Deep-layer (>30 cm) sampling is important to accurately estimate soil carbon (C) stocks (Baker et al., 2007; Harrison et al., 2011; Syswerda et al., 2011). Volume-based estimation is typically used to quantify C stocks (FAO, 2017) and requires bulk density:

\[ C = \%C \times BD \times h \]  

where \( C \) = soil C stock (Mg ha\(^{-1}\)), \( \%C \) = carbon concentration, \( BD \) = bulk density (g cm\(^{-3}\)), and \( h \) = soil depth (cm).

Apart from quantitative pits (i.e., measuring the entire soil mass and volume), the most accurate BD samples are taken manually from the sidewalls of soil pits. The samples are taken with metal cylinders of known volume, which are driven into the soil pit wall. Yet, even these samples can be compacted depending on the method (Rogers and Carter, 1987; Folegatti et al., 2001; Siqueira et al., 2008).

The BD samples are oven-dried for 24 h at 105°C, and BD is calculated as the dry weight (DW) of the soil divided by volume of the cylinder, excluding weight of coarse fragments (i.e., >2-mm-diam. rocks and gravel) (Throop et al., 2012):

\[ BD_{pit} = \frac{(DW_t - DW_c)}{V_t} \]  

where \( BD_{pit} \) = soil bulk density (g cm\(^{-3}\)), \( DW_t \) = total dry weight (g), \( DW_c \) = coarse fragments dry weight (g), and \( V_t \) = total cylinder volume.

The pit method is disruptive and tedious and cannot effectively capture spatial heterogeneity across the landscape. Because of these limitations, many researchers began to use deep-core hydraulic samplers. Soil cores collected using a hydraulic sampler can be used for both C analysis and BD determination, of which the latter is calculated as follows:

**Abbreviations:** BD, bulk density; DW, dry weight.
where $BD_{hs} = \text{bulk density (g cm}^{-3})$ from hydraulic sampler, $FW_t = \text{total fresh weight of core segment (g), } DW_{ss} = \text{sub-sample oven-dried weight from core segment (g), } FW_{ss} = \text{sub-sample fresh weight from core segment (g); } d = \text{core segment length (cm), and } A = \text{core area (cm}^2)$. 

While this approach avoids the limitations of the pit method, the retrieved cores are prone to artificial compaction or gaps induced during sampling. This can lead to an increase or decrease of $BD_{hs}$ and an over- or underestimation of C stocks (Rogers and Carter, 1987) [Eq. 1]. Soil compaction is especially problematic. It is neither clear where this induced compaction takes place (top- or subsoil) nor which conditions consistently influence core compaction (e.g., wet vs. dry, frozen vs. unfrozen soil, soil texture, coarse fragments, experience of the sampling personnel). A recent study showed that $BD_{hs}$ was not affected compared with $BD_{pit}$ until ~30 cm (Beem-Miller et al., 2016); however, deeper soil layers may be more susceptible to sampling errors. This is increasingly important as deep-layer C estimates are required to calculate accurate C stocks and hydraulic sampling systems have become widely used. The aim of this study was (i) to estimate the error induced by hydraulic sampling and its impact on C stock estimation and (ii) to compare methods of correcting this error.

**Material and Methods**

The study was conducted on a 25.8-ha field in central Iowa, planted with corn (*Zea mays*, L.) and soybean (*Glycine max* (L.) Merr.) in annual rotation under a conventional tillage system (41.975° N, 93.694° W, 315 m asl). The soils belong to the Clarion–Webster–Niclot association of the Des Moines lobe, and are mesic Typic Hapludolls, mesic Aquic Hapludolls, and mesic Typic Endoaquolls (Hernandez-Ramirez et al., 2010). Subsoil parent material is loamy till, and soil texture is loam to clay loam. The volumetric water content was 0.34 cm$^3$ cm$^{-3}$ at time of sampling, with the groundwater depth at ~120 cm.

Three soil pits were dug to a depth of 150 cm along the downslope of the study field within 50 m in February 2017. Soil samples were taken at 0- to 15-cm, 15- to 30-cm, 30- to 60-cm, 60- to 90-cm, and 90- to 120-cm increments from three sides of each pit using stainless steel cylinders of 8 cm diameter and 5 cm height ($n = 45$ samples). All samples were processed as described above, and $BD_{pit}$ was calculated. Subsamples from each depth increment were air-dried, fine ground, and analyzed for C using a CN analyzer (Flash 1112, Thermo Finnigan).

