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

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

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91 Abstract

92 Increasing atmospheric CO₂ and its feedbacks with global climate have sparked renewed interest 93 in quantifying ecosystem C budgets including quantifying belowground pools. Belowground 94 nutrient budgets require accurate estimates of soil mass, coarse fragment content, and nutrient 95 concentrations. It has long been thought that the most accurate measurement of soil mass and 96 coarse fragment mass has come from excavating quantitative soil pits. However, this 97 methodology is labor intensive and time consuming. We propose that diamond tipped rotary 98 cores are an acceptable if not superior alternative to quantitative soil pits for the measurement of 99 soil mass, coarse fragment content, carbon (C) and total nitrogen (N) concentrations. We tested 100 the rotary core methodology against traditional quantitative pits at research sites in CA, NV, and 101 NY, USA. We found that soil cores had 16% higher estimates of < 2 mm soil mass than 102 estimates obtained from quantitative pits. Conversely, soil cores had 8% lower estimates of 103 coarse fragment mass compared to quantitative pits. There were no statistical differences in 104 measured C or N concentrations between the two methods. At the individual site level, 105 differences in estimates for the two methods were more pronounced, but there was no consistent 106 tendency for cores to over or under estimate a soil parameter when compared to quantitative pits. 107 108 **Running Title:** Revisiting C and N sampling 109 110 111 Key words: Soil sampling, soil pit, soil core, coarse fragment, carbon, nitrogen

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113

INTRODUCTION

116 Estimating soil mass and rock content is an essential part of determining nutrient contents in 117 ecosystems (Harrison et al. 2003). This has become increasingly important with the current 118 interest in global climate change and soil carbon (C) content. Soils typically contain the largest 119 and most difficult pool of C to estimate (Homann et al. 2001). Several methods have been 120 utilized for measuring soil mass and rock content including: punch cores, machine-driven core 121 drills, truck mounted corers, impact hammer driven cores, and even explosives (Tuttle et al. 122 1984; Jurgensen et al. 1977; Hayden and Robbins 1975; Robertson et al. 1974; Schickedanz et al. 123 1973; McIntyre and Barrow 1972; Hayden and Heinemann 1968). However, none have proven 124 to be as universally accepted or applicable as the large-excavation, quantitative soil pit (Johnson 125 et al. 2005; Harrison et al. 2003; Hamburg et al. 1984). In 1997 researchers proposed a motor-126 driven core sampler for taking intact samples from rocky soils at the Long Term Forest 127 Productivity (LTSP) plots in southern Missouri, USA (Ponder and Alley 1997; Powers et al. 128 1989). They determined that the core device was effective at retrieving undisturbed soil cores for 129 estimation of bulk density, root biomass, and nutrient contents to a depth of 35 cm (Ponder and 130 Alley 1997). We believe that this device, a motor-driven, diamond-tipped rotary corer, has the 131 potential to supplement or replace the traditional excavated quantitative pit for estimating soil 132 mass, rock content, and nutrient concentrations through the soil profile. 133 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, 145 146 can be used to sample many locations efficiently, and allows for more straightforward estimates 147 of soil and rock mass on a volume and areal basis. Two or three people can operate the device in an area roughly 9 m^2 . The core bit is large enough to obtain a quantitative sample, but with an 148 149 internal core diameter of only 7.62 - 9.5 cm, minimizes soil excavation. We have been able to 150 core to a depth of 1 m in times ranging from 20 - 45 minutes, and deeper sampling is possible. 151 The rotary core device cuts through large coarse fragments eliminating bias introduced by 152 including or excluding large coarse fragments that protrude only partway into quantitative pits 153 (Figure 2). Calculations for estimating root, rock, and soil mass and volume are obtained directly 154 from individual core samples (Figure 3). Additionally, the rotary core device is relatively 155 portable weighing roughly 29 kg, can be transported on a pack frame over large distances and 156 rough terrain, and can be assembled using pre-existing components and easily manufactured 157 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

