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Revisiting Soil C and N Sampling: Quantitative Pits vs. Rotary Cores

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Abstract

Increasing atmospheric CO₂ and its feedbacks with global climate have sparked renewed interest in quantifying ecosystem C budgets including quantifying belowground pools. Belowground nutrient budgets require accurate estimates of soil mass, coarse fragment content, and nutrient concentrations. It has long been thought that the most accurate measurement of soil mass and coarse fragment mass has come from excavating quantitative soil pits. However, this methodology is labor intensive and time consuming. We propose that diamond tipped rotary cores are an acceptable if not superior alternative to quantitative soil pits for the measurement of soil mass, coarse fragment content, carbon (C) and total nitrogen (N) concentrations. We tested the rotary core methodology against traditional quantitative pits at research sites in CA, NV, and NY, USA. We found that soil cores had 16% higher estimates of < 2 mm soil mass than estimates obtained from quantitative pits. Conversely, soil cores had 8% lower estimates of coarse fragment mass compared to quantitative pits. There were no statistical differences in measured C or N concentrations between the two methods. At the individual site level, differences in estimates for the two methods were more pronounced, but there was no consistent tendency for cores to over or under estimate a soil parameter when compared to quantitative pits.

Running Title: Revisiting C and N sampling

Key words: Soil sampling, soil pit, soil core, coarse fragment, carbon, nitrogen

INTRODUCTION

Estimating soil mass and rock content is an essential part of determining nutrient contents in ecosystems (Harrison et al. 2003). This has become increasingly important with the current interest in global climate change and soil carbon (C) content. Soils typically contain the largest and most difficult pool of C to estimate (Homann et al. 2001). Several methods have been utilized for measuring soil mass and rock content including: punch cores, machine-driven core drills, truck mounted corers, impact hammer driven cores, and even explosives (Tuttle et al. 1984; Jurgensen et al. 1977; Hayden and Robbins 1975; Robertson et al. 1974; Schickedanz et al. 1973; McIntyre and Barrow 1972; Hayden and Heinemann 1968). However, none have proven to be as universally accepted or applicable as the large-excavation, quantitative soil pit (Johnson et al. 2005; Harrison et al. 2003; Hamburg et al. 1984). In 1997 researchers proposed a motor-driven core sampler for taking intact samples from rocky soils at the Long Term Forest Productivity (LTSP) plots in southern Missouri, USA (Ponder and Alley 1997; Powers et al. 1989). They determined that the core device was effective at retrieving undisturbed soil cores for estimation of bulk density, root biomass, and nutrient contents to a depth of 35 cm (Ponder and Alley 1997). We believe that this device, a motor-driven, diamond-tipped rotary corer, has the potential to supplement or replace the traditional excavated quantitative pit for estimating soil mass, rock content, and nutrient concentrations through the soil profile.

Quantitative soil pits are typically hand or machine excavated pits where all of the material is removed from the pit, separated by size fraction, and weighed. Excavating quantitative soil pits can be laborious, time consuming and destructive which precludes their use in small plots. The volume of the pit is estimated by measuring the dimensions of the pit or back calculating the volume of the pit from the mass and density of material removed. This enables researchers to

calculate nutrient budgets on a mass per area basis. Estimates of pit volume are still difficult in rocky soils because of large coarse fragments which may protrude into the pit wall. It is imperative that the rock content of the soil regolith is accurately estimated as well as the soil mass so that reliable estimates of nutrient content may be calculated. Additionally, quantitative pits require the use of sub-sampling, moisture corrections, and extensive back calculations to obtain estimates for root, rock, and soil mass and volume. These calculations are not necessarily complex, but introduce cumulative errors into the estimates (Figure 1).

