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# Loss-on-ignition for scalable soil carbon monitoring: separating useful method transfer equations from misleading conversion factors

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**Abstract.** A central challenge for monitoring soil organic carbon (SOC) to support soil health assessment, national inventories, and carbon markets is the cost of sampling required to detect significant change over time. Direct measurement by dry combustion (DC) is relatively expensive and can limit sampling intensity. Loss-on-ignition (LOI) offers a low-cost alternative, but confusion persists regarding the sources of measurement error in LOI-derived SOC estimates across diverse soils. This study evaluated three LOI protocols that are in regular use today for measuring SOM and predicting SOC as measured by dry combustion (DC): ignition at 360 °C for 2 h (LOI360), 400 °C for 16 h (LOI400), and 550 °C for 3 h (LOI550), across 423 soil samples from contrasting landform regions, horizons, textures, and inorganic carbon contents. Method transfer equations were developed to relate soil organic matter (SOM) measured by LOI to SOC measured by DC. Those models were evaluated across multiple aspects of performance using measures of precision ($R^2$), accuracy (RMSE, MAE, and MedAE), and overall agreement (concordance correlation coefficient). The use of regression to relate method results, rather than estimating the SOC:SOM conversion ratio, is emphasized. The repeatability of the methods was also assessed by performing multiple measurements and calculating the coefficient of variation. LOI360 and LOI400 produced comparable and relatively strong relationships with DC-SOC, whereas LOI550 exhibited substantially lower precision and accuracy because of additional mineral mass losses during ignition. Only minor improvements over generalized models were achieved by localizing equations based on landform region or soil horizon, which suggests that broadly applicable transfer functions are sufficient for most applications. While DC provided the highest repeatability (CV = 2.60%), LOI400 demonstrated acceptable repeatability (CV = 5.3%) and accuracy (RMSE = 0.63 SOC%). LOI400 was also favourable because its method transfer equation had an intercept that was close to zero. These results demonstrate that LOI, when using an optimized and standardized protocol, is a practical and scientifically defensible approach for large-scale SOC monitoring. Although LOI measurements have more measurement error than DC analyses, their substantially lower cost enables much higher sampling densities, improving the characterization of spatial variability and increasing statistical power to detect SOC change. Additionally, LOI400 can serve as a preliminary screening tool to identify zones of contrasting SOC and guide targeted DC analyses, while also supporting the development of large ground-based SOC datasets for remote sensing and machine learning approaches to digital SOC mapping and monitoring.

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## 1 Introduction

Soil organic carbon (SOC) measurements are critical for soil health assessments, greenhouse gas inventories, monitoring, reporting, and verification frameworks, and carbon payment schemes (IPCC, 2006; FAO, 2020). In support of those activities, analytical uncertainty is not merely a laboratory issue: it affects whether spatial patterns are correctly characterized and whether temporal changes are detectable. Because soil organic matter (SOM) is central to soil structure, nutrient cycling, water retention, and the terrestrial carbon cycle, reliable data on both SOM and SOC are essential for agronomic interpretation and climate-related decision-making (Lal, 2004; Jackson et al., 2017).

Detecting changes in SOC is difficult due to measurement error and spatial variability, and because changes are often small relative to existing SOC stocks. Statistically defensible detection may require long monitoring intervals and a large number of samples (Smith, 2004; Ellert et al., 2008). These conditions highlight the importance of consistent and low-cost measurement methods. High-temperature dry combustion (DC) is widely accepted as the highest standard for measuring SOC; after measuring inorganic carbon, it can be used to predict SOM. Conversely, loss-on-ignition (LOI) remains the most widely used method (Hoogsteen et al., 2015) due to its lower cost and greater accessibility of equipment. LOI measures SOM, which is of high interest for agricultural productivity, and can be used to predict SOC.

Despite its enduring widespread use as a low-cost assessment of soil fertility and health, LOI is perceived in the scientific community as insufficient for change detection. However, it isn't clear whether this perception is based on concerns about LOI's accuracy, precision, repeatability, or a combination of these. Part of the uncertainty appears to stem from confusion over the meaning of those terms and their impact on statistical significance for change detection. Another major problem for LOI is that in practice, it is not a single method. A variety of protocols are used, based on assumptions and strategies that may not apply universally or be practical for field sampling with limited data. These protocols encompass the laboratory treatments and equations used to convert SOM to SOC, or vice versa.

