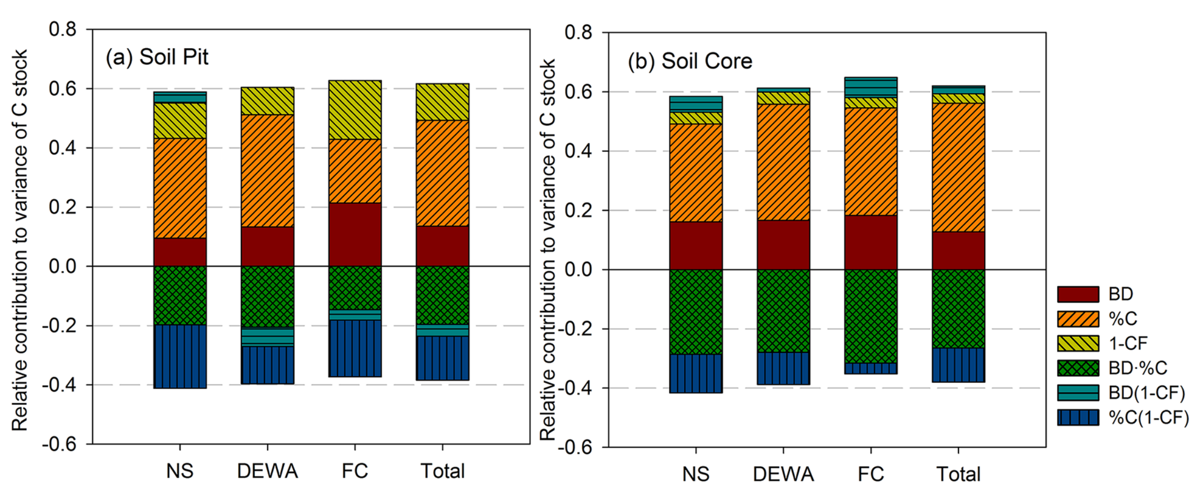

Fig. 4. Relative contributions of the variance and covariance of bulk density (BD), C concentration (%C), and soil volume (1 − coarse fragment content [CF]) to the variance of soil C stocks for the Neversink River Basin (NS), Delaware Water Gap Area (DEWA), and French Creek (FC) research sites using (a) the soil pit method and (b) the soil core method.

Assuming that the BD measured by the soil pit method is accurate and constant within each plot, it can be used to calculate how much soil should be in the core. In all the completed cores with no obstruction problem, the result shows that ∼32% of the soil was missed in the core method compared with the soil mass estimated by BD measured by the soil pit method. Compaction could therefore be the most important problem when using the composite coring technique in DRB forest soils. Obstruction and location selection also contribute to the difference between these sampling methods. In our case, only 71% of the soil cores reached the depth of 20 cm without being obstructed by rock.

In our study, %C, BD, and CF and their covariances had errors in different directions (see below), and as a result, the soil C stock, which is the product of these variables, showed no significant differences between the soil core and soil pit methods. Although the Wilcoxon signed-rank test indicated that the two methods generated the same C stocks (Table 4), results from the two methods were not well correlated due to high scatter (Fig. 3e), negating the possibility of creating a robust regression curve to correct the values of one method using the other because of the large error associated with the weak correlation. This outcome could be viewed as largely coincidental and not necessarily expected in all cases, but previous studies have observed relationships among the contributing variables, and thus an assessment of variability in soil C and N stocks requires a thorough examination of the sources of uncertainty, including potential covariances.

Source of Uncertainty in Soil Carbon Stocks in the Two Sampling Methods

The variation in soil C stocks at each research site and for all DRB plots combined was a combination of soil spatial variation and of biases and uncertainty associated with the sampling methods. If both methods were assumed equally accurate, the variance in C stocks would represent only the spatial variation in each variable in Eq. [2] because spatial variation would not be method dependent and Fig. 4a and 4b would have the same pattern. The observed patterns do indeed differ, and therefore methodological errors in each method are responsible for some of the variance in C stocks.