By contrast, the diamond tipped rotary core device creates relatively little surface disturbance, can be used to sample many locations efficiently, and allows for more straightforward estimates of soil and rock mass on a volume and areal basis. Two or three people can operate the device in an area roughly 9 m². The core bit is large enough to obtain a quantitative sample, but with an internal core diameter of only 7.62 – 9.5 cm, minimizes soil excavation. We have been able to core to a depth of 1 m in times ranging from 20 – 45 minutes, and deeper sampling is possible. The rotary core device cuts through large coarse fragments eliminating bias introduced by including or excluding large coarse fragments that protrude only partway into quantitative pits (Figure 2). Calculations for estimating root, rock, and soil mass and volume are obtained directly from individual core samples (Figure 3). Additionally, the rotary core device is relatively portable weighing roughly 29 kg, can be transported on a pack frame over large distances and rough terrain, and can be assembled using pre-existing components and easily manufactured parts.

We hypothesized that the rotary driven core device would provide similar estimates of rock mass, soil mass and C and N concentrations as obtained from quantitative soil pits. In order to test the rotary core device as an alternative to quantitative soil pits we conducted paired

comparisons of pit and core soil samples collected in three ecosystems within the conterminous US. We hypothesized that the study sites were unique to each-other and provided three viable replicates for our study. Finally we proposed that if differences occurred between methodologies they would be consistent across sites. We directly compared estimates of soil mass, coarse fragment mass, soil organic C%, and soil total N%.

MATERIALS AND METHODS

Study Design and Data Collection

Three study sites were chosen where existing data from quantitative soil pits had been collected in order to quantify soil mass, coarse fragment content, and C and N concentrations. In addition to quantitative pit data we used the core device to collect similar data immediately adjacent to soil pits. Two of the sites are in the western US; one in the Great Basin southwest of Austin, NV, and the other located in the Sierra Nevada Mountains northeast of Truckee, CA. The third site is located in the eastern US within Tompkins County, NY.

Experimental Areas

Underdown Canyon (39°15'11" N 117°35'83" W) is a Joint Fire Sciences Program Demonstration Area in the Shoshone Mountain Range located in Nye County, NV on the Humboldt-Toiyabe National Forest. The canyon is oriented east to west and study plots are located at elevations from 2,209 m to 2,227 m. Average annual precipitation averages 25 cm and arrives mostly as winter snow and spring rains. Average annual temperature ranges from -7.2 °C in January to 29.4 °C in July. Lithology of the Shoshone range consists of welded and non-welded silica ash flow tuff. Soils are classified as Coarse loamy mixed frigid Typic Haploxerolls.

The soils are extremely coarse grained and have weak to moderate structure. Vegetation is characterized by sagebrush (*Artemisia tridentata* Nutt. ssp. *vaseyana* [Rydb.]) and single leaf pinyon (*Pinus monophylla* Torr. & Frém) with lesser cover of Utah juniper (*Juniperus osteosperma* Torr. Little), and associated grasses and forbs (Rau et al. 2005).

The Truckee site (39°15'9" N, 120°49'23" W) is a 12.1 ha second growth, naturally regenerated, pure Jeffrey pine (*Pinus jeffreyi* Grev. and Balf.) stand located in Nevada County, CA, on the Tahoe National Forest. The site has a generally northeast aspect with a slope varying from 3 to 12 % at an elevation of 1,767 m. The mean annual precipitation is 69 cm, falling predominantly as snow between October and May. The mean annual temperature at the study site is 6 °C, and ranges from –12 °C in January to 29.4 °C in July. Soils are fine-loamy, mixed, frigid, Ultic Haploxeralfs derived from andesite. Understory vegetation on the site consists of sagebrush, bitterbrush (*Purshia tridentata* DC.), mule's ear (*Wyethia mollis* A. Gray), greenleaf manzanita (*Arctostaphylos patula* Green), and prostrate ceanothus (*Ceanothus prostrates* Benth.) (Murphy et al. 2006).