For more than 135 years, the "van Bemmelen factor" has served as the standard for converting SOM measurements to SOC, based on the assumption that SOM contains, on average, 58% SOC (van Bemmelen, 1890). However, a comprehensive review of literature spanning the 1820s to the 2000s led Pribyl (2010) to conclude that this factor is overestimated. An empirical median SOC content of 53% was reported, while the theoretical estimates had a mean of 50%. Although understanding the proportion of SOC within SOM is valuable, applying this proportion directly as a conversion factor is not equivalent to developing an equation that translates results between different analytical methods. A straightforward way to account for methodological differences is to include a y-intercept in a regression-based model for relating the two measurement methods. This paper proposes using method transfer equations derived from linear regression, offering a more flexible alternative to fixed conversion factors, which are limited in their ability to account for non-SOM mass losses during LOI.

High-temperature dry combustion (DC) is the standard method for direct SOC (DC-SOC) determination due to its high analytical precision (IPCC, 2000). Moreover, depending on the instrument used, nitrogen (N) and sulfur (S) can

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be measured simultaneously, and DC may be integrated with a mass spectrometer for stable isotope analysis (Chatterjee et al., 2009). However, DC has some disadvantages. For accurate C and N determination, the sample must be finely ground and well homogenized to ensure complete combustion and representative results. While grinding to ≤2 mm is standard for most other chemical soil analyses, finer grinding may be required for DC to minimize variability caused by localized organic matter concentrations or incomplete oxidation (Bertsch and Ostinelli, 2019). A more significant issue, for both cost and accuracy of the analysis, is that pretreatment is usually required for samples containing inorganic carbon (IC). Other drawbacks of this method include equipment costs ranging from $40,000 to $50,000 and operational costs, including high electricity consumption and the use of high-purity gases (Chatterjee et al., 2009; Johns et al., 2015). These constraints limit throughput, which is problematic when SOC monitoring requires dense sampling across heterogeneous landscapes.

Loss on ignition (LOI) is a commonly employed technique for determining SOM concentrations (LOI-SOM; Hoogsteen et al., 2015). For example, LOI-SOM is already included in the standard soil fertility analysis of many commercial labs. This approach offers a simple and inexpensive alternative because it estimates SOM from the mass lost after heating soil in a muffle furnace, and it has been widely used to estimate SOC using a standard conversion such as the van Bemmelen factor (Schulte and Hopkins, 1996). De Vos et al. (2005) compared LOI using 550 °C for 3 hours to DC, concluding that DC operating costs were 12 times more expensive and the process took 2.3 times longer to complete. Like DC, LOI has the advantage of not generating hazardous waste, whereas older methods, such as the Walkley-Black wet-combustion method, produce chromium (Cr) waste as a byproduct (Wang et al., 2012). Nonetheless, a universally standardized protocol for LOI has been elusive due to many factors that may affect the measurement, including soil mineralogy, calcium carbonate content, heating temperature, and time at that temperature.

Although LOI methods have been published in laboratory methods standards manuals (e.g., USDA-NRCS, 2022), the literature lacks consensus on the optimal temperature and ignition duration for measuring SOM using the LOI method. Temperatures ranging from 300 to 850 °C and durations from 2 to 28 hours have been suggested. Low temperatures of ignition over long durations (e.g., 375 °C for 16 hours) have been reported to be more accurate for measuring SOM than high temperatures over short periods (e.g., 850 °C for 0.5 hours) due to incomplete oxidation of C for short time periods or due to mass loss from minerals at high temperatures (Ball, 1964).

Many variations of LOI methods have been developed and reported. For example, using 430 °C for 24 hours, Davies (1974) observed no overestimation of organic matter concentration due to the presence of calcium carbonate. He recommended temperatures between 375 and 450 °C for both calcareous and non-calcareous soils, but not for soils containing gibbsite, for which the temperature should be reduced to 300 °C. On the other hand, Hoogsteen et al. (2015) concluded that a temperature of at least 550 °C for 3 hours should be used to completely oxidize the organic material. However, they also recommended using a clay correction factor to offset overestimation caused by mineral mass loss.

Wang et al. (2012) also reported that the optimal heating temperature and time depend on the mineralogy of the sediment source. For example, CO2 can be volatilized from siderite, dolomite, and rhodochrosite at 425 °C (Duval, 1963; Weliky et al., 1983). Goethite (FeOOH) can be dehydroxylated between 280 and 400 °C (Barshad, 1965), while

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gibbsite (Al(OH)3) loses an important quantity of mass around 300 °C (Davies, 1974). Similarly, Christensen and Malmros (1982) reported that the water loss from Fe or Mn oxyhydroxides could significantly affect LOI results.

