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Determining Soil Bulk Density for Carbon Stock Calculations: A Systematic Method Comparison

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Accurate and effective determination of soil bulk density (BD) is needed to monitor soil organic C (SOC) stocks and SOC stock changes. However, BD measurements are often lacking in soil inventories and BD is estimated by pedotransfer functions with substantial uncertainty. In a systematic method comparison, we evaluated different methods for BD determination in the field by comparing the performance of MINI (5 cm^3) and BIG (250 cm^3) sample rings and of three driving hammer probes differing in diameter, material, and extraction method. Bulk density determined with 100-cm^3 sample rings was defined as the reference method (REF). All methods were tested at five depth increments in nine subplots at four sites with differing soil texture and SOC content. All methods determined BD in the depth increments with low systematic error (8% for probes and 2% for sample rings). The random error of the probe samples was, on average, 50% higher than that of the ring samples when the cores of the probes were adequately corrected for compaction or stretching. The BD was significantly overestimated (by 2%) when determined with MINI rings, and the variation in BD was not reduced with BIG sample rings rather than the smaller REF sample rings. The performance of the driving hammer technique varied widely among probe types and sites. The sheath probe had the smallest systematic error of all probes tested and is recommended for soil inventories. All methods for estimating BD had smaller errors than pedotransfer functions.

Abbreviations: BD, bulk density; CV, coefficient of variation; MPE, mean prediction error; SDPE, standard deviation of the prediction error; SOC, soil organic carbon.

Core Ideas

- **Little is known about the methodological errors of bulk density quantification.**
- **Soil probes are easy to handle, but systematic methods comparisons are lacking.**
- **We compared methods among three soil ring types and three soil probe types at four sites.**

Carbon storage in soils exceeds that in vegetation and the atmosphere (Ciais et al., 2013). Thus, small changes in soil organic C (SOC) stocks could have severe impacts on the global C cycle. Reliable measurements of C concentration are an important prerequisite for detecting such small changes in SOC stocks (Goidts et al., 2009). Information on soil bulk density (BD) is essential in converting weight-based concentration data to volume- or area-based stock data. However, BD is a parameter that is only partly or never sampled in many soil inventories (Gruneberg et al., 2014; Reynolds et al., 2013; Saby et al., 2008). Pedotransfer functions are often applied instead to predict soil BD on the basis of SOC or soil organic matter content and soil texture data (Arrouays et al., 2012). It has been shown that most pedotransfer functions are suitable only for the agro-pedo-climatic conditions prevailing at the sites used to fit these functions (Martin et al., 2009). Under different conditions, they lead to substantial systematic errors (De Vos et al., 2005; Nanko et al., 2014; Vasiliniuc and Patriche, 2015). For a soil with a BD of 1.4 g cm^{-3}, a systematic measurement error of −0.01 to −0.51 g cm^{-3} (De Vos et al., 2005) would result in SOC stocks being underestimated by 1 to 36%.

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Because soils are very heterogeneous by nature, the spatial variability in both SOC and BD is high. Moreover, BD is variable with time due to soil shrinking and swelling and due to tillage and other agricultural management operations (Hopkins et al., 2009). This limits the potential to detect significant differences in SOC stocks between sites and changes with time or requires analysis of many samples for BD and SOC to detect these differences (Kravchenko and Robertson, 2011; Schrumpf et al., 2011). Determination of SOC and BD thus has to be both accurate and rapid to allow sufficient samples to be taken to adequately capture spatial variability.

Even though BD is a basic parameter in soil science and can be deduced simply from gravimetric and volumetric analyses, accurate and precise determination of BD is challenging. Goidts et al. (2009) identified BD as one of the most important sources of uncertainty when determining SOC stocks in agricultural soil at the field scale, and this is particularly the case for the topsoil (Don et al., 2007). Most manuals and standards for soil BD determination do not specify one method but describe a range of different methods (ISO, 1998; Smith and Mullins, 2000). Because BD is defined as mass per unit volume, it is most often determined by measuring the oven-dry weight of a known sample volume (core method). Alternatively, it can be determined by measuring the oven-dry weight and volume of a natural clod (clod method) or by excavating soil and then measuring its volume by determining the volume of: (i) water (rubber balloon method) or (ii) sand (sand replacement method) needed to fill the resulting small soil pit (ISO, 1998; Smith and Mullins, 2000) or (iii) by comparing three-dimensional pictures of the surface before and after excavation (photogrammetric method; Bauer et al., 2014). There are also indirect methods that utilize the soil's effect on γ radiation (Wells and Luo, 1992) and near-infrared reflectance spectroscopy to deduce its BD (Moreira et al., 2009).

Many different tools are used within the core method (ISO, 1998). The most commonly used are sample rings of 100-cm^3 volume that are inserted horizontally into the wall of a soil pit. To sample the soil between stones or, conversely, to include stony material, sample rings with a smaller or larger volume are also applied on soils rich in coarse material. In some studies, applying the core method and investigating SOC stocks, sample rings are used in a soil pit to measure the BD, while soil samples to determine C or nutrient concentration are taken with soil probes around the pit to capture small-scale spatial heterogeneity (e.g., in German inventories of forest soils [Gruneberg et al., 2014] and agricultural soils [Bach et al., 2011]). This was devised as a compromise between precision and effort because the variation in BD was found to be smaller than the variation in SOC concentration (Don et al., 2007). Determination of both parameters in the same soil samples taken with driving hammer probes can be an effective alternative, providing the option to adequately capture the spatial variation in BD and in SOC (Don et al., 2007; Schrumpf et al., 2011; Walter et al., 2015). However, all these methods are subject to limitations such as errors due to the presence of stones and soil compaction during sampling (Lal and Kimble, 2001; Throop et al., 2012).

Field methods for BD determination in soil inventories need to be robust, simple, rapid and cheap. Robustness means that they yield accurate and precise results across a wide range of soil conditions, whereas accuracy refers to systematic deviation from the true value (systematic error or bias), and precision is the variability around the true value (random error or variability) and thus the closeness of agreement between independent measurements obtained under stipulated conditions (IPCC, 2006, p. G.9). It is questionable whether the various BD methods produce comparable results so that they can be used interchangeably. However, to date, the most common methods used for soil BD determination (the core method using sample rings and driving hammer probes) have not been systematically compared with regard to accuracy and precision.

The overall aims of this method comparison were to: (i) test the robustness of common BD methods, i.e., their accuracy and precision under largely diverging soil conditions; (ii) test the comparability of the methods, i.e., whether various core and driving hammer methods produce the same BD; and (iii) identify which of the methods is the most accurate, precise, and effective for determining soil BD to a depth of 1 m. Taking the commonly used 100-cm^3 sample rings as a reference, we tested whether sample material or the number of samples can be reduced without compromising precision by using sample rings with smaller or bigger volume. We also tested whether soil sampling with driving hammer probes can replace the more time-consuming and destructive determination of soil BD by 100-cm^3 sample rings in a soil pit. In this regard, we tested three different probes and evaluated different methods to correct for compaction or stretching of the soil cores. All methods were tested at four sites with contrasting soil texture and SOC content.

MATERIALS AND METHODS

Site Characteristics

The method comparison was performed at four sites under long-term cropland use in Lower Saxony, Germany. The time of sampling was several months after the last tillage event and thus the soil was settled. Sites with a negligible stone content, typical for cropland, were selected to test BD determination methods without this additional influencing factor. The four soils were very different with regard to texture and SOC content (Table 1). They comprised an Inceptisol with sandy texture (sand) in a research field in Braunschweig (52°18′ N, 10°26′ E), a Vertisol (clay) an Alfisol on loess (silt) near the village of Adenstedt (52°0′ N, 9°56′ E), and a Histosol (peat) close to the village of Bortfeld (52°18′ N, 10°24′ E). Peat soils contain the largest SOC stocks and their high C concentration and low BD make small errors in BD determination particularly relevant for SOC stock calculations. The terrain was flat and the slope negligible at all sites.

