## Forest, Range & Wildland Soils: Uncertainty in Forest Ecosystem Studies

# Estimating Carbon and Nitrogen Pools in a Forest Soil: Influence of Soil Bulk Density Methods and Rock Content

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#### **Core Ideas**

- Three bulk density methods used in a rocky soil gave different total bulk density values, but not fine bulk density.
- Bulk density sampling method had little effect on C and N pool estimates in the surface 20 cm of soil, but did so deeper in the soil.
- Small-diameter cores probably underestimated total soil bulk density and C and N pools at deeper soil depths.
- Rocks present on the soil surface cause an overestimation of soil C and N pools.

Soils with high rock content are common in many US forests, and contain large amounts of stored C. Accurate measurements of soil bulk density and rock content are critical for calculating and assessing changes in both C and nutrient pool size, but bulk density sampling methods have limitations and sources of variability. Therefore, we evaluated the use of small-diameter soil cores (SD), irregular soil volume excavation (IR), and a nuclear density gauge (ND) to measure bulk density and rock content, and estimate C and N pools in three 10-cm increments to a 30-cm depth in a glacial till soil in northern Wisconsin. Total and fine bulk densities were lower when measured with SD cores than with larger soil volume IR and ND methods. No differences in C pools among bulk density sampling methods were found in the 10-cm increments, but when combined to 30 cm, the C pool estimate with IR (81.6 Mg ha<sup>-1</sup>) was significantly higher than those of ND (75.3 Mg ha<sup>-1</sup>) and SD (73.4 Mg ha<sup>-1</sup>). No significant differences in N pools were detected in the 10-cm increments, but the 0- to 30-cm N pool estimates by IR (5.65 Mg ha<sup>-1</sup>) and ND (5 Mg ha<sup>-1</sup>) were higher than that of SD (4.22 Mg ha<sup>-1</sup>). Surface rocks could lower soil C and N pools by 20% or more. Overall, the bulk density method had little effect on soil C and N pool estimates in the surface 20 cm of this soil but did when sampled to 30 cm soil depth.

Abbreviations: IR, irregular soil volume excavation; ND, nuclear density gauge; OM, organic matter; SD, small-diameter soil cores.

A aintaining the long-term soil productivity of forest sites is a mandate for National Forests in the United States, and is also a requirement for both industrial and nonindustrial private forests working toward sustainable forest management (Reeves et al., 2012). Many land managers and climate change modelers use estimates of soil nutrient or C pools to indicate site productivity changes, alteration of biological activity, impacts from prescribed burns or wildfire, or C sequestration potential (Powers et al., 1998; Kulmatiski et al., 2003). Since soil is a major component of local, regional, and global C and nutrient cycles (Jobbágy and Jackson, 2000; Zinke and Stangenberger, 2000; Bernoux et al., 2002), reliable estimates of C and nutrient pools at given soil depths are needed (Homann et al., 1995; 2004). However, skeletal soils ( $\geq$ 35% rock content by volume) are estimated to comprise 33% of the US land area, and soils with such high rock contents are difficult to sample, especially for bulk density (Throop et al., 2012). Bulk density, C, and N concentrations are needed for calculating pool sizes and considerable variability in pools size can be introduced (Harrison et al., 2003).

Numerous methods have been used to determine soil bulk density, such as clods (Blake and Hartge, 1986), soil cores (Cunningham and Matelksi, 1968; Blake and Hartge, 1986; Rogers and Carter, 1987), excavation (Muller and Hamilton, 1992), sand cones (McLintock, 1959), penetration resistance (Herrick and Jones, 2002), and NDs (Blake and Hartge, 1986). Identifying the appropriate bulk den-

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sity sampling method for a particular soil is an important factor in estimating changes in nutrient pool size after forest management operations (Homann et al., 1995; Page-Dumroese et al., 1999). Most commonly, bulk density is taken by weighing soil of known volume; however, a high rock content (soil fraction > 2 mm size), wind-thrown trees, large roots, and mixing from soil disturbances make it difficult to obtain representative samples in many forest soils (Vincent and Chadwick, 1994; Eriksson and Holmgren, 1996; Homann et al., 2001; Kulmatiski et al., 2003).