161	comparisons of pit and core soil samples collected in three ecosystems within the conterminous
162	US. We hypothesized that the study sites were unique to each-other and provided three viable
163	replicates for our study. Finally we proposed that if differences occurred between methodologies
164	they would be consistent across sites. We directly compared estimates of soil mass, coarse
165	fragment mass, soil organic C%, and soil total N%.
166	
167	MATERIALS AND METHODS
168	Study Design and Data Collection
169	Three study sites were chosen where existing data from quantitative soil pits had been
170	collected in order to quantify soil mass, coarse fragment content, and C and N concentrations. In
171	addition to quantitative pit data we used the core device to collect similar data immediately
172	adjacent to soil pits. Two of the sites are in the western US; one in the Great Basin southwest of
173	Austin, NV, and the other located in the Sierra Nevada Mountains northeast of Truckee, CA. The
174	third site is located in the eastern US within Tompkins County, NY.
175	
176	Experimental Areas
177	Underdown Canyon (39°15'11" N 117°35'83" W) is a Joint Fire Sciences Program
178	Demonstration Area in the Shoshone Mountain Range located in Nye County, NV on the
179	Humboldt-Toiyabe National Forest. The canyon is oriented east to west and study plots are
180	located at elevations from 2,209 m to 2,227 m. Average annual precipitation averages 25 cm and
181	arrives mostly as winter snow and spring rains. Average annual temperature ranges from -7.2 °C
182	in January to 29.4 °C in July. Lithology of the Shoshone range consists of welded and non-
183	welded silica ash flow tuff. Soils are classified as Coarse loamy mixed frigid Typic Haploxerolls.

184 The soils are extremely coarse grained and have weak to moderate structure. Vegetation is 185 characterized by sagebrush (Artemisia tridentata Nutt. ssp. vaseyana [Rydb.]) and single leaf 186 pinyon (Pinus monophylla Torr. & Frém) with lesser cover of Utah juniper (Juniperus 187 osteosperma Torr. Little), and associated grasses and forbs (Rau et al. 2005). 188 The Truckee site (39°15'9" N, 120°49'23" W) is a 12.1 ha second growth, naturally 189 regenerated, pure Jeffrey pine (*Pinus jeffreyi* Grev. and Balf.) stand located in Nevada County, 190 CA, on the Tahoe National Forest. The site has a generally northeast aspect with a slope varying 191 from 3 to 12 % at an elevation of 1,767 m. The mean annual precipitation is 69 cm, falling 192 predominantly as snow between October and May. The mean annual temperature at the study 193 site is 6 °C, and ranges from -12 °C in January to 29.4 °C in July. Soils are fine-loamy, mixed, 194 frigid, Ultic Haploxeralfs derived from andesite. Understory vegetation on the site consists of 195 sagebrush, bitterbrush (Purshia tridentata DC.), mule's ear (Wyethia mollis A. Gray), greenleaf 196 manzanita (Arctostaphylos patula Green), and prostrate ceanothus (Ceanothus prostrates Benth.) 197 (Murphy et al. 2006).

198 The Tompkins County sites (42° 16-25' N, 76° 23-40' W) near Ithaca, NY, consist of eight 199 sampling locations, two of which were never plowed while the remaining six were abandoned 200 from agriculture 50-100 years prior to sample collection (Flinn et al. 2005). The sites had 201 variable slope and aspect with a mean elevation of 292 m. Mean annual precipitation is 93 cm, 202 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 203 204 these sites consist of Dystrudepts, Fragiaquepts, and Fragiudepts developed in till deposited by 205 Wisconsinan glaciation over bedrock of Devonian shale (Neeley 1965). The dominant tree 206 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.).
- 211