Soil Sampling

All sites were sampled in March 2011. In May 2012, the sandy soil was resampled about 50 m away from the first sampling plot to test the temporal reproducibility of the methods, e.g., by resampling under conditions with different soil water content. The soil was drier at the second sampling, with mean water content in the 0- to 30-cm soil depth of 10%, compared with 16% at the first sampling.

For each sampling site and sampling occasion, a 15-m-long, 1-m-wide, and 1-m-deep soil pit was excavated and partitioned into nine subplots of 1.5-m length (Fig. 1). In each of these subplots, soil samples were taken to determine the BD at five fixed depth increments (0–10, 10–30, 30–50, 50–70, and 70–100 cm) using six different methods. The methods included three different sample rings and three different driving hammer probes.

Sample rings of 100-cm^3 volume (53-mm diameter by 51-mm height; Eijkelkamp) were used as the reference method (REF) because this follows ISO (1998) and is the common sample ring size in German soil surveys. In addition, small sample rings of 5-cm^3 volume (MINI; 18-mm diameter by 20-mm height, custom-made product), and large sample rings of 250-cm^3 volume (BIG; 84-mm diameter by 50-mm height, Eijkelkamp) were used to determine BD. The sharpened steel cylinders were attached to dedicated ring holders and inserted horizontally into the soil profile. The soil-filled rings were carefully excavated, detached from the ring holder, and cleaned from soil adhering to the ring. The soil samples inside the ring were then placed in paper bags, where they were stored until drying. At each depth increment in each subplot, seven samples were taken with the REF and MINI rings and four samples with the BIG sample rings. Thus, for each soil depth and site, 63 replicates were taken for REF and MINI and 36 replicates for BIG. Before sampling, the exact positions were marked in the profile using a stencil to ensure the same layout of sampling design for all subplots and sites (Fig. 1).

At a distance of 1 to 2 m behind the soil profile, soil cores were taken with driving hammer probes. These probes differed with respect to diameter, shape of the cutting ring, and sample extraction technique (see photos in the supplementary material). The core sampler (sheath probe) has an inner diameter of 60 mm, the soil core is stored in a polyethylene film liner inside the cylinder, and the cutting shoe is screwed to the core sampler (Nordmeyer Geotool GmbH). The soil column cylinder sampler (window probe) is larger (inner diameter 84 mm; Eijkelkamp), and the core cutter for this probe is left in the soil and needs to be removed after the cylinder. The soil sample can be extracted from the cylinder via a window in the probe. With an inner diameter of 100 mm, the sampler with a polyvinylchloride (PVC) liner (liner probe) was the largest of the probes tested (Carl Hamm GmbH). The core cutter of this probe remains attached, and the soil core is stored in a PVC tube inserted in the cylinder.

One soil core was taken in each of the nine subplots per site with each of these probes. The sampling locations for these different probes were also marked (Fig. 1). All soil column cylinder samplers were hammered into the soil to the 1-m depth with an electrically driven percussion hammer (Wacker EH 23, Wacker Neuson) and recovered with manual (for the window probe; Eijkelkamp) or hydraulic extractors (for the sheath and liner probes; Eijkelkamp). All cores were cut according to the five depth increments at 10, 30, 50, and 70 cm. For each soil core, the total length of the core and the depth of the hole were also recorded to allow compaction and stretching during the soil coring procedure to be accounted for.

All samples were dried at 105′C to constant weight, and the total dry mass was divided by the sampled volume to obtain the BD value. Because the soil did not contain particles >2 mm at any of the sites, the fine soil density is equivalent to the BD.

Table 1. Soil properties: Clay, silt, sand, and soil organic C (SOC) contents at different depths in the four soils sampled.

Sampled soil	Depth	Clay	Silt	Sand	SOC
	cm	%			
Sand	0–10	5	27	68	1.39
	10–30	6	26	68	1.09
	30–50	4	12	84	0.08
	50–70	5	1	94	0.05
	70–100	5	2	93	0.06
Silt	0–10	14	80	6	1.26
	10–30	15	81	4	1.24
	30–50	15	81	4	0.69
	50–70	16	81	3	0.23
	70–100	18	80	2	0.10
Clay	0–10	36	51	13	2.02
	10–30	38	49	13	1.66
	30–50	57	37	6	0.85
	50–70	56	36	8	0.58
	70–100	57	38	5	0.69
Peat	0–10	nd†	nd	nd	17.43
	10–30	nd	nd	nd	13.62
	30–50	nd	nd	nd	42.49
	50–70	nd	nd	nd	26.07
	70–100	16	78	6	1.57

† nd, not determined.

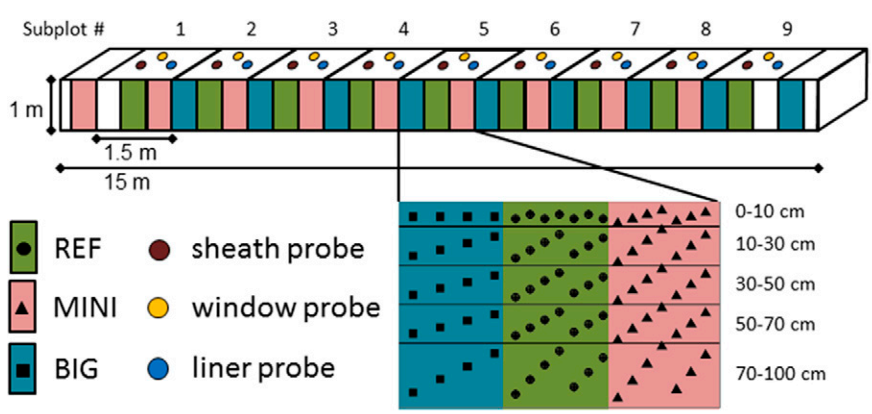

Fig. 1. Sampling scheme applied for all sites and sampling campaigns, indicating the positions used for sample ring sampling (REF, MINI, and BIG) marked with a stencil in Subplot 5 and those of the driving hammer probes (sheath, window, and liner) in all subplots.

Correction Techniques for Bulk Density Calculations

During soil sampling with driving hammer probes, soil compaction or stretching may occur due to, e.g., vibrations originating from the machine. Various correction techniques to optimally account for these changes in soil core length were tested (Fig. 2). Bulk density values calculated for the depth increments sampled with the different probes without accounting for any compaction or stretching are designated uncorrected BD (BD_{unc}) in the following. The simplest correction technique allocated the difference between the length of the soil core ($d_{tot,core}$) and the depth of the hole ($d_{tot,hole}$) linearly to the entire core (Fig. 3):

$$d_{i,\mathrm{cor}} = \frac{d_{\mathrm{tot,\ hole}}}{d_{\mathrm{tot,\ core}}} d_{i,\mathrm{unc}} \qquad [1]$$

where $d_{i,cor}$ is the corrected and $d_{i,unc}$ the uncorrected length of the ith depth increment. The BD values obtained by this linear correction are designated BD_{lin} in the following.