Soil cores of various diameters are often used to determine bulk density but they exclude rocks that are larger than the core cylinder, thereby underestimating rock content and possibly skewing C and nutrient pool calculations (Page-Dumroese et al., 1999; Kulmatiski et al., 2003). The use of large-diameter core samplers, irregular soil volume methods, or soil pit excavation are likely to give better estimates of soil heterogeneity and therefore of soil bulk density (Howard and Singer, 1981; Page-Dumroese et al., 1999). A rapid and nondestructive estimate of soil bulk density can also be obtained with a ND, which may have better precision than other methods in rocky soils (Fleming et al., 1993; Tominaga et al., 2002; Timm et al., 2005; Holmes et al., 2011). However, operator certification is required to use this instrument, and its weight restricts its use to soils with easy access (Page-Dumroese et al., 1999; Timm et al., 2005). If ND bulk densities are used for soil nutrient pool calculations, they also must be corrected for rock content, which requires some destructive soil sampling and introduces an additional error factor (Fleming et al., 1993).

In addition to rock content, the type and size classes of rocks (gravel, cobbles, etc.) are also important in determining the suitability of soil bulk density sampling methods and to convert soil C and nutrient concentrations to total pool contents (Fleming et al., 1993; Holmes et al., 2011; Throop et al., 2012). In general, bulk density sampling requires one to take soil volumes that are large relative to the scale of rock content and variability within the soil profile (Vincent and Chadwick, 1994; Wilding et al., 2001; Kulmatiski et al., 2003). Additional information is also needed on whether rocks contain organic matter (OM) or if rocks on the soil surface affect the soil sampling area (Poesen and Lavee, 1994; Harrison et al., 2003; Whitney and Zabowski, 2004).

Bulk density has a relatively high degree of variability, especially in soils with a high rock content (Cunningham and Matelski, 1968), so the sampling method should be unbiased and not substantially add to that variability. Understanding the limitations and sources of variability implicit in different bulk density sampling methods is also critical for evaluating changes in soil properties and nutrient contents resulting from forest management operations (Goidts et al., 2009). An additional concern is the amount of labor and expense needed to collect and process the bulk density samples and C or nutrient concentrations.

Therefore, we conducted a study to determine the impact of different sampling methods on estimates of soil bulk density and C and N pools in a rocky forest soil. This entailed: (i) using three methods to measure soil bulk density (IR, ND, and SD), (ii) assessing the effect of the bulk density sampling method on soil C and N pools, and (iii) evaluating the relative importance of the factors used in soil C and N pool calculations (bulk density, rock content, and C and N concentration).

# MATERIALS AND METHODS Site Description and Treatments

The site is located on the USDA Forest Service Argonne Experimental Forest in north-central Wisconsin (Fig. 1; 45°45′00″N, 89°03′00″W). The stand is predominately sugar maple (*Acer saccharum* Marsh.) with white ash (*Fraxinus americana* L.), basswood (*Tilia americana* L.), yellow birch (*Betula alleghaniensis* Britt.), and eastern hemlock (*Tsuga canadensis* (L.) Carr. The soil is an Argonne sandy loam (a coarse-loamy, mixed, superactive, frigid, Alfic Oxyaquic Fragiorthod), which was formed on a glacial till plain with 0 to 15% slope, and has a moderately deep (50–100 cm) fragipan. Approximately 60% of the soil surface was cov-

ered with rocks and boulders (Soil Survey Staff, 2006).

The study was conducted in three replicated 1-ha plots that were previously established in stands with two different thinning treatments: light retention (13.8 m<sup>2</sup> ha<sup>-1</sup> remaining basal area) and heavy retention (17.2 m<sup>2</sup> ha<sup>-1</sup> remaining basal area). Thinning was conducted using the single-tree selection method. Previous disturbance history shows the stands had been cut six times since 1951: first by horses (1951), then by mechanical logging equipment (tractors, tracked Iron Mule, rubber-tired forwarders) in subsequent harvests (Strong, 1997). Both thinning



Fig. 1. Location of the Argonne Experimental Forest in northern Wisconsin, United States.

treatments were bole-only harvested during the winter when snow depths were 40 to 100 cm, and all logging slash was left on site. There are no records of harvesting occurring on frozen soil during these thinnings. Permanent log landings created in 1951 were used during successive harvests to minimize the trafficked area. Three control 1-ha plots were also established in adjacent uncut stands with no history of timber removal since a selective cut in 1905, for a total of nine study plots (3 thinning treatments  $\times$  3 replicates). These plots were previously used in a soil compaction study in which bulk density samples were taken with SD [6.2 cm; Tarpey et al., (2008)].