212 Soil Pit Sampling

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

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

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

230	volume (> 10 mm rock mass / Db_{rock}), the < 10 mm soil volume (< 10 mm soil mass moisture
231	corrected / Db_{soil}), and > 10 mm root volume (> 10 mm dry root mass / Db_{root}) (Johnson et al.
232	2005). For the Tompkins pits, volume was calculated using measured depths for 25 points on a
233	18 cm grid (Hamburg 1984). Total pit bulk density was then calculated by dividing the estimated
234	rock and $< 2 \text{ mm}$ soil mass by the pit volume.
235	
236	Soil Core Sampling
237	Soil cores were extracted at locations corresponding to each soil pit. Soil samples
238	corresponding to the depth increments excavated in pits were removed from each bore hole for a
239	total of 72 samples at Underdown, 72 samples at Truckee, and 50 pooled samples at Tompkins (4
240	cores were taken at each pit, one at each side).
241	The method utilizes a 7.62 cm (for Underdown, NV and Truckee, CA sites) and 9.5 cm (for
242	Tompkins, NY) internal-diameter diamond-tipped core device manufactured by Diteq TM , and is
243	driven by a two-person rotary Briggs and Stratton TM power head, allowing it to core through
244	rocks and soil with minimal compaction (Ponder and Alley 1997). Each sample increment was
245	extracted before the core was driven to the next depth increment. This methodology should help
246	to further minimize compaction of each depth increment. Cores were bagged individually,
247	brought back to the lab, dried at 100° C for 48 hours, and weighed. Cores were then sieved to 2
248	mm.
249	
250	Sample Analyses
251	Soil samples < 2 mm were ground using an IKA impact head TM type mill for Underdown and
252	Truckee, and a Retsch Mixer Mill [™] , 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).

257

258 Statistical Analyses

259 We analyzed four key soil variables for differences between the three test sites and the two 260 methods used to collect the data (soil pits vs. soil cores). Variables tested included: < 2 mm soil 261 mass, > 2 mm coarse fragment mass (rock mass), soil C%, and soil total N%. All other variables 262 of interest including regolith bulk density and C and N content can be calculated using these 263 estimates. All comparisons were evaluated using SASTM generalized linear mixed effects models 264 (Proc GLIMMIX). Site and sample type differences were evaluated using site as a main effect 265 and sample type as a block within site. Soil depth and interactions terms could not be directly 266 analyzed with the mixed model because the number of depth increments and the depth of 267 individual increments were variable across sites. Mean comparisons were made with Tukey's 268 test (P < 0.05) after confirming significant main effects and interactions with the mixed models 269 (P < 0.05). Tukeys' tests were also used to evaluate differences between sample types at 270 individual soil depth increments (P < 0.05).

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RESULTS AND DISCUSSION

273 The three sites differed significantly for all four variables tested (P < 0.05). This analysis 274 confirms that the three sites provide three statistically distinct locations to test our main 275 hypothesis. When all three sites were grouped, core samples resulted in 16% higher estimates (P

276 = 0.0078) for < 2 mm soil mass when compared to soil pit samples (Figure 4). Conversely, core 277 samples resulted in 8% lower estimates (P = 0.0043) of coarse fragment mass when compared to 278 pit samples (Figure 4). Estimates of soil C% and N% were statistically similar between sampling 279 methodologies (Figure 4). 280 The simple pooling of sample type estimates may lead the reader to believe that cores 281 universally result in higher estimates of < 2 mm soil mass (Figure 4). However, this was not the 282 case in our comparison. The Sample Type x Site term in the mixed model indicates that there 283 were significant interactions for all of the variables tested (Table 1). Our comparisons of the 284 three sites indicate that there was no consistent bias for a sampling method to over- or under-285 estimate soil variables (Figure 5). This is contrary to our original hypothesis. Soil cores only 286 resulted in higher estimates of < 2 mm soil mass at the Tomkins, NY site, while estimates for 287 soil mass were similar between methods at Truckee, CA and Underdown, NV (Figure 5). Coarse 288 fragment estimates were similar between methods at Tomkins, NY and Underdown, NV, but 289 higher when estimated with pits in Truckee, CA (Figure 5). 290 It is not entirely clear why each site displayed its own unique differences between sample type