Among the layer silicate clay minerals, kaolinite loses about 14% of its mass by dehydroxylation at temperatures between 400 and 600 °C, with the greatest loss around 500 °C (Tan et al., 1986; Karathanasis and Harris, 1994). Dehydration by evaporation of interlayer water in vermiculite and smectite occurs at temperatures greater than 250 °C. The 2:1 layer silicate minerals (e.g., illite, chlorite, vermiculite, and smectite) lose about 5% of their mass by dehydroxylation between 500 and 900 °C (Barshad, 1965; Tan et al., 1986; Jackson, 2005; Karathanasis and Harris, 1994). For non-layer silicate minerals, mass losses of 15.84%, 2.45%, and 2.02% have been reported when using LOI at 550 °C for gypsum, goethite, and dolomite, respectively (Sun et al., 2009). These authors suggested a correction for mass loss due to dehydration and dehydroxylation when working with subsurface samples - where organic C concentration is likely to be low - or soils containing appreciable quantities of goethite, kaolinite, or vermiculite. However, accurate correction requires mineral abundance data, which are typically more expensive to obtain on a per-sample basis than conducting DC analyses. Table 1 summarizes the known temperature thresholds for mass loss from soil components other than organic matter.

**Table 1: Summary of minerals observed to lose mass in the range of temperatures used in LOI methods (Barshad, 1965; Tan et al., 1986; Karathanasis and Harris, 1994; Jackson, 2005; Kohobhange et al., 2019).**

| Mineral Type | Name | Key Temperature Threshold |
|---|---|---|
| 1:1 layer silicate | Kaolinite | Dehydroxylation 400-600 °C (~14%) |
| 2:1 layer silicate | Chlorite | Dehydroxylation 500 - 900 °C (~5%) |
| 2:1 layer silicate | Illite | Dehydroxylation 500 - 900 °C (~5%) |
| 2:1 layer silicate | Smectite | Dehydration at 250 °C;<br>Dehydroxylation 500 - 900 °C (~5%) |
| 2:1 layer silicate | Vermiculite | Dehydration at 250 °C;<br>Dehydroxylation 500 - 900 °C (~5%) |
| Carbonate | Calcite | $CO_2$ volatilized at 675 °C |
| Carbonate | Dolomite | $CO_2$ volatilized at 425 °C;<br>Mass loss at 550 °C (~2%) |
| Carbonate | Rhodochrosite | $CO_2$ volatilized at 425 °C |
| Carbonate | Siderite | $CO_2$ volatilized at 425 °C |
| Sulfate | Gypsum | Mass loss at 550 °C (~16%) |
| Aluminum hydroxide | Gibbsite | Dehydroxylation at 250-300 °C (31%) |
| Iron Oxyhydroxide | Goethite | Dehydroxylation 280-400 °C (10-11%) |
| Tectosilicate | Feldspar | None |
| Tectosilicate | Quartz | None |
| Phyllosilicates | Mica | None |

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The approach for converting SOM to SOC depends on the goal. The conversion factors of van Bemmelen (1890) and Pribyl (2010) assume that the conversion should be based on the proportion of organic carbon contained in organic matter. However, other factors – such as mass loss from clay minerals – introduce additional variables into the relationship between laboratory methods. This additional complication suggests that a conversion equation, rather than a simple proportional composition of C in SOM (even if known for each soil sample), should be applied to more accurately relate results from one method to another. Konen et al. (2002) used linear regression equations to convert LOI-SOM to DC-SOC. The high coefficient of determination (R2) results for their linear equations supported the inclusion of an intercept to improve conversion precision. In addition, a Tukey's test for significant differences between the slopes and intercepts of equations for contrasting Major Land Resource Areas (MLRAs) suggested that the predictive equations should be locally customized. However, a generalized conversion equation encompassing the pooled data was not tested against the localized conversion equations. Considering that the resources to measure both LOI-SOM and DC-SOC to create customized equations for every locality might often be impractical, determining the precision of a generalized equation would help assess the suitability of LOI to detect change in SOC over time and space.