The Tompkins County sites (42° 16-25' N, 76° 23-40' W) near Ithaca, NY, consist of eight sampling locations, two of which were never plowed while the remaining six were abandoned from agriculture 50-100 years prior to sample collection (Flinn et al. 2005). The sites had variable slope and aspect with a mean elevation of 292 m. Mean annual precipitation is 93 cm, with more precipitation on average in summer than winter. Mean annual temperature is 7.8 °C, with monthly mean temperatures ranging from -5.2 °C in January to 20.4 °C in July. Soils at these sites consist of Dystrudepts, Fragiaguepts, and Fragiudepts developed in till deposited by Wisconsinan glaciation over bedrock of Devonian shale (Neeley 1965). The dominant tree species include sugar maple (*Acer saccharum* Marsh.), red maple (*A. rubrum* L.), American

207 beech (*Fagus grandifolia* Ehrh.), and white ash (*Fraxinus americana* L.). Other species present
208 include red oak (*Quercus rubra* L.), eastern hemlock (*Tsuga canadensis* (L.) Carrière), white
209 pine (*Pinus strobes* L.), quaking aspen (*Populus tremuloides* Michx.), black birch (*Betula lenta*
210 L.), and black locust (*Robinia pseudoacacia* L.).

212 **Soil Pit Sampling**

213 In Underdown, NV 18 total soil pits were excavated. Individual pits measured 50 x 50 cm and
214 were excavated in four consecutive depth increments (0-8, 8-23, 23-38, and 38-52 cm) for a total
215 of 72 samples. In Truckee, CA 24 soil pits measuring 50 x 50 cm were excavated in three
216 consecutive depth increments (0-20, 20-40, and 40-60 cm) for a total of 72 samples. At the
217 Tompkins, NY sites ten 71 x 71 cm soil pits (3 pits at one site; one pit per site at the other seven
218 sites) were excavated in five consecutive depth increments (0-10, 10-20, 20-30, 30-40, and 40-50
219 cm) for a total of 50 samples.

220 Forest floor material was removed prior to mineral soil excavation. All material from each
221 depth increment was removed from pits and field-sieved to 10 mm. Roots were manually
222 separated from rocks > 10 mm. The soil and rock fractions were weighed in the field using a
223 spring scale. Sub-samples of less than 10 mm soil weighing approximately 2 - 10 kg each were
224 collected from each depth increment by hand or using a metal scoop. Sub-samples were returned
225 to the lab, weighed, and sieved to 2 mm. To calculate percent moisture, a sub-sample was dried
226 at 100° C for 24 hours or until the sample no longer lost mass (Figure 1).

227 For the Underdown and Truckee sites, bulk density of the < 10 mm fraction was calculated by
228 taking a 100 cm³ sample using an impact sampler at each depth increment prior to soil removal.
229 Total pit volume was calculated for each depth increment by adding the estimated > 10 mm rock

volume (> 10 mm rock mass / Db_{rock}), the < 10 mm soil volume (< 10 mm soil mass moisture corrected / Db_{soil}), and > 10 mm root volume (> 10 mm dry root mass / Db_{root}) (Johnson et al. 2005). For the Tompkins pits, volume was calculated using measured depths for 25 points on a 18 cm grid (Hamburg 1984). Total pit bulk density was then calculated by dividing the estimated rock and < 2 mm soil mass by the pit volume.

Soil Core Sampling

Soil cores were extracted at locations corresponding to each soil pit. Soil samples corresponding to the depth increments excavated in pits were removed from each bore hole for a total of 72 samples at Underdown, 72 samples at Truckee, and 50 pooled samples at Tompkins (4 cores were taken at each pit, one at each side).

The method utilizes a 7.62 cm (for Underdown, NV and Truckee, CA sites) and 9.5 cm (for Tompkins, NY) internal-diameter diamond-tipped core device manufactured by DiteqTM, and is driven by a two-person rotary Briggs and StrattonTM power head, allowing it to core through rocks and soil with minimal compaction (Ponder and Alley 1997). Each sample increment was extracted before the core was driven to the next depth increment. This methodology should help to further minimize compaction of each depth increment. Cores were bagged individually, brought back to the lab, dried at 100° C for 48 hours, and weighed. Cores were then sieved to 2 mm.