In reality, however, soil in the uppermost depth increment tends to be loosened and stretched, while the subsoil below 50 cm tends to be subject to compaction. Thus, a second correction method that accounts for compaction and stretching effects varying within the soil profile ($BD_{profile}$) was tested (Fig. 3). In this correction method, a fixed stretching of 1 cm was assumed for the depth increment 0 to 10 cm, while the depth increments 10 to 30 and 30 to 50 cm were assumed to remain unchanged, as observed at many of the soil cores. The corrected length $d_{i,cor}$ of the two bottom depth increments was calculated as

$$d_{i,\mathrm{cor}} = \frac{d_{\mathrm{tot,\ hole}} - 50 + 1}{d_{\mathrm{tot,\ core}} - 50} d_{i,\mathrm{unc}} \qquad [2]$$

Third, correction techniques that were optimized for each type of probe ($BD_{opt\ probe}$), each site ($BD_{opt\ site}$), and each type of probe at each site ($BD_{opt\ ps}$) were applied. The most adequate $d_{i,cor}$ was identified by minimizing:

$$\sum_{i=1}^{5} \left[\left| \sum_{j=1}^{9} \left(\mathrm{BD}_{\mathrm{REF}i} - \mathrm{BD}_{j,\mathrm{cor}} \right) \right| \right]_i \qquad [3]$$

where BD_{REFi} is the mean BD determined with the REF ring at the respective site in the respective ith depth increment and

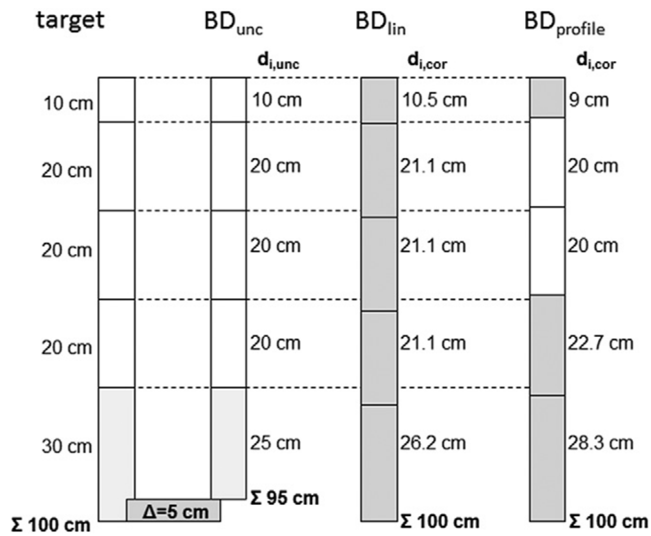

Fig. 3. Example of correction for 5-cm compaction of a soil core when the soil was sampled to the 1-m depth. The schemes depict the soil cores and the depth increments (d_i) of the targeted sampling (target), the extracted soil core (BD_{unc}), and the hypothetical core after linear correction of the compaction (BD_{lin}), after additional correction for loosening in the uppermost d_i, and compaction or stretching occurring only in depth increments below 50 cm ($BD_{profile}$).

$BD_{j,cor}$ is the BD calculated for the jth subplot from the currently tested $d_{i,cor}$, which is

$$d_{i,\mathrm{cor}} = d_{i,\mathrm{unc}} - a + b\left(d_{\mathrm{tot,\ hole}} - d_{\mathrm{tot,\ core}} \right) \qquad [4]$$

The values of $BD_{opt\ probe}$, $BD_{opt\ site}$, and $BD_{opt\ ps}$ were calculated from the respective most adequate $d_{i,cor}$ (Supplemental Table S1). The second sampling campaign at the sand site was considered an extra site in Eq. [3] because the performance of the probes differed as much between the two campaigns as among the four sites.

Data Handling and Statistics

The final data set comprised 4274 BD data points (1458 REF ring, 1464 MINI ring, 818 BIG ring, 175 sheath probe, 160 window probe, and 199 liner probe measurements). At the peat site, the depth increment from 70 to 100 cm was eliminated because the peat layer was shallower than 80 cm. Missing data due to unclear labeling, loss of sample material, or shorter soil cores further reduced the target data set by 4%, while elimination of outliers reduced it by <1%. The outliers eliminated were identified by visual checks of the histogram and boxplots of the BDs for each method, site, and depth increment, confirmed by the Grubb's test for outliers (Komsta, 2011), and tagged with comments during sampling or in the laboratory protocol indicating potential problems.

Spatial autocorrelation of BD was examined for each depth increment at each site with semivariograms of the REF data. Because no autocorrelation was detectable

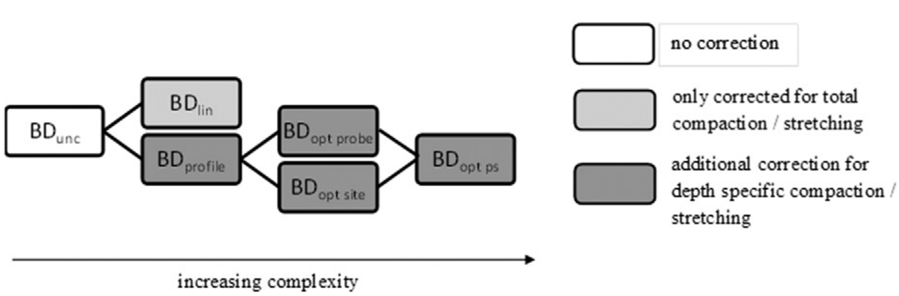

Fig. 2. Schematic diagram of correction techniques applied for compaction or stretching of soil cores.

in 75% of all cases and the range of autocorrelation was larger than the length of a subplot in only 8% of cases, autocorrelation was not considered for optimization of BD correction. We also disregarded spatial dependencies for the calculation of mean and median BDs and the associated standard deviations and errors. However, we did account for spatial dependencies in further statistical analyses by including subplot as a random effect in linear mixed effect models.

The general data evaluation and the comparison of the BD values obtained by the different methods with the BD determined with the REF sample rings (BD_{REF}) for the depth increments at each site were based on the $BD_{profile}$ data. For assessment of the accuracy of BD determination with the driving hammer probes regarding the entire sampled soil depth, the combined mass was calculated (sum of all samples from the surface up to the target soil depth) and divided by the respective volume of the hole. These mean BDs of the total soil cores ($BD_{probe,mean}$) were compared against $BD_{REF,mean}$ and $BD_{profile,mean}$, which are the weighted means of the BD_{REF} and $BD_{profile}$, respectively, of the entire sampled soil depth.

The first comparison of the six methods to determine BD was based on the respective means, medians, and quartiles, standard deviations and standard errors of the means, as well as the coefficient of variation (CV). Differences in the CV relative to the respective CV of the BD_{REF} were analyzed by paired *t*-tests and regarded as significant at $p < 0.05$. The sample size needed for detecting a BD difference of 5% was calculated for each site, depth increment, and method with power analyses using the pwr package (Champely, 2015) of the R software Version R-3.1.1 (R CoreTeam, 2013), which was used for all statistical analyses. This 5% difference was chosen as an example for an intended minimum detectable difference between two inventories. The power was set to 0.8 and the significance level to 5%.

In a second step, the six methods were compared comprehensively by linear mixed effect models using the R software's nlme package, which is applicable for linear and nonlinear mixed effects models (Pinheiro et al., 2013). The models were fitted on the reduced data set (4247 data points; see above). The study sites, depth increments, and subplots were taken into account as random factors. Because homoscedasticity was not assured, the variances were weighted by site. The methods were tested as fixed effect. The best model was selected using the Akaike information criterion (AIC) during successive model expansion. For the best fits, R^2 was assessed as the quadratic correlation coefficient (coefficient of determination) between modeled and measured BD.