Further complicating the use and comparison of LOI measurements is the diversity of method parameters employed, which has contributed to LOI being perceived as a method with low precision for predicting SOC. Various factors may influence LOI-SOM results under different methods; therefore, measurements of SOM with varying temperatures and durations are unlikely to be directly comparable. This study seeks to disambiguate the performance of LOI by testing three protocols: heating for two hours at 360 °C (Schulte and Hopkins, 1996), heating for sixteen hours at 400 °C (USDA, 2022), and heating for three hours at 550 °C (Hoogsteen et al., 2015). Comparison of these methods using a wide range of soil samples enabled us to: 1) assess the relationships between DC and the different LOI protocols; 2) evaluate the accuracy, precision, and repeatability of method transfer equations; 3) determine how inorganic carbon, landform region, horizons, and their different combinations affect LOI-SOC predictions; and 4) assess whether localized method transfer equations provide meaningful improvements over generalized method transfer equations. By separating the method transfer equations' performance from assumptions about SOM-to-SOC conversion factors, this study addresses a practical question for SOC monitoring: under what conditions can LOI be used as a scalable and defensible alternative to dry combustion?

## 2 Materials and Methods

### 2.1 Study area and soil samples

A collection of 424 samples was assembled to represent a diversity of soil conditions from four distinct landform regions across Iowa, USA (Figure 1). These regions encompass a diverse range of glacial landscapes, characterized by contrasts in parent material, mineralogy, and topography (Table 2). Land use in these areas is predominantly corn and soybean production. One extreme outlier with consistently high values across all LOI treatments was excluded to avoid biasing model evaluation, resulting in a final dataset of 423 samples.

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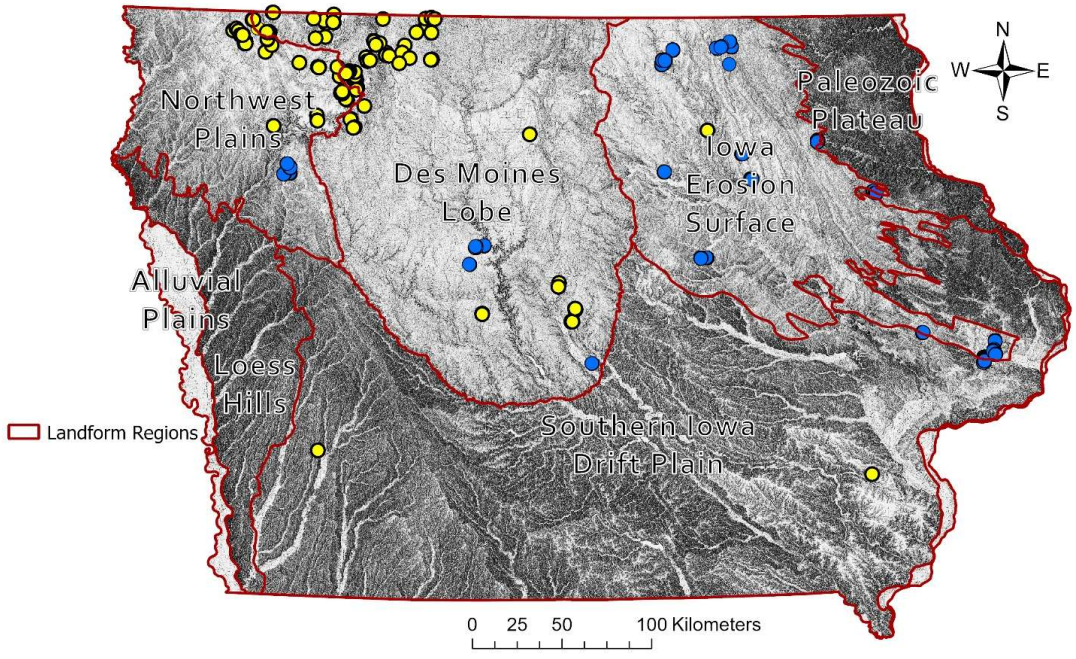


**Figure 1: Distribution of sample locations within differing landform regions, shown on an elevation hillshade to indicate relief (© U.S. Geological Survey, 2020). Blue locations were taken using a hand probe, while yellow locations were collected using a truck-mounted hydraulic soil probe (Giddings Machine Company, Windsor, CO).**

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**Table 2: Descriptions of soil landscapes where soil samples were collected and analyzed for this study. Note that all minerals exhibit spatial variability across landscapes and with depth. The presence of calcite in this study was identified in individual samples by testing soil pH.**

| Landform Region[a] | Dominant Parent Material | Clay mineralogy (Handy et al., 1955; Khan, 1991) | Landscape | Example Soil Series and Taxonomic Subgroup |
|---|---|---|---|---|
| DML | Clay loam, calcareous till | Calcite, goethite, smectite, and mica; Smaller amounts of kaolinite and hydroxyl-interlayered minerals | Closed basins with low relief | Clarion and Canisteo (Udolls – Aquolls) |
| IES | Silty clay loam, Peoria loess (variable thickness or absent) mixed with or over Pre-Illinoian till | Calcite and feldspar; some mica; variable illite and kaolinite | Broad dissected plains | Kenyon and Clyde (Udolls – Aquolls) |
| SIDP | Silty clay loam, Peoria loess (~3 m thick) over Pre-Illinoian till | Calcite and smectite; variable illite and kaolinite | Dissected hills and flats | Marshall and Tama (Udolls) |
| NWP | Silty clay loam, Peoria loess (~5 m thick) over Pre-Illinoian till | Calcite, feldspar, smectite, variable illite, and kaolinite | Gently rolling hills | Galva and Sac (Udolls) |