Sample Analyses

Soil samples < 2 mm were ground using an IKA impact headTM type mill for Underdown and Truckee, and a Retsch Mixer MillTM, type MM200 for Tompkins. Samples from Underdown

and Truckee were analyzed using a LECO Truspec[®] CN analyzer, and samples from Tompkins County were analyzed with an Elementar Vario EL[®] III elemental analyzer. Samples in our study did not contain significant inorganic C as determined by an HCl digest. Therefore, all measured C was attributed to be organic C (OC).

Statistical Analyses

We analyzed four key soil variables for differences between the three test sites and the two methods used to collect the data (soil pits vs. soil cores). Variables tested included: < 2 mm soil mass, > 2 mm coarse fragment mass (rock mass), soil C%, and soil total N%. All other variables of interest including regolith bulk density and C and N content can be calculated using these estimates. All comparisons were evaluated using SAS[™] generalized linear mixed effects models (Proc GLIMMIX). Site and sample type differences were evaluated using site as a main effect and sample type as a block within site. Soil depth and interactions terms could not be directly analyzed with the mixed model because the number of depth increments and the depth of individual increments were variable across sites. Mean comparisons were made with Tukey's test ($P < 0.05$) after confirming significant main effects and interactions with the mixed models ($P < 0.05$). Tukeys' tests were also used to evaluate differences between sample types at individual soil depth increments ($P < 0.05$).

RESULTS AND DISCUSSION

The three sites differed significantly for all four variables tested ($P < 0.05$). This analysis confirms that the three sites provide three statistically distinct locations to test our main hypothesis. When all three sites were grouped, core samples resulted in 16% higher estimates (P

= 0.0078) for < 2 mm soil mass when compared to soil pit samples (Figure 4). Conversely, core samples resulted in 8% lower estimates ($P = 0.0043$) of coarse fragment mass when compared to pit samples (Figure 4). Estimates of soil C% and N% were statistically similar between sampling methodologies (Figure 4).

The simple pooling of sample type estimates may lead the reader to believe that cores universally result in higher estimates of < 2 mm soil mass (Figure 4). However, this was not the case in our comparison. The Sample Type x Site term in the mixed model indicates that there were significant interactions for all of the variables tested (Table 1). Our comparisons of the three sites indicate that there was no consistent bias for a sampling method to over- or under-estimate soil variables (Figure 5). This is contrary to our original hypothesis. Soil cores only resulted in higher estimates of < 2 mm soil mass at the Tomkins, NY site, while estimates for soil mass were similar between methods at Truckee, CA and Underdown, NV (Figure 5). Coarse fragment estimates were similar between methods at Tomkins, NY and Underdown, NV, but higher when estimated with pits in Truckee, CA (Figure 5).

It is not entirely clear why each site displayed its own unique differences between sample type and regolith physical properties, but it could be due to the size and distribution of coarse fragments or the method by which pit volume was estimated. If the regolith contains very few, but rather large boulder size coarse fragments, the likelihood of encountering one with a large quantitative pit is greater than with a small diameter soil core. This is due to the relationship between cross sectional area and volume. A small increase in cross sectional area sampled can result in a large change in the volume sampled. This is likely the case in Truckee, CA where several very large boulders either inhibited the completion of a pit, or were removed from pits. However, when soil cores were taken in Truckee, CA, we encountered no obstructions to the 60

cm sample increment, and removed no complete rock samples from the rotary core. Conversely, if the soil profile has a more spatially uniform and heterogeneous size distribution of coarse fragments it is likely that the diamond tipped rotary core will proportionately sample those coarse fragments. Estimates of pit volume at the Truckee, CA and Underdown, NV sites were done by back-calculating the volume of the pit from rock mass, rock density, soil mass, and soil density. Pit volume estimates at the Tomkins, NY site were made by measuring the dimensions of the pit. This methodology is problematic due to the inability to dig vertically walled pits, and to account for large rocks protruding into the pit. Over estimating the volume of the pit would result in the lower estimate of soil mass using pit measurement methodology.