291 and regolith physical properties, but it could be due to the size and distribution of coarse 292 fragments or the method by which pit volume was estimated. If the regolith contains very few, 293 but rather large boulder size coarse fragments, the likelihood of encountering one with a large 294 quantitative pit is greater than with a small diameter soil core. This is due to the relationship 295 between cross sectional area and volume. A small increase in cross sectional area sampled can 296 result in a large change in the volume sampled. This is likely the case in Truckee, CA where 297 several very large boulders either inhibited the completion of a pit, or were removed from pits. 298 However, when soil cores were taken in Truckee, CA, we encountered no obstructions to the 60

299 cm sample increment, and removed no complete rock samples from the rotary core. Conversely, 300 if the soil profile has a more spatially uniform and heterogeneous size distribution of coarse 301 fragments it is likely that the diamond tipped rotary core will proportionately sample those 302 coarse fragments. Estimates of pit volume at the Truckee, CA and Underdown, NV sites were 303 done by back-calculating the volume of the pit from rock mass, rock density, soil mass, and soil 304 density. Pit volume estimates at the Tomkins, NY site were made by measuring the dimensions 305 of the pit. This methodology is problematic due to the inability to dig vertically walled pits, and 306 to account for large rocks protruding into the pit. Over estimating the volume of the pit would 307 result in the lower estimate of soil mass using pit measurement methodology. 308 Soil C% and N% were similar when measured with pits and cores at the Tomkins, NY site, but 309 were higher when measured with pits in Truckee, CA, and lower in when measured with pits in 310 Underdown, NV (Figure 5). The result of the inconsistent patterns in soil nutrient concentrations 311 between measurement types is unclear at this time, but clearly influences estimates of soil C and 312 N pools. One potential explanation for the lack of difference between methods at Tomkins, NY 313 could be that the core samples at this site are a composite of 4 cores taken around the perimeter 314 of the soil pit. Due to the extreme heterogeneity of the soil medium it is possible that a single 315 core does not integrate the mean soil nutrient concentration that would be obtained from a 316 quantitative pit sample. A composite sample of several cores may give a better estimate of mean 317 soil concentration in a small area around a pit. Another potential source of error in the 318 measurements of soil C and N concentration could come from the grinding of rock fragments 319 and the inclusion of these grindings into < 2 mm soil C and N concentrations. This might be 320 especially true in soils derived from sedimentary deposits which contain high concentrations of 321 C or N. (Halloway and Dahlgren 1988; Whitney and Zabowski 2004). We analyzed coarse

322 fragment chemistry as a follow up to our initial findings. We determined that coarse fragments 323 could contribute to total regolith C and total N content, but there was no bias towards greater soil 324 concentration of C and total N in cores relative to pits, that could be attributed to rock grinding. 325 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 326 327 represented by rock grinding (\approx 1 cm) would be less than 12% of the total area and volume 328 sampled. We estimated that coarse fragments account for 29 - 62% of the regolith mass using pit 329 estimates, and 39 - 58% of regolith mass using core estimates. Across the three sites coarse 330 fragments accounted for 2 - 15% of total regolith C content when measured with pits and 7 - 9% 331 when measured with cores. Coarse fragments accounted for 5 - 30% of total regolith N content 332 when measured with pits and 13 - 19% when measured with cores. The coarse fraction often is 333 assumed inert and neglected; however, several researchers have documented the importance of 334 including coarse fraction estimates in nutrient budgets (Fernandez et al. 1993; Ugolini 1996; 335 Corti et al. 1998; Harrison et al. 2003). We hypothesize that soil embedded in coarse fragment 336 pores or cracks is the dominant source of C and N associated with the coarse fraction in our 337 study. Although grinding of the coarse fraction may not be a significant source of soil C and N in 338 our study, future work is needed to test the effects of how rock grinding influences estimation of 339 other nutrient pools including base cations. Rock material is the primary source of base cations in 340 soils and therefore excessive grinding and powdering of rock material may lead to an 341 overestimation of soil base cation content.