For the soil core method, variance in C stocks was mainly driven by the variance in %C (Fig. 4b), which was consistent with previously reported results from multiple sites in Europe (Schrumpf et al., 2011), two grassland sites in Germany (Don et al., 2007), and five cropland sites in Australia (Holmes et al., 2011). The relative contribution of %C to the total variance at our research sites was smaller than in the cited studies, which may have been caused by the complexity of the temperate forest in the DRB and the larger distances among our sampling plots. The importance of %C in determining the variance of C stocks has been shown to decrease with increasing spatial scale (Goidts et al., 2009).

The relative contribution of %C to the variance in C stocks was smaller for the soil pit method than the soil core method, while the contribution of BD and CF were larger. The DRB soils had a mean CF of 40%, larger than the stony soils assessed by Hoffmann et al. (2014) where CF introduced the largest spatial variance in a mountainous boreal forest. The CF therefore probably plays an important role in controlling the soil C stocks in the DRB. Unlike the pit method, the soil core method failed to properly detect the large spatial variation in CF representing conditions at the DRB study sites. The CF tended to be underestimated using the soil core method because of the obstruction and location selection problems mentioned above.

The assessment of soil C stock variance can be improved by incorporating the covariance between input variables (Panda et al., 2008) because these variables are not independent. The covariance between BD and %C counterbalanced a portion of the C stock variance. Several studies have previously demonstrated a strong negative relationship between BD and %C (e.g., Federer et al., 1993; Périé and Ouimet, 2008), which results from a combination of the "lightening" of the heavier mineral soil by lighter organic matter and from increased aggregation and porosity because of the binding ability of organic matter. In addition, the covariance between BD and %C was greater for the soil core method than the soil pit method. This was due, in part, to the compaction problem of the soil core method, which led to underestimating BD and overestimating %C at the same time, reflected in the negative covariance between BD and %C. The covariance between %C and soil volume (1 − CF) was also found to be negative and counterbalanced a portion of the C stock variance. A negative relationship between (1 − CF) and %C has been commonly observed (e.g., Johnson et al., 2012) and occurs because incoming organic matter is exposed to decreasing amounts of reactive soil mineral surfaces (i.e., silt and clay) and thus becomes more concentrated as the CF increases. The covariance between BD and (1 − CF) was small and positive for the soil core method, reflecting a greater CF associated with a smaller BD. The BD is typically greater in rocky soils (Mehler et al., 2014), but the obstruction problem is probably more severe in soils with greater rock content (Holmes et al., 2011), thus inducing a negative covariance between BD and CF for the core method but not the soil pit method.

Previous studies of the source of uncertainties in soil C stock were based on the soil core sampling method alone (Hoffmann et al., 2014). Our deconstruction of the sources of variance in both methods demonstrates that the importance of %C and its covariance with BD in determining the spatial variance of C stocks might have been overemphasized because of the limitations of the soil core method. By comparing the source of soil C stock variation in two sampling methods, we found that the soil pit method better estimated the spatial variance of BD, %C, and CF and their covariance and therefore the variance of soil C stock in the rocky soils of the DRB forest. The soil core method did not adequately estimate the magnitude of the variance and the direction of the covariance in these variables due to the physical limitations of the sampling method.

Soil Carbon and Nitrogen Stocks in Three Sites of the Delaware River Basin Forest

Previous studies (e.g., Harrison et al., 2003; Vadeboncoeur et al., 2013; and references cited therein) have concluded that the quantitative soil pit method has fewer sources of systematic errors in measuring soil C and N stocks relative to the soil core method and as such should be the reference or standard method against which others are compared. Our results are consistent with this conclusion, which is especially important for the stony soils in the DRB. The pit method also provides more complete C and N stock estimates by including a deeper mineral soil horizon compared with the FIA sampling method. Therefore, the following discussion is based on the soil pit sampling method only.