Soil C% and N% were similar when measured with pits and cores at the Tomkins, NY site, but were higher when measured with pits in Truckee, CA, and lower in when measured with pits in Underdown, NV (Figure 5). The result of the inconsistent patterns in soil nutrient concentrations between measurement types is unclear at this time, but clearly influences estimates of soil C and N pools. One potential explanation for the lack of difference between methods at Tomkins, NY could be that the core samples at this site are a composite of 4 cores taken around the perimeter of the soil pit. Due to the extreme heterogeneity of the soil medium it is possible that a single core does not integrate the mean soil nutrient concentration that would be obtained from a quantitative pit sample. A composite sample of several cores may give a better estimate of mean soil concentration in a small area around a pit. Another potential source of error in the measurements of soil C and N concentration could come from the grinding of rock fragments and the inclusion of these grindings into < 2 mm soil C and N concentrations. This might be especially true in soils derived from sedimentary deposits which contain high concentrations of C or N. (Halloway and Dahlgren 1988; Whitney and Zabowski 2004). We analyzed coarse

fragment chemistry as a follow up to our initial findings. We determined that coarse fragments could contribute to total regolith C and total N content, but there was no bias towards greater soil concentration of C and total N in cores relative to pits, that could be attributed to rock grinding. Concentrations of C and N in coarse fragments were at least an order of magnitude lower than soil C and N estimates and the cross sectional area of the core which would have been represented by rock grinding (≈ 1 cm) would be less than 12% of the total area and volume sampled. We estimated that coarse fragments account for 29 - 62% of the regolith mass using pit estimates, and 39 - 58% of regolith mass using core estimates. Across the three sites coarse fragments accounted for 2 - 15% of total regolith C content when measured with pits and 7 - 9% when measured with cores. Coarse fragments accounted for 5 - 30% of total regolith N content when measured with pits and 13 - 19% when measured with cores. The coarse fraction often is assumed inert and neglected; however, several researchers have documented the importance of including coarse fraction estimates in nutrient budgets (Fernandez et al. 1993; Ugolini 1996; Corti et al. 1998; Harrison et al. 2003). We hypothesize that soil embedded in coarse fragment pores or cracks is the dominant source of C and N associated with the coarse fraction in our study. Although grinding of the coarse fraction may not be a significant source of soil C and N in our study, future work is needed to test the effects of how rock grinding influences estimation of other nutrient pools including base cations. Rock material is the primary source of base cations in soils and therefore excessive grinding and powdering of rock material may lead to an overestimation of soil base cation content.

CONCLUSIONS

We believe the diamond tipped rotary core device tested in this comparison is a viable alternative to quantitative soil pits. Although the core estimates were not identical to pit estimates at all of our test sites, the overall difference between methods was not greater than 16%. More importantly it does not appear that the core device consistently over- or under-estimates any specific soil regolith property when compared to quantitative pits. This device has the potential to increase a researcher's sample size (n) because of its relatively low time requirements compared to pit sampling. This methodology will prove important in large landscape scale studies with significant heterogeneity or in repeated measures studies where large sample size (n) is required to detect a significant change. Furthermore we believe the core device provides unbiased estimates of coarse fragment and sample volume in most soils because large coarse fragments are cut clean and proportionately sampled. There are still unresolved differences among individual sites for several soil properties including soil mass, coarse fragment mass, and soil C and N concentrations. On certain soils it may be necessary to increase the sample size to adequately characterize large coarse fragments.

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365 **Table 1.** Results of the mixed model for differences between sites, sample types, and their interaction.