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CONCLUSIONS

344 We believe the diamond tipped rotary core device tested in this comparison is a viable 345 alternative to quantitative soil pits. Although the core estimates were not identical to pit 346 estimates at all of our test sites, the overall difference between methods was not greater than 347 16%. More importantly it does not appear that the core device consistently over- or under-348 estimates any specific soil regolith property when compared to quantitative pits. This device has 349 the potential to increase a researcher's sample size (n) because of its relatively low time 350 requirements compared to pit sampling. This methodology will prove important in large 351 landscape scale studies with significant heterogeneity or in repeated measures studies where 352 large sample size (n) is required to detect a significant change. Furthermore we believe the core 353 device provides unbiased estimates of coarse fragment and sample volume in most soils because 354 large coarse fragments are cut clean and proportionately sampled. There are still unresolved 355 differences among individual sites for several soil properties including soil mass, coarse 356 fragment mass, and soil C and N concentrations. On certain soils it may be necessary to increase 357 the sample size to adequately characterize large coarse fragments.

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		Soil Mass		Rock Mass		Soil C%		Soil N%	
	DF	<u>F</u>	<u>P</u>	<u>F</u>	<u>P</u>	E	<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								

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

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447	Figure 1. Sample processing regime, and unit conversion for each soil pit increment excavated.
448	Figure 2. Photos of the rotary core bit, the adapter shaft used to connect it to the power head, and
449	the power head. Note how cleanly the large coarse fragment has been cut by the core device. Top
450	scale is in inches, bottom scale is in cm. Models are J.J. Klima and the corresponding author at
451	USFWS, Hart Mountain Wildlife Refuge, OR.
452	Figure 3. Sample processing regime, and unit conversion for each core increment extracted.
453	Figure 4. Means and standard errors for the two sampling methods. Double asterisks indicate
454	statistically different means (Tukey's test $p < 0.05$).
455	Figure 5. Means and standard errors for the two sampling methods at each site and depth
456	increment. Double asterisks indicate statistically different means (Tukey's test $p < 0.05$).
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470 Figure 1.



 $\begin{array}{l} \mbox{Roots} > 2\ \mbox{mm} \ (\mbox{Kg}\ \mbox{ha}^{-1}) = \{\mbox{M}_{\mbox{Root}} \ (\mbox{g}) + \mbox{M}_{\mbox{root}} \ (\mbox{g}) / \ \mbox{V}_{\mbox{pit}} \ (\mbox{cm}^3)\} \bullet \ \mbox{d} \ \mbox{cm}) \bullet 100,000,000 \ \mbox{cm}^2) \bullet \ \mbox{C} \\ \mbox{Rocks} > 2\ \mbox{mm} \ \mbox{(Kg}\ \mbox{ha}^{-1}) = \{\mbox{M}_{\mbox{Rock}} \ \mbox{(g)} + \mbox{M}_{\mbox{rock}} \ \mbox{(g)} / \ \mbox{V}_{\mbox{pit}} \ \mbox{cm}^3)\} \bullet \ \mbox{d} \ \mbox{cm}) \bullet 100,000,000 \ \mbox{cm}^2) \bullet \ \mbox{C} \\ \mbox{Soil} < 2\ \mbox{mm} \ \mbox{(Kg}\ \mbox{ha}^{-1}) = \{\mbox{M}_{\mbox{Soil}} \ \mbox{(g)} - \mbox{M}_{\mbox{rock}} \ \mbox{(g)} / \ \mbox{V}_{\mbox{pit}} \ \mbox{(cm}^3)\} \bullet \ \mbox{d} \ \mbox{cm}) \bullet 100,000,000 \ \mbox{(cm}^2) \bullet \ \mbox{C} \\ \mbox{Where} \ \mbox{(C)} = \mbox{nutrient concentration in fraction} \ \mbox{math math and} \ \mbox{d} \ \mbox{d} = \mbox{depth of the pit increment} \\ \end{array}$

Figure 2.



483 Figure 3.