Soil C and N stocks within the DRB were greatest at the NS site in the north and smallest at the FC site in the south, consistent with the climate gradient shown in Table 2. A warmer and drier climate tends to favor organic matter decomposition processes more than the C input from productivity, resulting in less accumulation of soil organic matter in the southern area (Guo et al., 2006; Fissore et al., 2008). Other factors such as soil texture, topography, land use history, and forest type might also play an important role in determining the soil C and N stocks (Garten and Ashwood, 2002).

The soil C and N stocks measured at our three research sites were much smaller than the forested riparian zones in the DRB (100.3 Mg C ha^{-1} and 5.6 Mg N ha^{-1} in the 0–30-cm mineral soil) (Bedison et al., 2013). On the other hand, the upland forest plots in the Catskills region had smaller C and N stocks (19.0 Mg C ha^{-1} and 1.96 Mg N ha^{-1} in the 0–20-cm mineral soil) (Johnson, 2013) than our NS plots. Drainage conditions can change soil C and N stocks dramatically, where soil organic matter stocks were up to a factor of 10 smaller in the upland than in the riparian zones in small watersheds in the NS (Ashby et al., 1998). Although our plots were randomly located at each research site, they were representative of the complex landscape, and our results adequately represent a regional mean of high C in the valleys and low C on the slopes.

The soil C and N stocks measured in our NS plots were comparable with other studies in the northern hardwood forest (Huntington et al., 1988; Finzi et al., 1998; Bedison and Johnson, 2009; Johnson et al., 2009), but the DEWA and FC sites had much smaller C and N stocks. The overall average soil C and N stocks in all our plots were therefore smaller than most studies in the northern hardwood forest. This large-scale pattern was consistent with the C and N stocks within the DRB, which further emphasizes the climate control on soil C and N. The DRB lies in the transition area between northern hardwood, which is dominated by the maple–beech–birch species group, and the mixed deciduous forest, which is dominated by oak–hickory (Table 2). Forest species composition and any temporal change in composition may be other factors affecting the accumulation of C and N in the soil (Laganiere et al., 2013).

Implication for Regional Estimates of Soil Carbon and Nitrogen Stocks

While other methods have previously been used (e.g., rotary core), the composite core is the most extensively used method and the quantitative pit method is the most labor intensive approach used to estimate soil C and N stocks. The selection between the two methods depends on the aim of the study and the trade-offs between accuracy and efficiency. Other studies have suggested that less effort was required using the soil core method to detect the same magnitude of change with time in soil C and N stocks (Kulmatiski et al., 2003; Gruneberg et al., 2010). However, to estimate the soil C and N stocks at the regional scale (such as the DRB in this study), the accuracy of the sampling method is of crucial importance to capture the heterogeneity of the forest soil.

Soil survey data, such as the FIA soil data, are normally used as a data source to assess C and N stocks at the regional scale, to map the spatial distribution of soil properties, and to improve soil C and N simulation models (Meersmans et al., 2008; Ungaro et al., 2010). However, the accuracy of soil data collected by the core method used in FIA and their ability to capture the spatial variation of soil C and N stocks are questionable compared with the quantitative soil pit method. The various errors associated with the various problems of the sampling method, some of which may cancel each other out while others might be additive depending on the situation, might be accumulated in spatial extrapolation and introduce larger uncertainty to the regional estimates of soil C and N stocks.

ACKNOWLEDGMENTS

This study was supported by US Forest Service Grant no. 14-JV-11242306-083. We thank Lukas Jenkins, Adam Cesaneka, Jingyu Ji, Vanessa Eni, Ashley Crespo, Jason Cole, Alexa Dugan, and Matthew Patterson for assistance in field sampling and laboratory analyses of soil samples. We also acknowledge the private landowners who permitted access to their properties for soil sampling.

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