		Soil Mass		Rock Mass		Soil C%		Soil N%	
	<u>DF</u>	<u>F</u>	<u>P</u>	<u>F</u>	<u>P</u>	<u>F</u>	<u>P</u>	<u>F</u>	<u>P</u>
Site	2	105.16	<0.0001	17.2	<0.0001	15.5	<0.0001	7.53	0.0058
Error A = Replicate (Site)	59								
Sample Type	1	33.73	0.0078	1.1	0.0043	0.64	0.3048	0.05	0.8238
Sample Type x Site	2	16.71	<0.0001	9.67	<0.0001	23.76	<0.0001	37.3	<0.0001
Error B = Depth x Replicate (Site)	206								

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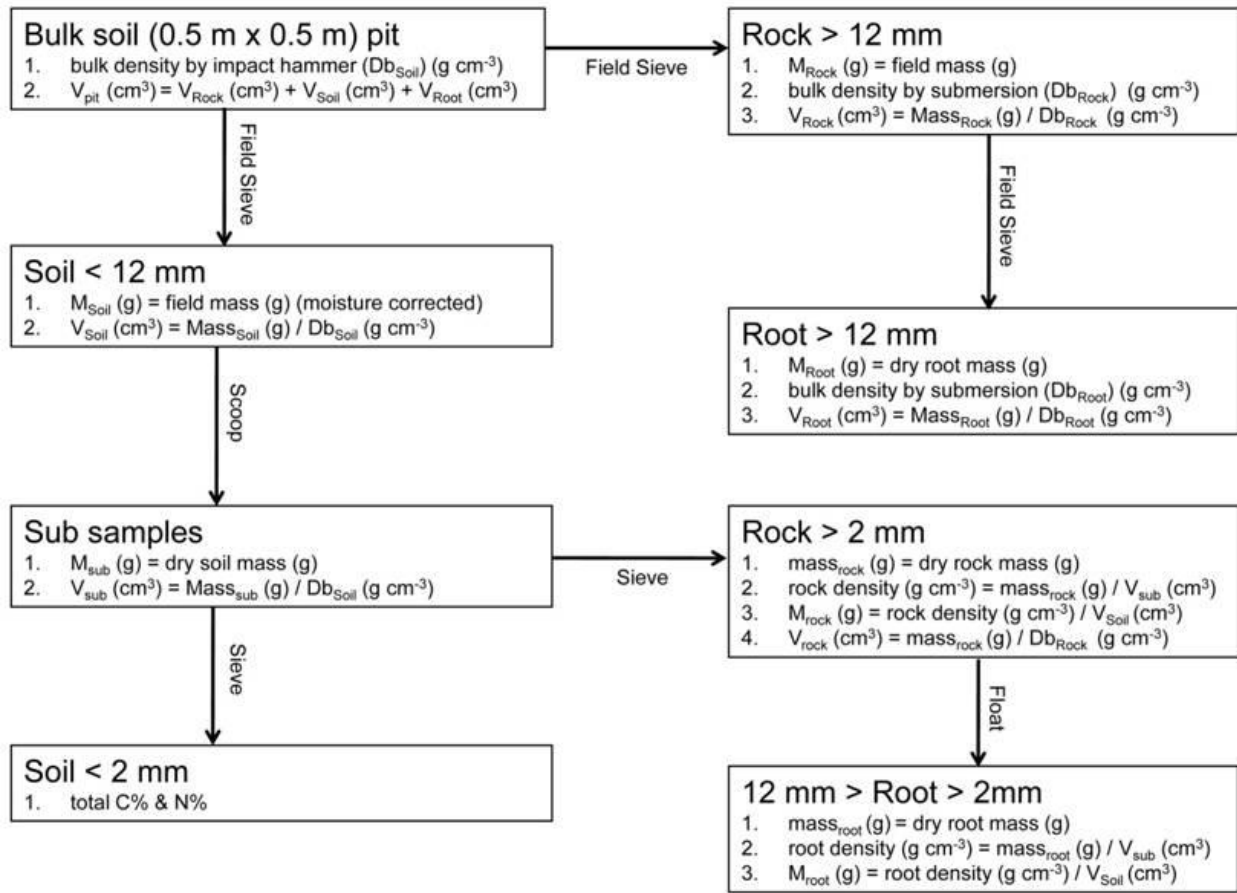
Figure 1. Sample processing regime, and unit conversion for each soil pit increment excavated.