As a measure of the accuracy (or systematic error or bias), the mean prediction error (MPE) was calculated for each method and correction technique:

$$\mathrm{MPE}=-\sum\left(\mathrm{BD}\ -\mathrm{BD}_{\mathrm{REF}}\right) \qquad [5]$$

As a measure of the precision (or random error), the standard deviation of the prediction error (SDPE) was calculated:

$$\mathrm{SDPE}=\sqrt{\frac{1}{n-1}\sum_{i=1}^{n}\left[\left(\mathrm{BD}_i-\mathrm{BD}_{\mathrm{REF}}\right)-\mathrm{MPE}\right]^2} \qquad [6]$$

where BD_i is the BD determined with the respective method and correction technique for each *i*th combination of replicate, depth increment, subplot, and site and BD_{REF} is the mean of the BD determined with the REF method for the respective depth increment and site. The SDPEs were plotted against the MPEs to compare the performance of the various methods. The closer the points are to the origin, the better the overall accuracy of the method.

RESULTS

Bulk Density Determined with Sample Rings

The mean BD_{REF} ranged from 0.20 g cm^{-3} in the 30- to 50-cm depth increment at the peat site to 1.70 g cm^{-3} in the 50- to 70-cm depth increment at the silt site. The sand and silt sites had similar mean BD (1.58 g cm^{-3} for 0–100 cm), while the mean BD was lower at the clay site (1.47 g cm^{-3}) and peat site (0.38 g cm^{-3}). Except for the peat site, there were clear depth profiles, with the lowest BD values in the 0- to 10-cm depth increment and the highest BDs below the 30-cm depth (Fig. 4A). At the peat site, the BD decreased with soil depth, which is typical for drained and degraded peat soils (Schwärzel et al., 2002). There were some outlier values of BD in the 30- to 50-cm depth increment at the peat site, indicating a transition zone from the more dense material in the 10- to 30-cm depth increment (mean BD of 0.56 g cm^{-3}). In line with the high variability and very low BD, the CV was largest for the 30- to 50-cm depth increment at the peat site (25%, Fig. 4B). However, no roots or faunal channels or cracks were observed in the peat that might explain the high variability. The lowest CV was found for the 50- to 70- and 70- to 100-cm depth increments at the silt and clay sites, respectively (both <2.5%). The accuracy of the alternative methods is evaluated in the following based on the comparison with BD_{REF}.

The BD values determined with the MINI sample rings were significantly higher than those determined with the REF rings (Supplemental Table S2). On average, the difference in mean BD for a particular depth increment determined with the MINI and REF rings was 1.6% or 0.02 g cm^{-3} (Fig. 4C). The overestimation of BD with the MINI sample rings was largest at the sand site, in particular for the second sampling campaign. Taking all sites and depth increments into account, the CV of the BD values determined with the MINI sample rings (8.1%) was significantly higher than that of the BD_{REF} (6.7%). At the silt, clay, and peat sites in particular, BD values obtained using the MINI sample rings had higher CVs than those obtained using the REF rings (Fig. 4B).

There was a small but significant difference between BD measured with the BIG sample rings and BD_{REF} (Supplemental Table S2). The BIG sample rings underestimated the BD by, on average, 0.7% or 0.006 g cm^{-3} (Fig. 4C). At the clay site, the BD determined with the BIG sample rings was smaller than the BD_{REF} for all depth increments. Across all sites, the CVs

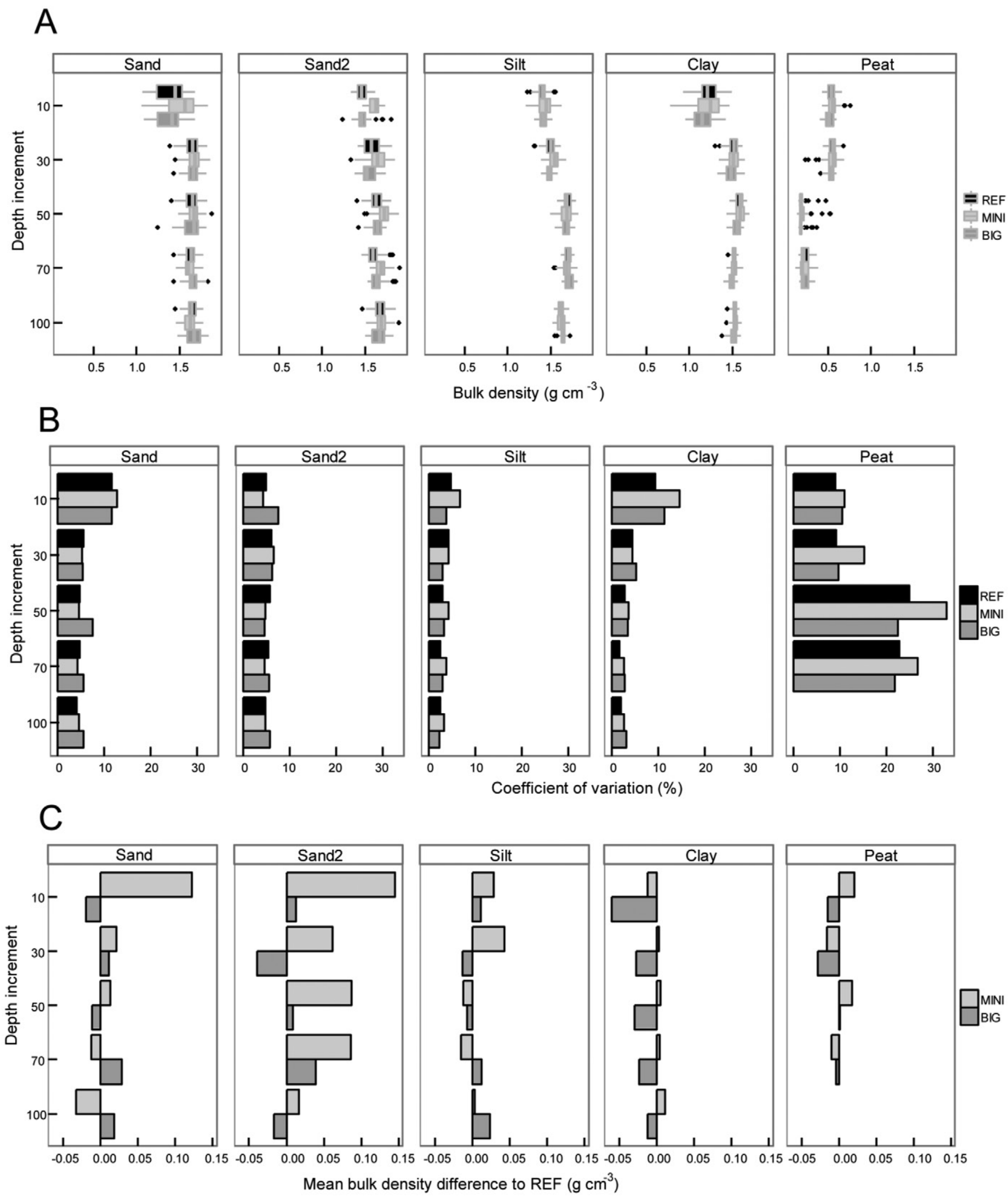

Fig. 4. Bulk density (BD) determined with reference (REF), MINI, and BIG sample rings for five depth increments at four sites: (A) boxplots showing the median, quartiles, and outliers of BD measurements, (B) respective coefficients of variation, and (C) systematic error of mean BD determined with the sample rings relative to the respective mean BD_{REF}.

of BD values obtained using the BIG sample rings were in the same range as observed for the REF sample rings. However, the CVs were somewhat lower at the peat and silt sites and higher at the sand and clay sites. Consequently, more samples were required to detect a BD difference of 5% with the BIG sample rings than with the REF sample rings at the sand and clay sites, but fewer samples were needed at the peat and silt sites (Table 2; Supplemental Table S3).