[a] DML = Des Moines Lobe; SIDP = Southern Iowa Drift Palin; IES = Iowa Erosion Surface; NWP = Northwest Plain.

All samples were analyzed for soil texture class and pH. All samples with a pH greater than seven were tested for the presence of inorganic carbon. All locations included surface horizon samples to a depth of 20 cm. Of those, 79% were collected using a truck-mounted hydraulic soil sampler (Giddings Machine Company, Windsor, CO) to a depth of 200 cm. Of the total samples, 50% corresponded to A, 35% to B, and 15% to C horizons. This distribution of horizons included a range of SOM concentrations and textural classes (Figure 2).

Particle size analysis (PSA) was conducted using laser diffraction with a Malvern Mastersizer 3000 and Hydro EV attachment. Samples were oven-dried at 65 °C for 24-48 hours, depending on initial moisture content and sample texture. After drying, the samples were ground, sieved to a 2-mm mesh size, and homogenized by passing them through a Haver RT 12.5-mm splitter three times. The Malvern Mastersizer 3000 passes laser pulses through a water suspension of soil particles. The laser's interaction with the particles alters the angle of the light, which is measured by the instrument. Mie diffraction theory determines the equivalent spherical diameter of the soil particles (Malvern Instruments, 2017). Pre-treatment of samples for PSA and quality control protocols was conducted as described by Miller and Schaetzl (2012).

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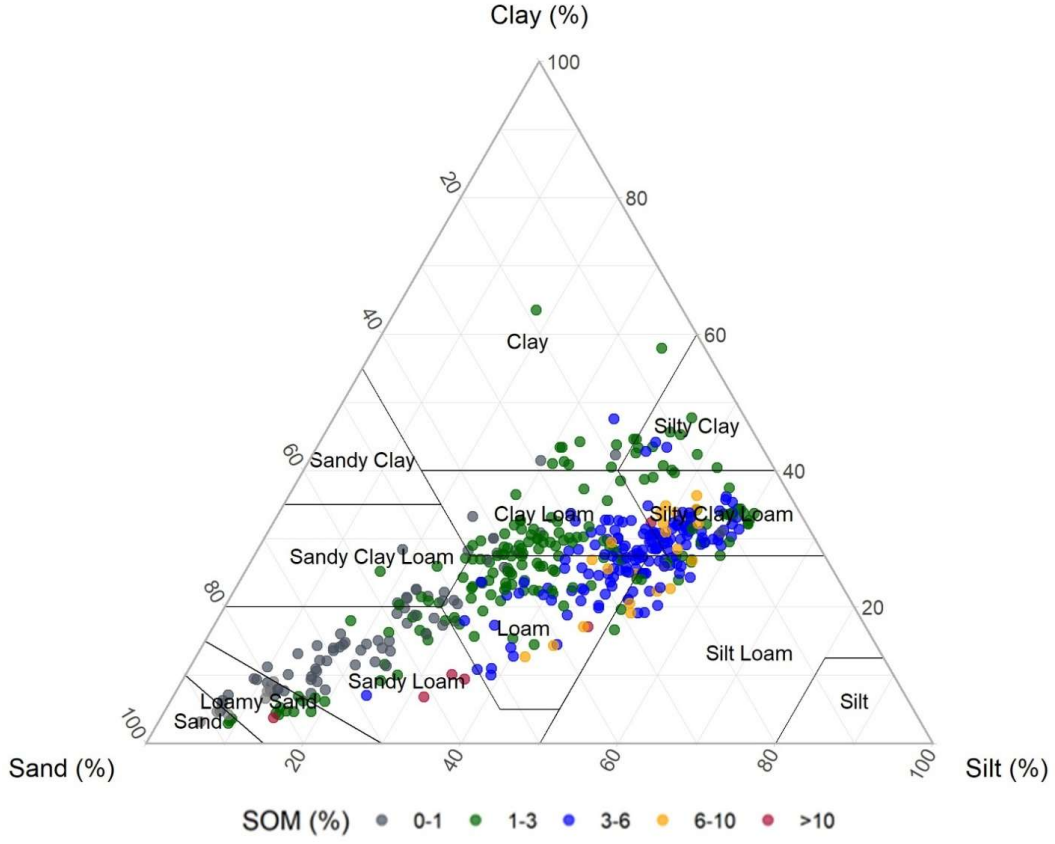