Two soil cores (~3.8 cm diam.) were taken within 3 m at three sides of each soil pit, using a hydraulic deep soil core sampling system (Giddings Inc.) ($n = 18$ cores). The $BD_{hs}$ was estimated using Eq. [3] for the same depth intervals as $BD_{pit}$. The depth of the sampling hole ($d$) was measured, and $BD_{hs}$ was corrected for compaction (Beem-Miller et al., 2016):

$$BD_{corr} = BD_{hs} \times \frac{d}{d_s}$$

where $BD_{corr}$ = corrected $BD_{hs}$ (g cm$^{-3}$); $d = $ core length (cm), and $d_s$ = sampling hole depth (cm).

Thereafter, the C stock $C_{pit}$, $C_{hs}$, and $C_{corr}$ were calculated with Eq. [1] using $BD_{pit}$, $BD_{hs}$, and $BD_{corr}$, respectively.

In addition, the mass-based, equivalent C stock, $C_{eqm}$, was calculated (Wendt and Hauser, 2013). Here, BD is not required, and a cubic curve fit is used with cumulative soil mass (Mg ha$^{-1}$) as independent variable and cumulative C mass (Mg ha$^{-1}$) as dependent variable, calculated from the difference of $DW_t$ and $DW_s$. The $C_{eqm}$ can then be calculated for any reference soil mass ($m_{ref}$) using the curve fit parameters. In our case, $m_{ref}$ of each depth increment was set to the soil mass occupying the volume and area of a soil core segment with the bulk density of the soil pit, $BD_{pit}$ (Table 1):

$$m_{ref} = V_{hs} \times BD_{pit} / A \times 100$$

where $m_{ref}$ = reference soil mass (Mg ha$^{-1}$) and $V_{hs}$ = core segment volume (cm$^3$).

Differences and relationships among BD and C stocks were statistically analyzed with linear regression, ANOVA, and Tukey post-hoc test ($p < 0.05$). Analysis was run in SigmaPlot 11.0. The calculation of $C_{eqm}$ was run in R (see Supplemented Material).

**Results**

The difference in duplicate $BD_{hs}$ ranged from −0.29 to 0.31 g cm$^{-3}$, with soil depth 30 to 60 cm being least affected and 90 to 120 cm being most affected. The difference between $BD_{pit}$ and $BD_{hs}$ ranged from −0.27 to 0.77 g cm$^{-3}$, and soil layers 60 to 120 cm were more affected than the top soil (Fig. 1A–B). Linear regressions between $BD_{pit}$ and $BD_{corr}$ were significant, and root mean square error (RMSE) was 0.15 and 0.17 g cm$^{-3}$, respectively (Fig. 1C–D). There were significant differences among BD methods, with $BD_{corr}$ being smaller than $BD_{pit}$ and $BD_{hs}$. In contrast, $C_{pit}$, $C_{hs}$, $C_{corr}$, and $C_{eqm}$ stocks were not significantly different (Table 1), and a linear regression was significant with RMSE of 4.72, 6.31, and 6.48 Mg ha$^{-1}$, respectively (Fig. 2).

### Table 1. Mean (± SE) of C stock, bulk density (BD), and reference soil mass ($m_{ref}$) from 0 to 120 cm from pits (pit), hydraulic sampling (hs), corrected hydraulic sampling (corr), and equivalent soil mass (eqm) approach ($n = 3$ pits).