Figure 2. Photos of the rotary core bit, the adapter shaft used to connect it to the power head, and the power head. Note how cleanly the large coarse fragment has been cut by the core device. Top scale is in inches, bottom scale is in cm. Models are J.J. Klima and the corresponding author at USFWS, Hart Mountain Wildlife Refuge, OR.

Figure 3. Sample processing regime, and unit conversion for each core increment extracted.

Figure 4. Means and standard errors for the two sampling methods. Double asterisks indicate statistically different means (Tukey's test $p < 0.05$).

Figure 5. Means and standard errors for the two sampling methods at each site and depth increment. Double asterisks indicate statistically different means (Tukey's test $p < 0.05$).



$$\text{Roots} > 2 \text{ mm (Kg ha}^{-1}\text{)} = \{M_{\text{Root}} (g) + M_{\text{root}} (g) / V_{\text{pit}} (\text{cm}^3)\} \cdot d (\text{cm}) \cdot 100,000,000 (\text{cm}^2) \cdot C$$

$$\text{Rocks} > 2 \text{ mm (Kg ha}^{-1}\text{)} = \{M_{\text{Rock}} (g) + M_{\text{rock}} (g) / V_{\text{pit}} (\text{cm}^3)\} \cdot d (\text{cm}) \cdot 100,000,000 (\text{cm}^2) \cdot C$$

$$\text{Soil} < 2 \text{ mm (Kg ha}^{-1}\text{)} = \{M_{\text{Soil}} (g) - M_{\text{rock}} (g) - M_{\text{root}} (g) / V_{\text{pit}} (\text{cm}^3)\} \cdot d (\text{cm}) \cdot 100,000,000 (\text{cm}^2) \cdot C$$

Where (C) = nutrient concentration in fraction % and (d) = depth of the pit increment