205 **Figure 2: Distribution of soil texture class and SOM (%) concentrations of soil samples evaluated in this study. Different colors represent the range of SOM (%) concentrations.**

### 2.2. Lab analysis

#### 2.2.1. Dry combustion

210 DC-SOC was treated as the standard reference method for accurately measuring SOC, which was calculated as the difference between total carbon and IC. Solid samples ground to <0.25 mm were dried at 105 °C before being weighed for analysis. Then, 30 mg of these samples were flash-combusted in an oxidative environment to determine total carbon, producing carbon dioxide from the carbon compounds in the samples (Schepers et al., 1989). Carbon dioxide ($CO_2$) was quantified using a thermal conductivity detector (FlashSmart Elemental Analyzer, Thermo Fisher
215 Scientific, Waltham, MA, USA). The instrument was calibrated before each analytical sequence using five atropine standards to generate the first-order calibration curve. Calibration was accepted with an $R^2 > 0.999$. Three reference soil standards were analyzed following calibration. To proceed with sample analysis, these samples were required to agree within ±3 standard deviations of the reference values. For quality control, one duplicate experimental sample

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and one laboratory control sample were included for every ten experimental samples. Duplicate sample data were acceptable if within ±10% relative difference (RPD).

Inorganic carbon was analyzed using a pressure calcimeter to measure the pressure increase from $CO_2$ gas evolution after adding acid to soil samples containing carbonate minerals. Eleven soil standards amended with calcium carbonate, ranging from 0-2.4% IC, were used to generate a daily, linear calibration curve with an $R^2 > 0.995$. Two check samples of reference calcareous soil standards were run with each set of calcareous samples and were required to agree within ±10% of the known reference value to be acceptable (Sherrod et al., 2002).

Quality control for IC determination included one duplicate sample for every ten experimental samples. Three laboratory control samples of reference calcareous soil standards and one laboratory blank were run with every set. Acceptable laboratory control data had a recovery within ±3 SD of the expected mean IC percentage, and the laboratory blank was accepted if the area counts were less than those of the lowest standard. If the blank and the reference sample analyses fell outside the expected ranges, the entire run was repeated.

#### 2.2.2. Loss on ignition

All LOI-SOM protocols were run using a muffle furnace (Lindberg 51000 Series, model 51442). Sixty-two Fisher brand FB-965-E crucibles were filled with 8 g of < 2-mm oven-dry soil per run. Before furnace ignition, samples were oven-dried at 105 °C overnight, cooled in a desiccator, and weighed to 0.01 g. Three protocols were applied to assess the effects of ignition duration and temperature. For protocols LOI360, LOI400, and LOI550, the ignition temperature and duration were 2 hours at 360 °C (Schulte and Hopkins, 1996), 16 hours at 400 °C (USDA, 2022), and 3 hours at 550 °C (Hoogsteen et al., 2015), respectively. LOI-SOM calculations were performed using equation (1), where ODSM and ISM represent the oven-dry soil mass and ignited soil mass, respectively.

$$LOI\ (g\ Kg^{-1}) = \frac{(ODSM - ISM)}{ODSM} \times 1000 \quad (1)$$

### 2.3. Statistical analysis

#### 2.3.1. Defining precision, accuracy, and repeatability

The terms precision, accuracy, and repeatability, among others, are often conflated or have different meanings in various contexts. This research was a comprehensive exercise across all three aspects because the process included evaluating the method conversion equations' ability to represent patterns in the data (goodness-of-fit), how closely those method conversion models matched the target variable, and the variability due to measurement error. For clarity in this research, the terms 'precision' and 'accuracy' are reserved for describing the performance of the method transfer models. High precision indicates relationships between the regressor and response variables are consistent (low dispersion), while high accuracy indicates that predictions are close to the reference values (unbiased) (Figure 3). Repeatability in this context is equivalent to analytical/measurement precision, where the same operator and instrument are used to measure the variability observed when the same sample is analyzed multiple times.