## Soil Sampling, Analyses, and Calculations Nuclear Density Gauge and Soil Excavation

Five random sampling points were selected in each of the two thinning treatments and the uncut control plots (5 sample points  $\times$  3 treatments  $\times$  3 replicates; n = 45). If a surface rock > 20 cm in diameter was on a sampling point, another point was the selected within 1 m. Since differences in soil bulk density can come from both the method used and soil spatial variability, the same points were used for the IR sampling and ND measurements.

**Nuclear Density Gauge.** A Troxler Electronic 3440 series surface moisture-density gauge (Troxler Electronic Laboratories, Research Triangle Park, NC) in the direct transmission mode was used. The forest floor was removed at each sample point, and the probe containing a <sup>137</sup>Cs source was lowered to 10, 20, and 30 cm soil depths. A 1-min measurement was taken at each depth to get soil density values between the <sup>137</sup>Cs source and the soil surface (Blake and Hartge, 1986). These readings were corrected for soil moisture content at each sampling point.

Soil Excavation. After the ND measurements were completed, an irregular hole was sequentially dug to 0- to 10-, 10- to 20-, and 20- 30-cm soil depths using the ND probe location as the center point. All soil was removed from each depth, weighed, and placed in separate plastic zip bags for subsequent laboratory analyses. In approximately 20% of IR holes, all the large cobbles and stones could not be removed, so we measured the remaining rocks to estimate their volume. To get an accurate estimate of the soil volume removed, the hole was filled with expanding polyurethane foam, a weighted cardboard plate was placed on the surface (Muller and Hamilton, 1992), and the foam was left to cure overnight. This technique worked well in this rocky soil, as expansion of the foam filled all the irregularities in the unevenly formed soil holes. After the foam had cured, the cast was extracted from the hole, packed carefully to avoid volume changes, and sent to the USDA Forest Service Laboratory in Moscow, ID, for processing.

### The SD Soil Core Method

In an earlier study, Tarpey et al. (2008) used a small (6.2 cm diameter by 10 cm deep) impact soil core sampler to determine the thickness, bulk density, and C and N concentrations of the surface A horizon and underlying B horizon in these thinned and uncut plots. Cores were taken vertically to a 30-cm soil depth at 20 grid points spaced 20 m apart on each treatment plot (3 treat-

## Laboratory Analyses and Calculations

All SD and IR samples were weighed, dried to a constant weight at 105°C, and sieved through a 2-mm sieve to separate the fine soil and coarse soil fraction, then weighing each fraction separately. Soil particles, small rocks, and roots were removed from the outside of foam casts and cast volume determined by water displacement (Muller and Hamilton, 1992). Total bulk density ( $\rho_{bT}$ ) was calculated by dividing the oven-dried soil mass by the volume of the soil core or foam cast volume. Fine fraction bulk density ( $\rho_{bT}$ ) was calculated by the formula:

$$\rho_{bs} = \frac{\rho_{bT} \left( 1 - g_r \right)}{1 - \nu_r}, \qquad [1]$$

where  $g_r$  is the gravimetric rock content, which was obtained by dividing the mass of rocks by the total sample mass (Andraski, 1991). Volumetric rock content ( $v_r$ ) was calculated as:

$$v_{\rm r} = \rho_{\rm bT} \left( \frac{g_{\rm r}}{\rho_{\rm br}} \right), \qquad [2]$$

where the average rock density  $(\rho_{br})$  for glacial till was assumed to be 2.78 Mg m<sup>-3</sup> (Benson and Trast, 1995). The soil water and rock content values of the IR samples were used in the ND fine fraction bulk density calculations.

A subsample of the <2 mm mineral soil was taken from each 10-cm soil depth (IR method) and A and B horizon (SD method), finely ground in a ball mill, and analyzed using a Leco TruSpec analyzer (Leco Corp, St. Joseph, MI) for C and N concentration. Estimates of soil C and N contents were calculated using both fine fraction and total bulk density values (Cromack et al., 1999). We did not analyze the coarse fragment component (>2 mm), which has been found to contain appreciable amounts of C and N in some western US forest soils (Harrison et al., 2003; Whitney and Zabowski, 2004; Harper and Tibbett, 2013).