472 Figure 2.



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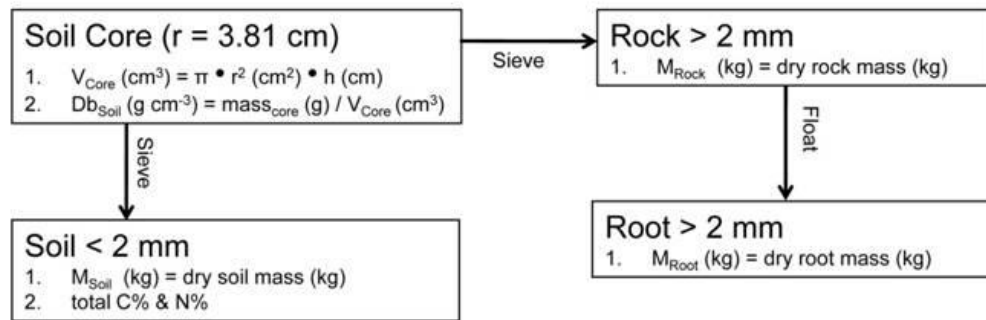
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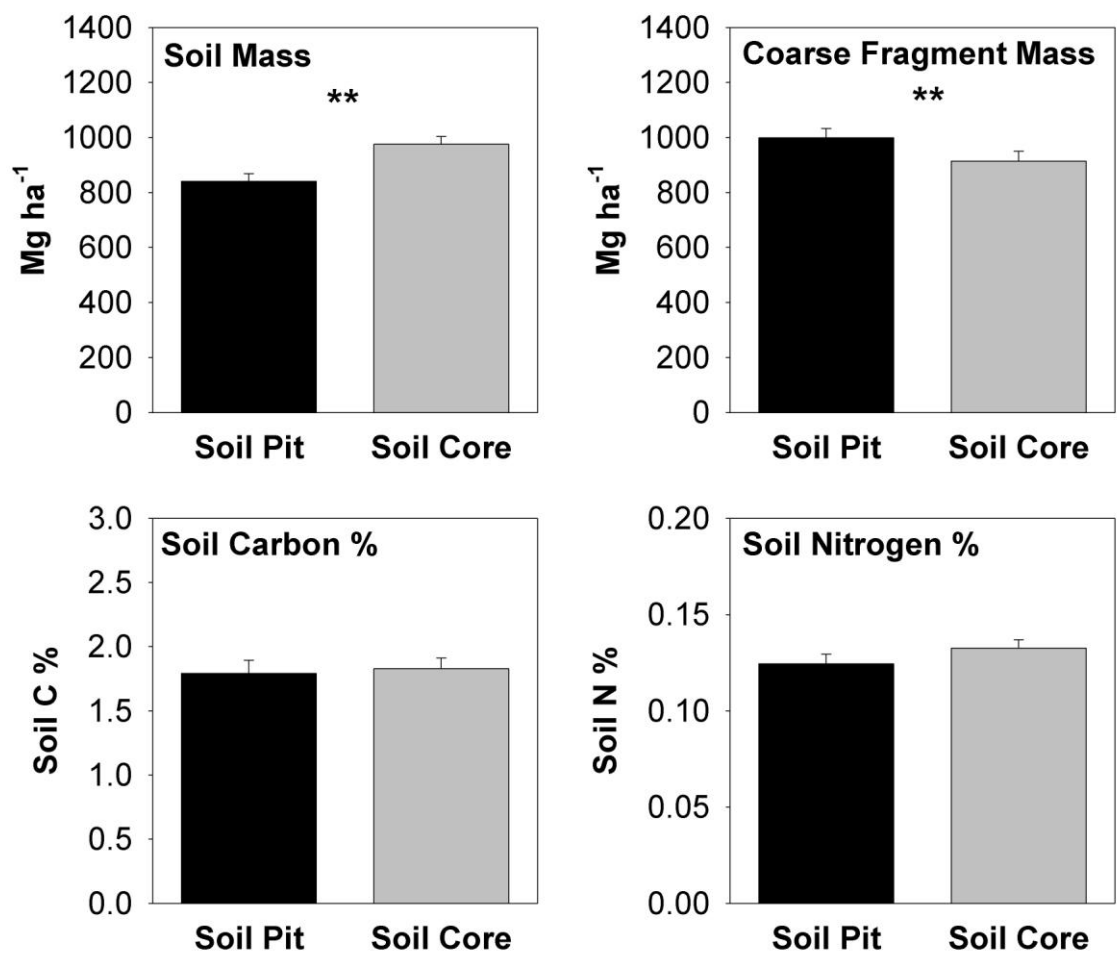
$$\text{Roots} > 2\text{ mm (Kg ha}^{-1}\text{)} = \{M_{Root} (kg) / V_{core} (cm^3)\} \cdot d (cm) \cdot 100,000,000 (cm^2) \cdot C$$

$$\text{Rocks} > 2\text{ mm (Kg ha}^{-1}\text{)} = \{M_{Rock} (kg) / V_{core} (cm^3)\} \cdot d (cm) \cdot 100,000,000 (cm^2) \cdot C$$

$$\text{Soil} < 2\text{ mm (Kg ha}^{-1}\text{)} = \{M_{Soil} (kg) / V_{core} (cm^3)\} \cdot d (cm) \cdot 100,000,000 (cm^2) \cdot C$$

Where (C) = nutrient concentration in fraction % and (d) = depth of the core increment

485 Figure 4.



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