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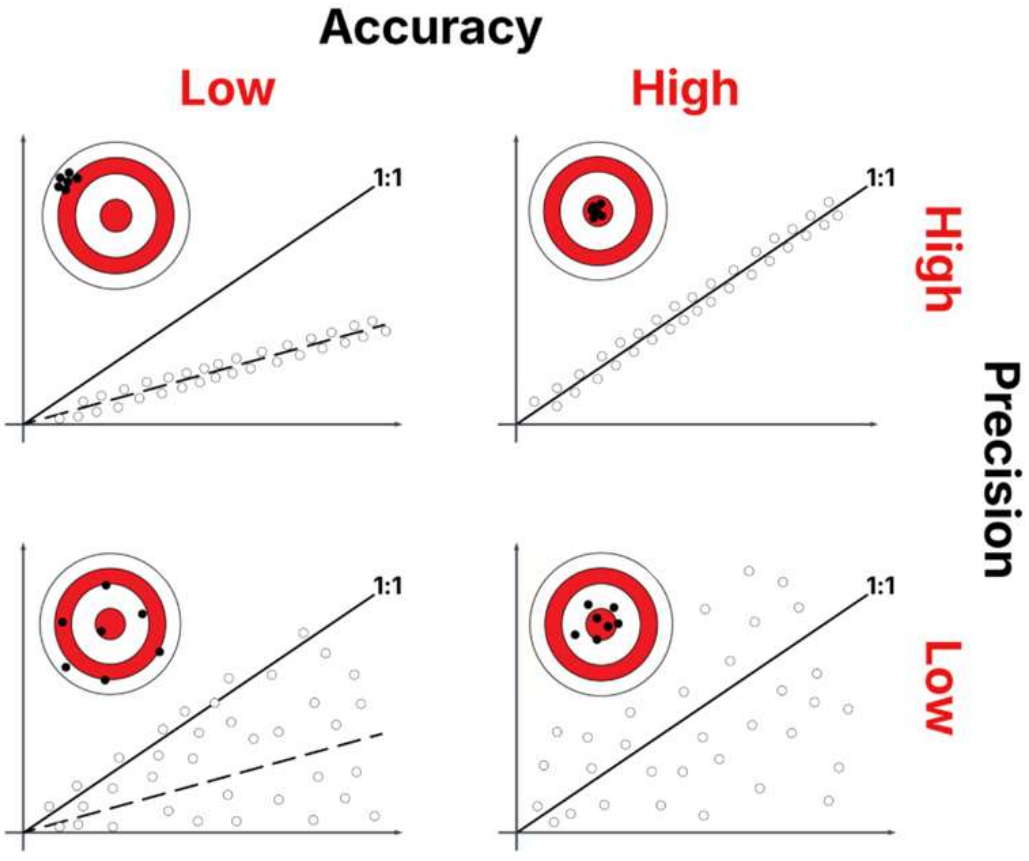


**Figure 3: Conceptual illustrations of accuracy and precision, including the scatterplot scenario where the target is also a variable. The 1:1 line represents the perfect match between predicted values and observed values. The upper right panel demonstrates high accuracy and precision, with predictions closely matching observations and minimal scatter. The upper-left panel shows low accuracy but high precision, where predictions are consistent but systematically biased. The lower-right panel shows high accuracy but low precision, with predictions centered around the 1:1 line and exhibiting substantial scatter. The lower left panel shows low accuracy and precision, with predictions that are both biased and highly variable.**

### 2.3.2. Precision

Traditionally, the strength of equations describing observed relationships between SOM and SOC has been assessed with $R^2$. However, our analysis requires comparison of multiple sample sets with different variances, and $R^2$ is explicitly based on variance. In the $R^2$ equation (2), the residuals from the mean model are compared to the residuals of the prediction model being tested (3).

$$R^2 = 1 - \frac{SS_{residual}}{SS_{total}} \qquad (2)$$

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where $SS_{total}$ is the total sum of squares, or the total variance (3), and $SS_{residual}$ is the residual sum of squares, or the unexplained variance (4).

$$SS_{total} = \sum (y_i - \bar{y}_i)^2 \quad (3)$$

$$SS_{residual} = \sum (y_i - \hat{y}_i)^2 \quad (4)$$

where $y_i$ are the observed values, $\hat{y}_i$ are the predicted values, and $\bar{y}_i$ is the sample mean.

$R^2$ values for sample sets with low variance tend to be low because there is little variance to explain. Conversely, when there is more variance to explain, $R^2$ values will tend to be higher. Given the nature of $R^2$, smaller sample sizes are more likely to yield inflated $R^2$ values. Therefore, $R^2$ is used to assess the strength of the relationship between DC-SOC and LOI-SOM and to evaluate the precision of individual method transfer equations. However, when determining the optimal method for translating LOI-SOM measurements to a DC-SOC standard, accuracy is the key consideration.