### **Statistical Analyses**

A completely randomized design was used to identify differences among means when all three methods for measuring bulk density were combined. Factors used in these analyses were: soil depth (0–10 cm, 10–20 cm, and 20–30 cm), sampling method (ND, IR, and SD), and thinning level (heavy, light, and none). The response variables examined were: total bulk density, fine fraction bulk density, C and N concentrations, C and N pools, the C/N soil ratio, and rock content. All factors and interactions were included in the initial ANOVA models. When significant main effect and interaction terms were identified, post-hoc test-

# Table 1. Bulk density (total and fine fraction) and rock content at three soil depths measured with three sampling methods at the Argonne Experimental Forest.

|                       | Soil depth         |              |                    |              |                    |              |  |  |  |  |
|-----------------------|--------------------|--------------|--------------------|--------------|--------------------|--------------|--|--|--|--|
| Sampling method       | 0–10               | cm           | 10–2               | :0 cm        | 20–30 cm           |              |  |  |  |  |
|                       | Bulk density       | Rock content | Bulk density       | Rock content | Bulk density       | Rock content |  |  |  |  |
|                       | Mg m <sup>-3</sup> | %            | Mg m <sup>-3</sup> | %            | Mg m <sup>-3</sup> | %            |  |  |  |  |
| Total bulk density    |                    |              |                    |              |                    |              |  |  |  |  |
| Excavation            | 1.02 (0.04) aA†‡§  | 19.3 (3.0)¶  | 1.21 (0.05) b      | 23.5 (3.3)   | 1.58 (0.05) c R    | 23.4 (3.9)   |  |  |  |  |
| Nuclear density gauge | 1.05 (0.03) m A    | _            | 1.33 (0.03) n      | _            | 1.37 (0.03) n S    | _            |  |  |  |  |
| Small-diameter core#  | 0.87 (0.01) r B    | 5.5 (0.4)¶   | 1.22 (0.01) s      | 8.3 (0.5)    | 1.24 (0.01) s ST   | 8.7 (0.6)    |  |  |  |  |
| Fine bulk density     |                    |              |                    |              |                    |              |  |  |  |  |
| Excavation            | 0.87 (0.04) a      | _            | 0.99 (0.04) a      | _            | 1.31 (0.05) b X    | _            |  |  |  |  |
| Nuclear density gauge | 0.91 (0.04) m      | _            | 1.12 (0.04) n      | _            | 1.18 (0.05) n XY   | _            |  |  |  |  |
| Small-diameter core   | 0.78 (0.02) r      | -            | 1.04 (0.02) s      | _            | 1.05 (0.02) s Y    | -            |  |  |  |  |

+ Different small letters within methods indicate significant differences across soil depths at p = 0.05.

 $\ddagger$  Different capital letters in the same soil depth indicate significant differences among sampling methods at p = 0.05.

§ Values in parentheses are the SE of the mean.

¶ This indicates that the rock contents at the 0- to 10-cm soil depth are significantly lower than the other two soil depths within the same sampling method.

# Rock content in small-diameter cores were significantly lower than rock content in the excavation and foam method at all three soil depths (p = 0.05).

ing to separate the means was conducted via Tukey's Studentized Range Test; critical values were adjusted with the LSMean option of SAS (SAS Institute, Cary, NC) when samples sizes were not equal in all treatment combinations. All analyses were accessed at an  $\alpha = 0.05$  significance level and were conducted with SAS version 9.4.

## **RESULTS** Soil Bulk Density

Similar to Tarpey et al. (2008), we found no effect of previous stand thinning on soil bulk densities, so thinning treatments were combined in our subsequent analyses (p = 0.23). As expected, total bulk density increased with soil depth, especially at 20 to 30 cm, and was generally lower when measured with the SD method than with the IR and ND methods (Table 1). This is reflected in the low rock content of the SD core samples (F = 158.4, p = < 0.001), which ranged from 1 to 20%, as compared with rock contents of 15 to 80% from IR holes. Total bulk density measured with SD cores showed a strong negative correlation with the fine soil fraction C and N concentration at all three depths (Table 2). In contrast, C and N concentrations in soil collected via the IR method were positively correlated with total bulk density, except at the 0- to 10-cm depth, but increased at lower soil depths.