Table 2. Comparison of soil profile mean bulk density (BD) determined with different sample rings (REF, MINI, and BIG) and driving hammer probes (sheath, window, and liner) at different sites (sand, silt, clay, and peat): mean BD (g cm^{-3}) and means across depth increments of standard deviation, systematic error (SysE; the difference between the mean BD and respective mean reference BD), coefficient of variation, and the number of samples needed from each of two plots to detect a difference of 5% from the mean BD determined with the respective methods at the different sites (*N*; $\Delta = 0.05 \times$ mean BD; significance level $p < 0.05$; power = 0.8).

Ring or probe type	Sand					Sand 2					Silt					Clay					Peat				
	Mean	SD	SysE	CV	N	Mean	SD	SysE	CV	N	Mean	SD	SysE	CV	N	Mean	SD	SysE	CV	N	Mean	SD	SysE	CV	N
	— g cm^{-3} —			%		— g cm^{-3} —			%		— g cm^{-3} —			%		— g cm^{-3} —			%		— g cm^{-3} —			%	
REF	1.59	0.10		6	30	1.59	0.08		5	20	1.58	0.05		3	9	1.47	0.06		4	17	0.38	0.05		16	206
MINI	1.61	0.10	0.02	6	33	1.67	0.08	0.08	5	17	1.59	0.07	0.01	4	15	1.47	0.08	0.00	6	34	0.38	0.07	0.00	21	340
BIG	1.59	0.11	0.01	7	36	1.59	0.09	0.00	6	24	1.58	0.05	0.01	3	7	1.44	0.07	−0.03	5	24	0.37	0.05	−0.01	16	188
Sheath probe	1.60	0.11	0.01	7	35	1.52	0.15	−0.07	10	74	1.63	0.12	0.05	8	57	1.52	0.06	0.05	4	11	0.36	0.07	−0.03	17	262
Window probe	1.67	0.11	0.08	6	28	1.52	0.20	−0.07	13	118	1.61	0.04	0.04	3	6	1.53	0.05	0.06	4	16	0.41	0.09	0.03	25	647
Liner probe	1.67	0.09	0.08	6	35	1.53	0.16	−0.06	11	92	1.64	0.07	0.06	5	17	1.55	0.07	0.08	4	16	0.39	0.04	0.01	12	99

Compaction and Stretching during Sampling with Driving Hammer Probes

While sampling the soil with driving hammer probes, the soil cores were compacted by up to 16% (window probe in the sand) and stretched by up to 10% (sheath probe in the silt) (Supplemental Fig. S1). There were significant differences among probe types, sites, and sampling campaigns in this regard. With the sheath probe, the length of the soil cores was changed, on average, by 6 ± 3% (compaction of 7 ± 4 and 3 ± 2% at the sand and peat sites, respectively, but stretching of 8 ± 2 and 4 ± 2% at the silt and clay sites, respectively). The soil cores of the window probe were compacted, on average, by 6 ± 5%, while the change was significantly smaller for soil cores taken with the liner probe (3 ± 3%). Compaction was very site specific and differed between the first and second sampling campaigns at the sand site. For all probes, the greatest compaction was observed at the second sampling campaign at the sand site.

Bulk Density Determined with Driving Hammer Probes

Bulk density determined with the probes (BD_{unc}) differed systematically by, on average, 2% from BD_{REF}, which is a mean systematic error of 0.02 g cm^{-3}. However, for individual soil depth increments, the systematic error was up to 0.39 g cm^{-3} (sheath probe). The liner probe showed larger systematic errors (0.04 g cm^{-3} or 3%) than the window probe (0.02 g cm^{-3} or 2%) or the sheath probe (<0.01 g cm^{-3} or <1%). The random error was, for all probe types, around 0.2 g cm^{-3}, which is equivalent to a CV of 9%. The systematic and random errors in probe-derived BD varied widely among sites and probe types and changed substantially with optimized correction techniques (systematic error 1–20%, random error <0.05–0.41 g cm^{-3}).

Taking the compaction or stretching into account and correcting the BDs for these changes reduced the random error and thus increased the precision of BD determination. The most pronounced effect of the different correction techniques on the BD was observed for the 0- to 10- and 70- to 100-cm depth increments at the sand site (Supplemental Fig. S2). For all probes, the best correction technique was that optimized for each type of probe at each site ($BD_{opt\ ps}$), followed by the correction to $BD_{opt\ site}$ and $BD_{opt\ probe}$. Except for the sheath probe, the second, non-site-specific correction to the $BD_{profile}$ also reduced the random error compared with the linear correction technique (Fig. 5).

The $BD_{probe,mean}$ of the whole soil core was mostly comparable to the $BD_{REF,mean}$, the weighted mean of the BD_{REF}, with no general underestimation or overestimation with specific probe types (Supplemental Fig. S3). The particularly high $BD_{probe,mean}$ values for the sheath probe at the silt site were accompanied by high measured compaction of the soil cores (Supplemental Fig. S1). Apart from that, the difference between $BD_{probe,mean}$ and $BD_{REF,mean}$ generally decreased with increasing soil depth (Supplemental Fig. S4). It decreased by up to 90% when the entire soil core was taken into account instead of only the 0- to 10-cm depth increment.

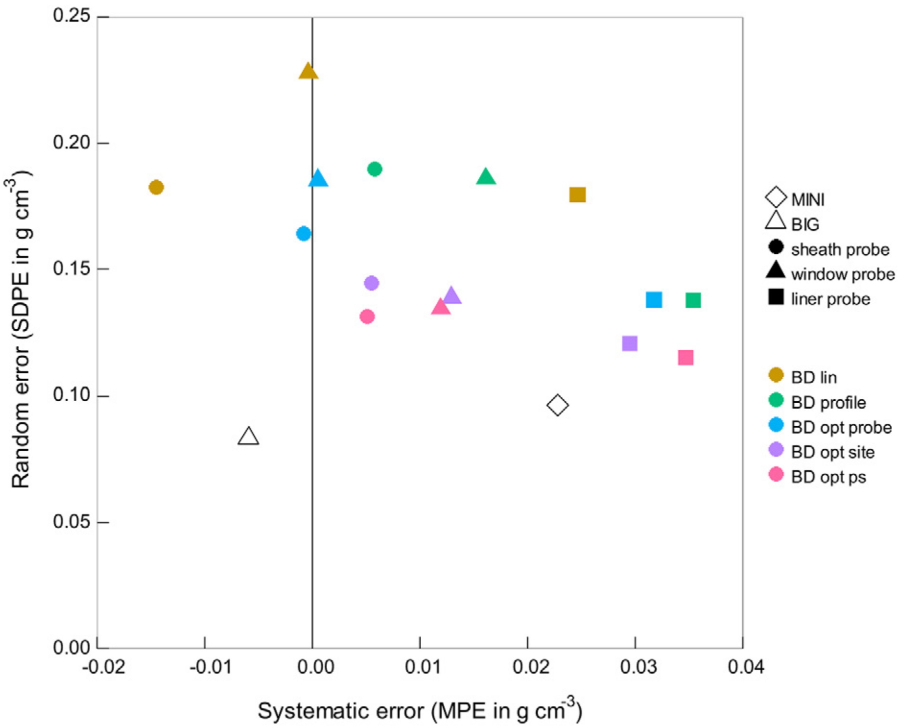

Fig. 5. Random error vs. systematic error for all methods (shapes) and correction techniques (colors) used to determine soil bulk density (BD): random error is given as the mean prediction error (MPE) plotted against the systematic error given as the standard deviation of the prediction error (SDPE). The correction techniques included stretching and compaction linearly corrected for the whole core (BD lin) or varying within the soil profile (BD profile) and optimum correction specific for each probe type (BD opt probe), for each site or soil type (BD opt site), or for each site and probe type (BD opt ps).