<table>
<thead>
<tr>
<th>Depth</th>
<th>$C_{pit}$</th>
<th>$C_{hs}$</th>
<th>$C_{corr}$</th>
<th>$C_{eqm}$</th>
<th>$BD_{pit}$</th>
<th>$BD_{hs}$</th>
<th>$BD_{corr}$</th>
<th>$m_{ref}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>cm</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0–15</td>
<td>61.10 ± 16.62</td>
<td>61.34 ± 17.53</td>
<td>57.95 ± 16.58</td>
<td>61.67 ± 17.71</td>
<td>1.38 ± 0.08</td>
<td>1.37 ± 0.07</td>
<td>1.29 ± 0.06</td>
<td>2.061 ± 132</td>
</tr>
<tr>
<td>15–30</td>
<td>37.85 ± 5.99</td>
<td>38.73 ± 5.97</td>
<td>36.53 ± 5.65</td>
<td>38.53 ± 4.52</td>
<td>1.40 ± 0.03</td>
<td>1.43 ± 0.03</td>
<td>1.35 ± 0.03</td>
<td>4.296 ± 253</td>
</tr>
<tr>
<td>30–60</td>
<td>39.14 ± 6.86</td>
<td>38.84 ± 6.57</td>
<td>36.65 ± 6.21</td>
<td>44.59 ± 10.13</td>
<td>1.49 ± 0.03</td>
<td>1.48 ± 0.04</td>
<td>1.40 ± 0.03</td>
<td>8,790 ± 310</td>
</tr>
<tr>
<td>60–90</td>
<td>43.92 ± 19.84</td>
<td>41.83 ± 16.6</td>
<td>39.48 ± 15.64</td>
<td>41.18 ± 17.1</td>
<td>1.58 ± 0.03</td>
<td>1.56 ± 0.08</td>
<td>1.48 ± 0.07</td>
<td>13,508 ± 300</td>
</tr>
<tr>
<td>90–120</td>
<td>77.96 ± 9.22</td>
<td>76.22 ± 3.78</td>
<td>71.48 ± 3.8</td>
<td>70.79 ± 12.37</td>
<td>1.60 ± 0.03</td>
<td>1.59 ± 0.12</td>
<td>1.49 ± 0.11</td>
<td>17,884 ± 475</td>
</tr>
</tbody>
</table>
Fig. 1. Bulk density measured in soil pits (BD$_{pit}$), soil cores (BD$_{hs}$), and soil cores with correction factor applied (BD$_{corr}$) in 0 to 120 cm (all in g cm$^{-3}$), with differences (A) among BD$_{hs}$ duplicate samples and (B) among BD$_{pit}$ and BD$_{hs}$. In addition, linear regression analysis with BD$_{pit}$ as independent variable and (C) BD$_{hs}$ and (D) BD$_{corr}$ as dependent variable. **p < 0.01.

Fig. 2. Carbon stocks calculated for soil pits (C$_{pit}$), soil cores (C$_{hs}$), correction factor applied to soil cores (C$_{corr}$) and using the equivalent soil mass approach (C$_{eqm}$) in 0 to 120 cm depth (all in Mg ha$^{-1}$). Linear regression analysis with C$_{pit}$ as independent variable and (A) C$_{hs}$ (B) C$_{corr}$ and (C) C$_{eqm}$ as dependent variables. **p < 0.01.
Discussion

The BD was affected by hydraulic sampling, and BD$_{hs}$ varied between core duplicates and relative to BD$_{pit}$, especially below 60-cm depth. Higher BD$_{hs}$ values indicate sampling-induced soil compaction, whereas lower BD$_{hs}$ values may be the result of gaps inside or alongside the soil core. The core diameter could vary among cores, so that a constant parameter $d$ in Eq. [3] may induce an additional error. While RMSE of BD$_{hs}$ versus BD$_{pit}$ and C$_{hs}$ versus C$_{pit}$ were high, these differences were not significant, similar to the results of Beem-Miller et al. (2016).

The BD$_{corr}$ significantly underestimated BD$_{pit}$, and C$_{corr}$ RMSE was higher than without correction; hence, this approach is not suitable to correct for compaction. Equation [4] assumes a constant rate of compaction along the soil core, but differences in BD were higher in the subsoil than in the topsoil. Average C$_{eqm}$ was similar to C$_{pit}$ in 0 to 15 cm but overestimated C stocks from 15 to 60 cm and underestimated C stocks from 60 to 120 cm (Table 1). The highest RMSE values were found for C$_{eqm}$ vs. C$_{pit}$ yet there was no significant difference in C stock. Our results indicate that BD$_{hs}$ can represent true BD and C in the topsoil (Table 1) but may be susceptible to under- or overestimation of subsoil BD (Fig. 1). This is partly in contrast to Beem-Miller et al. (2016), who found no significant difference between BD$_{corr}$ and BD$_{pit}$ or C$_{eqm}$ and C$_{pit}$ at shallower soil depths using quantitative pits and considering effect of rock fragments. In this study, sampling-induced errors may have been affected by shallow groundwater and high clay content. Note that we did not test sampling under different conditions, such as texture, soil textural changes with sampling depth, water content, coarse material, and sampling personnel. Therefore, further studies are needed to verify BD$_{hs}$ and the suitability of C$_{eqm}$ and BD$_{corr}$ to correct for sampling errors in subsoil.

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References