The increase in total bulk density from the 10- to 20-cm to the 20- to 30-cm soil depth was much greater when it was measured with the IR method than with the ND (Table 1). Rock contents increased with soil depth, which made it difficult to remove similar volumes for each 10-cm soil increment in the IR sample collection (0-10 cm, 1.9 L; 10-20 cm, 1.4 L; 20-30 cm, 1.1 L). In contrast, the soil volumes measured by the ND were not known, as the distance the neutrons traveled from the radiation source into the surrounding soil was affected by soil moisture, rock content, and heterogeneity at each soil increment (Ølgaard and Haahr, 1968; Greacen and Schrale, 1976; Fleming et al., 1993). In addition, a smaller volume of soil was measured by the ND than the IR at each depth because of the triangular shape of the return path from the radiation source to the collector. Therefore, the different IR and ND soil bulk densities at 20- to 30-cm probably resulted from measuring different soil volumes.

Fine bulk densities (soil fraction <2 mm) also increased with

increasing soil depth for all sampling methods but were only significantly different between the SD and IR methods at 20 to 30 cm (Table 1). Rock content had little correlation with fine bulk densities from SD sampling, but was negatively correlated with fine bulk densities measured with the IR and ND methods (SD, r= 0.03, p = 0.72; IR, r = -0.54, p = 0.0001; ND, r = 0.53, p = 0.0003). Fine bulk densities were also negatively correlated with fine fraction C concentrations, especially in SD cores (Table 2).

### **Carbon and Nitrogen**

As expected, C concentrations were significantly higher in the 0- to 10-cm soil depth than at 10- to 20-cm and 20- to 30-cm (Table 3; F =

Table 2. Correlation of the core and excavation methods of the percentage of C and N concentrations with selected factors at three depths.

|                    | % C        |                 |            |                 | % N        |                 |            |                 |
|--------------------|------------|-----------------|------------|-----------------|------------|-----------------|------------|-----------------|
|                    | Excavation |                 | Small core |                 | Excavation |                 | Small core |                 |
|                    | r          | <i>p</i> -value |
| 0–10 cm            |            |                 |            |                 |            |                 |            |                 |
| Total bulk density | -0.12      | 0.46            | -0.81      | < 0.0001        | -0.09      | 0.56            | -0.75      | < 0.0001        |
| Fine bulk density  | -0.31      | 0.04            | -0.51      | < 0.0001        | 0.13       | 0.41            | -0.47      | < 0.0001        |
| Rock content       | 0.33       | 0.03            | -0.21      | 0.009           | 0.52       | 0.0003          | -0.19      | 0.02            |
| 10–20 cm           |            |                 |            |                 |            |                 |            |                 |
| Total bulk density | 0.59       | < 0.0001        | -0.50      | < 0.0001        | 0.60       | < 0.0001        | -0.52      | 0 < 0.0001      |
| Fine bulk density  | 0.09       | 0.53            | -0.27      | 0.0007          | 0.16       | 0.42            | -0.29      | 0.0002          |
| Rock content       | 0.54       | 0.0002          | -0.13      | 0.09            | 0.54       | 0.0003          | -0.12      | 0.12            |
| 20–30 cm           |            |                 |            |                 |            |                 |            |                 |
| Total bulk density | 0.44       | 0.0027          | -0.42      | < 0.0001        | 0.45       | 0.0023          | -0.44      | < 0.0001        |
| Fine bulk density  | -0.38      | 0.01            | -0.39      | < 0.0001        | -0.35      | 0.02            | -0.47      | < 0.0001        |
| Rock content       | 0.64       | < 0.0001        | -0.13      | 0.09            | 0.64       | < 0.0001        | -0.08      | 0.33            |

#### Table 3. Soil C and N concentrations and C/N ratio using fine bulk density values from three sampling methods.

|                     | Soil Depth   |               |                |             |                |               |             |               |              |
|---------------------|--------------|---------------|----------------|-------------|----------------|---------------|-------------|---------------|--------------|
|                     | 0–10 cm      |               |                | 10–20 cm    |                |               | 20–30 cm    |               |              |
| Method              | C conc.      | N conc.       | C/N            | C conc.     | N conc.        | C/N           | C conc.     | N conc.       | C/N          |
|                     | %            |               |                | %           |                |               | %           |               |              |
| Excavation          | 4.81 (0.73)† | 0.336 (0.051) | 14.5‡ (0.03)a§ | 2.31 (0.25) | 0.162‡ (0.021) | 15.2‡ (0.6)ab | 1.68 (0.21) | 0.111 (0.018) | 16.8‡ (0.4)c |
| Small-diameter core | 5.26 (0.20)  | 0.349 (0.017) | 15.5 (0.2)x    | 1.96 (0.08) | 0.106 (0.006)  | 20.3 (0.5)y   | 1.81 (0.03) | 0.95 (0.002)  | 20.4 (0.5)y  |

+ Values in parentheses are SE of the mean.