While $BD_{REF,mean}$ did not differ significantly (2% deviation) between the first and second sampling campaign at the sand site, $BD_{probe,mean}$ for all probe types differed substantially (9–17% deviation), indicating that sampling under different soil conditions may lead to substantial variability in the accuracy of the BD determination with driving hammer probes. This low reproducibility was also apparent in the $BD_{profile}$ of individual depth increments at the sand site. The first sampling campaign resulted in a higher $BD_{profile}$ than BD_{REF}, the second in a lower $BD_{profile}$ than BD_{REF}.

The systematic and random errors in the $BD_{profile}$ also differed widely among the sites, depth increments, and probe types. While no significant difference was detected between $BD_{profile}$ of the sheath probe and BD_{REF}, $BD_{profile}$ determined with the window probe was significantly higher than BD_{REF} based on the linear mixed effects model that included all methods, sites, depth increments, and subplots (Supplemental Table S2). This difference was, on average, 2% or 0.03 g cm^{-3} for the mineral soils and 8% or 0.02 g cm^{-3} for the peat site. Moreover, a significantly higher $BD_{profile}$ was determined with the liner probe than with the REF ring (on average, 3% or 0.04 g cm^{-3} higher BD). However, this difference was mostly driven by overestimation of the BD in the 70- to 100-cm depth increment. At some sites and for some depth increments, the accuracy was particularly low. For example, no matter which probe type was used, the $BD_{profile}$ determined with the driving hammer technique was higher (11–24%) than the BD_{REF} in the 70- to 100-cm increment at the silt site. At the clay site, the same was true for the 0- to 10-cm depth increment (12–25% overestimation).

The CV of the $BD_{profile}$, as a measure of the precision, varied between 1.3 and 58.4% (both determined with the window probe in the 10–30-cm depth increment of the clay site and the 50–70-cm depth increment of the peat site, respectively) (Fig. 6B). Considering all sites and depth increments, the CVs of the probes did not differ significantly from that of the BD_{REF}. However, the CV was much larger in specific cases, for example for the BD determined with all driving hammer probes at the second sampling campaign at the sand site or for the BD determined with the sheath probe at the silt site (Fig. 6B; Table 2). The CV was particularly large in the 0- to 10- and 50- to 70-cm depth increments at the peat site for the BD determined with the sheath and window probes, respectively. This high variability also led to the largest number of samples required to detect a 5% difference from the mean BD at the peat site, with, on average, 262 and 647 samples with the sheath and window probes, respectively, compared with 206 samples with the REF rings (Table 2). Conversely, the low CVs of BD values determined with the sheath and window probes at the clay site imply that fewer samples were required to detect a 5% difference in BD (on average, 11 and 16 samples, respectively) compared with the REF method (on average, 17 samples).

Aggregated Comparison of All Methods

The REF sample rings were considered to produce the "true" BD value in this method comparison. Accuracy therefore meant comparability to the REF sample ring results. The BIG sample rings permitted the most accurate and precise determination of BD because the systematic error (MPE) and random error (SDPE) were smallest among all methods tested (Fig. 5). The SDPE of the MINI sample rings was only slightly higher but the MPE was larger, reflecting the small systematic overestimation of BD with these rings. The systematic error in the $BD_{profile}$ determined with the sheath and window probes was similar or even smaller than that for the MINI sample rings. A higher systematic error was observed only for BD values determined with the liner probe. The different correction techniques for BD determined with the driving hammer method considerably reduced the random error (SDPE) for all probe types. With the best correction methods, the random er-

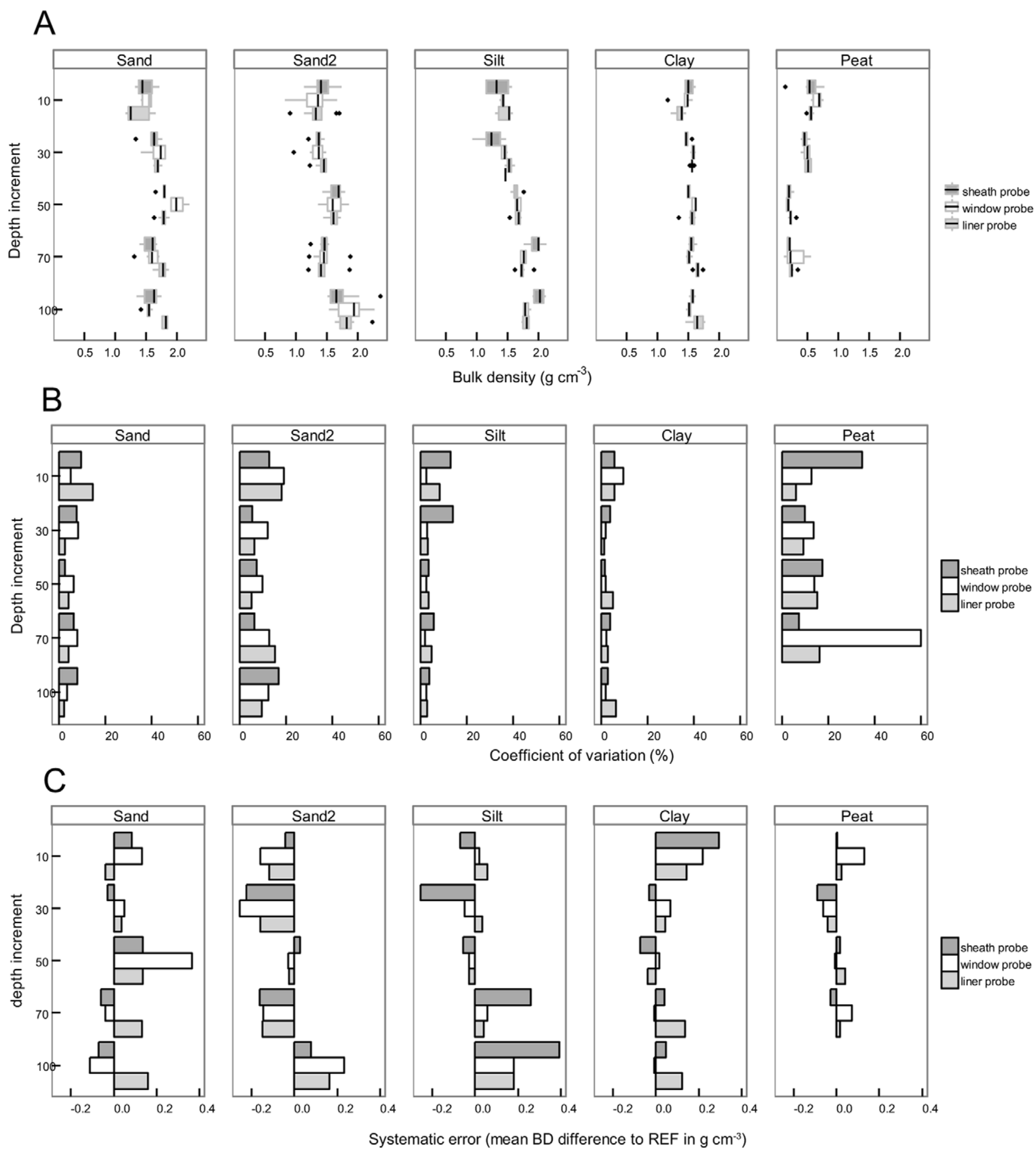

Fig. 6. Bulk density (BD) determined with sheath, window, and liner driving hammer probes for five depth increments at four sites: (A) boxplots showing the median, quartiles, and outliers of BD measurements, (B) respective coefficients of variation, and (C) systematic error of mean BD determined with the probes relative to the respective mean reference BD (REF).

ror in BD determined with soil probes was only 50% higher than that of BD determined with soil rings. However, the systematic error, expressed as the MPE, was reduced only for the sheath probe, but increased for the window and liner probes in the course of optimization of the BD correction methods.