‡These values are significantly different between methods at the same depth.

§ Different letters across depths indicate significant differences at p = 0.05.

112.8; p = 0.001), but there were no appreciable differences between the SD and IR sampling methods (p = 0.72) or at the three soil depths (method × depth, p = 0.22). Rock content and C concentration in soil collected via IR were positively correlated, which increased with soil depth (Table 2). In contrast, a very weak negative correlation of rock content with C concentration was found in SD soil cores. Similar results were found with N concentrations: soil depth (F = 96.6; p = 0.001), sampling method (p = 0.17), and method x depth interaction (p = 0.15). The positive correlation of N concentration with rock content was stronger than that of C, and also showed a pronounced soil depth effect.

As expected, C/N ratios increased with increasing soil depth but were significantly higher in SD soil cores than in soil collected from IR holes (Table 3). These differences in soil C/N ratios are also reflected in the correlation of C concentration to N concentration, which was significantly lower in the SD soil cores (r = 0.87, p = < .0001) than in soil from IR excavations (r = 0.99, p = < 0.0001) over the three soil depths.

Similar to C concentrations, soil C pools were significantly higher in the surface 0- to 10-cm than at the 10- to 20-cm and 20- to 30-cm soil depths (Table 4). Though not statistically significant, there were appreciable differences among sampling methods and the three 10-cm soil depths (method × depth F =2.26, p = 0.06). No differences in soil C pools among the three

methods were found when individual 10-cm increments were combined for a 0- to 20-cm soil depth. However, when soil depth was increased to 30 cm, the C pool estimate with the IR method was significantly higher than that with SD and ND, and reflected the large difference in soil bulk densities among the three methods at the 20- to 30-cm sampling depth.

Similar to C, soil N pools were also significantly higher in the surface 10 cm than in deeper soil depths (Table 4), and there was no significant sampling method × soil depth interaction (F =1.82, p = 0.12). However, when the 10-cm soil increments were combined, the N pool estimates of the IR and ND methods were significantly larger than the SD cores for both the 0-to 20- and 0- to 30-cm soil depths.

## DISCUSSION Bulk Density

Soil compaction is often difficult to determine in rocky soils because of rocks on the soil surface and variable rock content within the soil profile over short distances (Vincent and Chadwick, 1994). Accurate bulk density information is critical for estimating soil C or nutrient pools, and for predictions of possible soil changes resulting from climate change (Throop et al., 2012). Of the three methods used in our study, the SD soil cores gave the lowest total bulk density values, which resulted from only taking 0- to 30-cm soil cores where not stopped by rocks. In contrast, many of these rocks were sampled in the IR method or in soil volumes measured by the ND. Our results also reflect the sampling of more SD cores (20 per plot) relative to the number of samples taken with the IR and ND bulk density methods (5 per plot), as compared with other studies. Harrison et al. (2003) also found that SD soil cores (per plot) underestimated bulk density in a very gravelly sandy loam (68% rock content) but were similar to bulk densities from soil pits (one per plot) in a loamy sand with a lower rock content (36% rock content). In contrast, Page-Dumroese et al. (1999) reported that

Table 4. Carbon and N pools at three soil depths calculated using fine bulk density values from three sampling methods.

|  |               |              | Soil depth               |               |              |  |  |  |
|--|---------------|--------------|--------------------------|---------------|--------------|--|--|--|
| Sampling method  | 0–10 cm†      | 10–20 cm     | 20–30 cm                 | 0–20 cm       | 0–30 cm      |  |  |  |
|  |               |              | Carbon                   |               |              |  |  |  |
|  |               |              | —Mg ha <sup>-1</sup> ——  |               |              |  |  |  |
| Excavation   | 38.1 (4.3)‡a§ | 23.3 (3.1) b | 20.2 (2.2) b             | 61.4 (7.1) A¶ | 81.6 (9.0) A |  |  |  |
| Nuclear densitometer   | 35.4 (2.7) e  | 23.1 (1.7) f | 16.8 (1.1) f             | 58.5 (4.1) A  | 75.3 (5.1) B |  |  |  |
| Small-diameter core  | 32.5 (0.6) x  | 20.7 (0.4) y | 20.2 (0.3) y             | 53.2 (0.9) A  | 73.3 (1.1) B |  |  |  |
|  |               | Nitrogen     |                          |               |              |  |  |  |
|  |               |              | ——kg ha <sup>-1</sup> —— |               |              |  |  |  |
| Excavation   | 2677 (308) a  | 1666 (271) b | 1314 (19)b               | 4343 (554) X  | 5657 (728) X |  |  |  |
| Nuclear densitometer   | 2473 (200) e  | 1593 (150) f | 1063 (90) f              | 4030 (322) X  | 5135 (417) X |  |  |  |
| Small-diameter core  | 2083 (55) x   | 1085 (29) y  | 1048 (23) y              | 3168 (78) Y   | 4216 (94) Y  |  |  |  |
| + Soil C and N pools at 0 to 10 cm were significantly larger than C and N pools at 10 to 20 cm and |               |              |                          |               |              |  |  |  |

20 to 30 cm for all three sampling methods, p = 0.05.

**‡** Values in parentheses are the SE of the mean.

**§** Different lowercase letters indicate significant differences across soil depths within the same method, p = 0.05.

¶ Different capital letters indicate significant difference among sampling methods at the same soil depth, p = 0.05.

both total and fine bulk densities in a Montana soil (35% rock content) measured with SD soil cores (five per plot) were generally higher than those for IR (three per plot) and similar to those measured with the ND (three per plot) methods. Timm et al. (2005) found no difference between soil bulk densities measured with SD cores (three per plot) or a ND (eight per plot), but they only sampled to a 5-cm soil depth and no rock content was given.

Total soil bulk densities generally decrease as particulate (free) OM content increases (e.g., Federer et al., 1993; Périé and Ouimet 2008, ), which was shown by the negative correlation of total bulk density with C and N concentrations in soil sampled with SD cores. The low rock content SD soil has a relatively high fine soil fraction/rock volume ratio, so there would be less rock resistance to increased soil porosity (lower bulk density) with additional OM. In contrast, the high rock content soil sampled by the IR method was positively correlated with C and N concentrations (higher OM), the fine soil/rock volume ratio would be lower than in the SD core soil, and rock resistance to increased soil porosity from additional OM would be much greater. Consequently, soil with a high rock content could show little change in total bulk density with increased fine soil OM content.

Differences in total bulk density among the three soil sampling methods were mostly eliminated by removing rock content in fine bulk density calculations, which is similar to the results reported by Han et al. (2016). However, Stewart et al. (1970) and Alberto (1971) found that fine bulk densities decreased with increasing soil rock content, whereas in a review of rock impact on soil processes, Poesen and Lavee (1994) concluded that fine bulk density generally increases with increasing rock content but then decreases at higher rock content as OM in the fine soil fraction increases. These variable study results probably reflect differences in soil OM content, as fine bulk density in our soil was negatively correlated with C concentrations, especially in SD cores.

## Soil Carbon and Nitrogen Pools

Considering the lack of significant differences in fine bulk densities and C and N concentrations among our three methods, we were not surprised by the similarities in the C and N pool size at each 10-cm soil depth. Kulmatiski et al. (2003) found that SD soil cores gave similar soil C and N pool estimates as soil pits when sampling the surface 0 to 15 cm of glaciofluvial and glacial till soils in southern New England; similar to our study, they had difficulty taking soil core samples because of the high rock content. However, combining our 10 cm soil increments to the 30cm sampling depth indicated the C and N pools estimates were lower with SD cores than with the IR and ND methods. This raises the question of how deep to sample soils to measure the possible impacts of management activities or climate change on C and nutrient pools. Another question is the possible impact of incorporating rocky soil C and N pool results, which used different bulk density sampling methods, on the within-soil heterogeneity of databases (e.g., STATSGO) used in ecosystem modeling (Miller and White 1998).