In the linear mixed effects model based on the data set comprising four different soils, no additional soil parameter (SOC, sand, silt, or clay content) had a significant influence on the difference between the BD determined with the alternative methods and the REF sample rings. However, different optimal correction techniques at different sites indicated considerable site-specific differences in the performance of the driving hammer probes for BD determination. Thus, there is no universal rule for optimizing corrections and probe choice.

DISCUSSION

Optimization of Bulk Density Determination by the Choice of Sample Ring

The use of MINI sample rings led to overestimation of BD compared with BD determined with REF sample rings. This effect was particularly strong in the sandy soil, which is generally prone to compaction due to low structural stability. In a study comparing different sampling devices for soil BD determination to the 15.2-cm depth in a loam soil in North Dakota, Cihacek et al. (2015) did not find significant differences between BD values determined with different tubes inserted vertically into the soil. However, the detected differences, of around 3%, are comparable to the differences between the BDs determined in the present study with the MINI and REF sample rings. One potential reason for higher BDs determined with devices of smaller diameter is compaction due to friction at the cylinder wall. If the ratio of the cylinder wall to the volume is high, this friction will have a stronger influence on the accuracy of the BD determination than if the ratio is small. There was notably larger overestimation of BD with the MINI sample rings at the second sampling of the sand site compared with the first sampling. Potential reasons are differences in the soil water content during sampling and different technicians sampling the soil (see below). Due to large differences between sites and even within one site, no generally applicable correction factor for BD determined with MINI rings could be derived to correct for the overestimation of BD. As a consequence, MINI sample rings can only be recommended for soils where other sample rings cannot be applied, e.g., due to high stone content.

The higher CV found for BDs determined with MINI compared with REF and BIG sample rings contradicts the results of Cihacek et al. (2015), who found higher CVs for BDs determined with large-diameter tubes than with small-diameter tubes. However, we also found that the CV of the BIG sample rings was in the same range as that of the REF sample rings. Thus, similar numbers of replicates have to be collected with BIG samples rings, and thus a larger sample mass, compared with the smaller REF sample rings. Nevertheless, the overall accuracy of the BIG sample rings was good because the systematic error was generally small. An exception was the clay site, where BD values determined with BIG sample rings were smaller and the CV was slightly higher than that of the BD_{REF}. It can be speculated that the risk of soil crumbs being lost during sample ring preparation, which involves refilling the ring with loose material, is particularly high for sample rings with a large cross-sectional area. Therefore, even for sample rings with the recommended volumes (100–400 cm^3) (ISO, 1998), the cross-sectional area has to be considered in addition to the cylinder wall/volume ratio when choosing the optimal sampling device for a given soil.

Replacing Sample Rings with Driving Hammer Probes for Bulk Density Determination

The BD values of specific depth increments determined with sample rings were in every case more accurate than those determined with probes. Among the driving hammer probes, the liner probe had the lowest random error (7%) in BD values, but these values were significantly higher than those of the BD_{REF} (3%). Due to its highest systematic error of all probe types tested, the liner probe appears less suitable for BD determination. The systematic error was the lowest (<1%) with the sheath probe, but the random error was still 50% above that of the sample rings. The window probe displayed substantial systematic and random errors at the sand and peat sites, respectively, and is therefore not recommended. One potential reason is that while opening the window probe and extracting the soil, sample material may be lost.

Based on our results, we recommend the sheath probe for soil BD determination due to its small random error (8%) and smallest systematic error (<1%). The window probe can only be recommended for mineral soils (random error 7% and systematic error 2%). However, the performance of the probe types was site specific, and good agreement with BD_{REF} was only achieved with adequate correction for stretching and compaction of the soil probe core.

Compaction and Stretching of the Soil Core and Agreement with Reference Bulk Density of the Total Soil Depth

The impact of percussion drilling on BD in soil cores sampled with driving hammer probes, and thus on the difference between the length of the soil core and the sampled soil depth, may be due to: loosening of topsoil, compaction of subsoil, and stretching during extraction. Parfitt et al. (2010) compared the driving hammer method and the carving method for soil sampling down to the 1-m depth at 44 sites and found lower BD when determined with the driving hammer method, an effect that they attributed to disturbance of the soil structure. We observed both stretching and compaction of soil cores. While the soil cores were mostly longer than the sampled soil depth at the clay site, they were compacted at the sand site, in particular at the second sampling date when the soil was drier. This is in line with the results of Parfitt et al. (2010), who found smaller impacts of hammering on BD determination at higher soil water contents, and Hopkins et al. (2009), who highlighted the challenge of reproducible BD quantification due to seasonal variability in BD with shrinking and swelling of soil minerals. Estimating BD robustly thus remains challenging. However, the results for the resampled sand site showed little difference in BD when determined with sample rings but large differences when determined with probes. This indicates lower reproducibility of probe-derived BD and explains its higher variability.

Considering all sites, the soil cores sampled with the liner probe were the least compacted or stretched, but the accuracy of the corrected $BD_{profile}$ was the lowest of all three probe types tested. Thus, core length is no clear quality indicator as long as compaction or stretching is corrected for. The dimensions of the probe also have to be considered for the driving hammer method (Cihacek et al., 2015; Raper and Erbach, 1985; Smith and Mullins, 2000). Additional factors that may have a strong influence include the frequency or power of hammering (Smith and Mullins, 2000), the degree to which the probe is augered instead of pushed into the soil, the shape of the cutting shoe (Raper and Erbach, 1985), the resulting difference between the inner diameter of the sample probe and the diameter of the soil core, and the friction of the soil core in the probe during sampling. Thus, the diameter of the probe alone is not a reliable indicator of the quality of probe measurements.

The comparison of the mean BD of the whole soil core ($BD_{probe,mean}$) with the weighted mean of the BD_{REF} ($BD_{REF,mean}$) showed that underestimation or overestimation of the BD due to stretching or compaction of the whole soil core was low ($1 \pm 6\%$) when the total sampled soil depth was considered. Use of the sheath probe to determine BD at the silt site constituted the only exception, where overestimation of $BD_{probe,mean}$ and at the same time substantially longer soil cores than the sampling depth (6–10-cm difference) pointed to errors regarding measurement of the length of the hole (see below). We found greater variability of the BD in the topsoil than the subsoil (Fig. 6B), confirming the findings of Don et al. (2007). Thus, soil probes can be considered most suitable for deep soil sampling to determine BD and SOC stocks for the whole soil profile.

Agreement with Reference Bulk Density of Specific Depth Increments and Performance of Correction Techniques

The agreement of probe-derived BD with the BD_{REF} was improved with advanced correction techniques, e.g., when the compaction or stretching of cores was not corrected linearly for the whole core (BD_{lin}) but for the specific depth increments where stretching and compacting occurred ($BD_{profile}$). This means that compression or stretching was not homogeneously distributed within the soil core (Poeplau and Don, 2013; Walter et al., 2015). Consequently, the correction has to be adjusted to the respective location of compaction and stretching along the soil core. This is in line with the results of Raper and Erbach (1985), who compared BD values determined with pushed and augered soil samplers on a silty clay loam and found larger differences at the 0-to 20- than the 20- to 40-cm depth.