Numerous studies have documented the possible contribution of C contained in rocks to soil C pools (Ugolini et al., 1996; Harrison et al., 2003;Whitney and Zabowski, 2004; Homann et al., 2004; Harper and Tibbett, 2013), but the amounts are dependent on the rock type and rock content (Corti et al., 2002). We did not assess whether the rocks in our soil contained any C and N. However, most of the rocks in our glacial till soil came from the granitic Canadian Shield and have very few cracks and pores to hold OM. We used the specific gravity of 2.78 g cm<sup>-3</sup> for our glacial till rocks (Benson and Trast, 1995), which is higher than the 2.65 g cm<sup>-3</sup> usually used for soil minerals. Rock specific gravity can also affect fine bulk density calculations and should be considered when comparing the C pools of different soils (Holmes et al., 2011).

Numerous rocks were present on the surface of our soil. These are typically avoided during sampling, as we did when selecting the IR sample points. These surface rocks could have a major impact on many C and nutrient pool estimates, depending on the underlying soil volume they occupy. We did not measure the area of rock coverage on our plots, but a value of 60% is given in the Argonne soil series description (Soil Survey Staff, 2006). With this 60% value, and if we assume that one-third of the surface rocks on our plots extended to at least a soil depth of 30 cm, the C and N pools in our soil were overestimated by 20%. Currently, it is estimated that 7% of the soils in the conterminous United States have surface rocks ranging from 7.5 to 600 cm in diameter (Soil Survey Staff 2017), but surface rock coverage data for many soils in the mountainous western United States are not available in the SSURGO database. Although rocks protruding into soil pits and irregular hole excavations are sometimes measured (Eriksson and Holmgren, 1996; Rytter, 2012), we could not find any study which addressed the possible impact of surface rocks on soil C pool estimates.

# **Sampling Implications**

There are numerous methods for determining soil bulk density, but selecting which one to use becomes more difficult when the rock content of a soil increases. Our results and the other studies discussed above have shown that measuring bulk density in a soil with a high rock content can give decidedly different values, so the selection of an appropriate method is an important consideration when assessing forest management effects on soil compaction or the recovery of soil properties from previous soil disturbances. Overall, the more soil volume removed in sampling soils with high rock content, such as with the soil pit and irregular hole methods (e.g. McLintock, 1959; Flint and Childs, 1984; Page-Dumroese et al., 1999), the better the likelihood of obtaining accurate bulk density values. However, soil excavation methods are labor-intensive and often limit the number of replicate samples that can be taken. Good results can also be obtained with soil core methods if the core diameters are larger than most rocks being sampled (Jurgensen et al., 1977, Page-Dumroese et al., 1999).

The ND gave consistent estimates of total soil bulk densities in our study and may give better precision at deeper soil depths (e.g. Fleming et al., 1993). DeLong et al. (2012) found that the ND was the most rapid and gave better reproducibility for high rock content mine soils than four other bulk density methods, including IR. This method also allows for numerous nondestructive measurements, and no samples are taken to the laboratory for processing. However, operator certification is required, and the weight of the instrument restricts its use to soils with easy access (Page-Dumroese et al., 1999; Timm et al., 2005).

When bulk density measurements were used to calculate C and N pools in the surface 0 to 20 cm of soil, the sampling method had little effect on pool size estimates. Similar surface soil results were reported by Kulmatiski et al. (2003). This changed when the 10-cm sampling depths were extended to 0 to 30 cm, and the ND seemed to give the best values, as SD cores probably underestimated pool sizes and the IR method had some problems obtaining adequate soil volumes at the 20- to 30-cm soil depth. However, ND bulk densities used for soil pool size calculations must be corrected for rock content, which requires destructive soil sampling (Fleming et al., 1993), and soil samples must also be taken for soil C and nutrient analyses.

## CONCLUSION

Our study clearly shows that the three methods used in our study gave different total bulk density values with increasing soil depth, but this difference mostly disappeared when fine bulk densities were calculated. Consequently, the method of bulk density sampling had little effect on C and N pool estimates in the surface 20 cm of soil, but when the soil was sampled to 30 cm, SD cores were likely to have underestimated the pool sizes. The high rock content at the 20- to 30-cm depth made it difficult for the IR method to obtain adequate soil volumes at that depth in some excavations, so the ND would be the best choice for this soil. However, if only the ND was used, we still would have to take samples at each soil depth to determine the rock content and soil C and N concentrations. Though this was not done in our study, the area of rock coverage on the soil surface should also be measured, as well as trying to get an estimate of the mineral soil volume these rocks occupy. Although it may be difficult to get such information, it could have a major impact on soil C and N pool size estimates.

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