The depth increments with compaction or stretching differed among the sites and probe types. Consequently, the site- and probe-type-specific correction to $BD_{opt\ ps}$ was the best form of correction. The variation in correction techniques with sites was particularly important. However, it is not viable to calibrate correction functions regarding all depth increments at each study site and sampling date. Therefore, if BD variation with soil depth matters, we suggest applying a probe-type-specific correction ($BD_{profile}$) as a compromise that substantially reduced the random error but can be applied to all soil types. The additional error reduction by applying the correction for $BD_{opt\ site}$ was mostly smaller. Alternative correction techniques could be based on visual detection of those core sections where compaction or stretching occurred and correcting those sections only (Walter et al., 2015). However, this requires well-trained and experienced staff. For the calculation of SOC stocks or nutrient stocks of the whole soil profile, no correction techniques are required because the integration of the whole core is more accurate.

Comparison of Different Methods for Bulk Density Estimation

The accuracy and precision of BD by careful measurements clearly outperformed that of BD modeling using pedotransfer functions. Soil BD is generally most accurately and precisely determined by using sample rings at a soil profile wall. However, in this study, where BD determination was extensively tested at sites without coarse material, the determination of BD with driving hammer probes was much more accurate than in previous studies. For example, in their comparison of driving hammer probes and the carving method, Parfitt et al. (2010) found about 5% difference in BD and thus an average systematic error of 0.075 g cm^{-3} for a BD of 1.5 g cm^{-3}. In contrast, the difference between driving-hammer-derived $BD_{profile}$ and BD_{REF} was, on average, only 2% in our study. If BD is estimated using pedotransfer functions, the systematic error is even larger, as demonstrated by a study evaluating 12 pedotransfer functions for forest soils in Flanders (De Vos et al., 2005), where the median MPE of the pedotransfer functions was −0.23 g cm^{-3}. In our study, the maximum mean MPE was 0.04 g cm^{-3} for the liner probe. An evaluation of 22 pedotransfer functions for Romanian soils (Vasiliniuc and Patriche, 2015) also showed much higher systematic errors (MPE ranging from −0.25 to 0.26 g cm^{-3}). Regarding precision, the sample rings in our study performed much better (mean SDPE 0.08–0.10 g cm^{-3}) and soil probes slightly better (mean SDPE 0.13–0.19 g cm^{-3}) than the pedotransfer-function-derived BD (SDPE 0.14–0.36 g cm^{-3} or 0.16–0.22 g cm^{-3} for recalibrated models according to De Vos et al. [2005], and 0.15–0.28 g cm^{-3} according to Vasiliniuc and Patriche [2015]).

Sources of Uncertainty in Bulk Density Quantification

There are uncertainties and sources of errors in BD determination that are independent of the method used, e.g., mistakes or inaccurate field documentation. Accurate measurement of the hole depth with probe sampling is particularly critical—e.g., if 99 cm instead of 100 cm was measured, BD values would be 1% higher. The shallower the soil sampling depth, the more crucial the exact recording of sampled soil depths. Imprecise definition of the mineral soil surface and the position of cutting at defined depth increments are additional sources of error (Gifford and Roderick, 2003). Lack of data on the length of the soil core and the depth of the soil hole can lead to substantial errors in BD determination with driving hammer probes because no corrections for compaction and stretching can be applied.

The large number of samples taken for this systematic method comparison involved many technicians sampling the soil. Different people sampling is a potential source of error according to Kulmatiski and Beard (2004), who found significant sampler effects on BD determination. Nevertheless, we did not detect any systematic and clearly attributable person effect, although some methods were applied by several technicians at the same site or by different technicians in the second sampling campaign at the sand site.

Additional sources of error, like those arising on gravelly or stony soils (Throop et al., 2012) or cracking soils (Bauer et al., 2014), were not considered in this study. However, we did apply the methods at a peat site because, due to high C concentrations and low BDs, small errors in BD determination particularly affect SOC stock calculations here. Our results show that BD determination remains a challenge for organic soils but, except for the window probe, the driving hammer probe could be used to determine SOC stocks as accurately as REF sample rings. The repeated sampling at the sand site showed that the reproducibility of BD determination by REF and BIG sample rings is very good but is less accurate and precise with probes, although differences were not systematic and not attributable to environmental conditions or sampler effects. In summary, all ring methods and the sheath probe can accurately and precisely determine BD in all soil types and the window probe in mineral soils. The liner probe proved too inaccurate for general application in soil inventories used to determine SOC stocks. The sheath probe is probably the most effective of the methods tested here for large-scale soil inventories because, in addition to good overall accuracy, the storage of the soil core in foil ensures convenient and rapid sampling and thus lower labor costs. However, the initial investment costs for driving hammer probes are higher than for sample rings.

Soil BD influences SOC stocks linearly because the stocks are calculated by multiplying the SOC concentration by the BD. Thus, the systemic error in BD with the sheath probe sampling would cause a systemic error in estimated SOC stocks of between 0.3 and 8 Mg C ha^{-1} for the mineral soils. This is equivalent to the range of SOC stock changes within 10 yr observed in repeated regional-scale inventories (Schrumpf et al., 2011). Thus, a change in the methods used for BD quantification between repeated inventories could completely obscure SOC stock changes. In peat soils, the BD-derived systematic error in SOC stocks would be around 4%, which is equivalent to >50 Mg SOC ha^{-1}. These results illustrate the influence of equipment, sampling method, and correction techniques on BD and emphasize the need for thorough BD determination to obtain accurate and precise SOC stock estimates.

Considering the general uncertainties in soil sampling due to its spatial heterogeneity (Goidts et al., 2009), the probe-based methods offer the considerable practical advantage of fast and easy deployment, allowing a larger sample size compared with soil-pit-based methods with sample rings. Nevertheless, sample rings have unbeaten accuracy, precision, and reproducibility. Effective sampling would best combine the advantages of sample rings and probes, e.g., by using sample rings in a soil pit for basic BD determination and additional probe samples to account for spatial heterogeneity.

CONCLUSIONS

All methods tested here determined BD with low systematic error (profile mean <0.1 g cm^{-3} or <0.4 g cm^{-3} for each depth increment). The random error of the probe samples was, on average, 50% higher than that of the ring samples if the cores taken with the probes were adequately corrected for compaction or stretching. Our results suggest that driving hammer probes can be used to determine soil BD for total SOC stock calculations in all common soil textures, including peat soils, but provide less accuracy in SOC depth distribution than sample rings. Sample rings performed most robustly and can be regarded as the method of choice for shallow soil sampling, for small depth increments, and for repeated soil sampling. Sample probes are most suitable for deep sampling and as an effective tool to capture spatial heterogeneity. The sheath probe type had the highest accuracy of all driving hammer probes tested and is recommended for soil inventories. It is also the only probe recommended for peat soils. Probes offer the possibility to determine BD without time-consuming digging of soil pits and could help to decrease the uncertainties in SOC stock inventories that often lack BD measurements. However, in specific soil types and depth increments, the random and systematic errors of BD determined with probes can be large.

Bulk density is probably among the most underestimated soil parameter when it comes to determination in the field. In many studies, information on either the method or the number of replicates used for BD determination is lacking, or BD is not measured and reported at all. Missing BD values impose a large uncertainty error on estimates of SOC stocks and SOC stock changes, which can only be accurately detected based on equivalent soil mass. We detected statistically significant differences between methods of BD determination, but the differences were still small and it has to be emphasized that all measurement methods tested produced far better BD values than those derived from pedotransfer functions.

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