**2.3.3. Accuracy**

The ideal goal of a conversion equation is to yield results that accurately match the values obtained by the reference method. To assess the performance of a conversion model, predictions (i.e., LOI-SOC) should be compared with observed (i.e., DC-SOC) values and evaluated using metrics of deviation from the target values. Also, given the limitations of metrics that depend heavily on variance, assessing model performance solely by $R^2$ may be misleading. Moreover, a single metric captures only one dimension of a model's errors, highlighting a specific facet of the interaction between the model and the data (Chai and Draxler, 2014). As measures of accuracy, the two most widely used error metrics were adopted: root mean square error (RMSE) (5) and mean absolute error (MAE) (6) (Botchkarev, 2019).

$$RMSE = \sqrt{\frac{SS_{residual}}{n}} \quad (5)$$

$$MAE = \frac{1}{n}\sum_{i=1}^{n} |SS_{residual}| \quad (6)$$

According to Chai and Draxler (2014), RMSE is particularly appropriate when residuals are expected to be unbiased and normally distributed, as it disproportionately penalizes large errors and is sensitive to variance. This sensitivity makes RMSE effective for identifying models that produce occasional large deviations, especially when such deviations are relevant to the model's use case. In contrast, MAE treats all errors equally, providing a straightforward and robust measure to summarize error magnitude. Willmott and Matsuura (2005) advocate for MAE when the goal is to assess general predictive accuracy without inflating the influence of outliers or assuming normality. MAE is more interpretable in contexts where the frequency rather than the magnitude of large deviations is more relevant to model performance.

Because these accuracy metrics are influenced by the shape of the residual distribution, the skewness and kurtosis of the errors were also calculated in R using the moments package (Komsta and Novomestky, 2022). Skewness measures

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the symmetry of the residuals; values near zero indicate a balanced distribution. A positive skew suggests underestimation (long right tail), while a negative skew indicates overestimation (long left tail). On the other hand, kurtosis measures the 'heaviness' of the residual distribution's tails relative to a normal distribution. High kurtosis values indicate a concentration of small errors, accompanied by occasional large deviations, which can disproportionately affect the RMSE. To further enhance the robustness of the analysis, we included the median absolute error (MedAE). Unlike RMSE and MAE, MedAE does not rely on assumptions about residual distribution and is more resistant to the influence of outliers, providing a reliable measure of the typical magnitude of errors in non-normal contexts.

$$MedianAE = median\ |SS_{residual}| \quad (7)$$

Complementing the use of RMSE, p-values from the Shapiro–Wilk test were calculated to assess residual normality. For MAE, we applied the Kolmogorov–Smirnov test against a uniform distribution (KS-uniform) to determine whether residuals deviated from uniformity. Both tests were implemented in the base R stats package (R Development Core Team, 2025).

#### 2.3.4. Summarizing precision and accuracy

The concordance correlation coefficient (CCC) was used as the final summary of model performance for each sample-subset scenario. CCC was explicitly designed to assess the ability of one measurement method to reproduce the results from a 'gold-standard' method (Lin, 1989):

$$CCC = \frac{2\rho\,\sigma x\,\sigma y}{\sigma_x^2 + \sigma_y^2 + \left(\mu_x + \mu_y\right)^2} \quad (8)$$

where $\rho$ is the Pearson correlation coefficient between the two measurements, $\sigma_x^2$ and $\sigma_y^2$ are the corresponding variances, $\mu_x$and $\mu_y$ are the means of the two measurement methods. Accounting for both systematic bias and random variation simultaneously is useful in summary, but not necessarily diagnostic, because multiple factors could explain differences in CCC results (Wadoux and Minasny, 2024). Nonetheless, while not immune to differences in variance, the CCC is the most appropriate when the range and variance of the underlying real-world SOC concentrations are comparable.

The ability to predict DC-SOC from LOI measurements was tested under varying LOI treatment and soil conditions, arising from contrasting soil parent materials and horizons. While acknowledging the issues associated with comparing model performance statistics across different sample sets, we consider expected trends with increasing variance and examine the use-case impacts of the various components of accuracy and precision, as measured by the suite of statistics employed.

### 2.4. Experimental design

The relationship between DC and the different LOI protocols was quantified using linear least-squares regression. The precision of each LOI protocol was compared using the $R^2$ statistic. Then, the accuracy of the method transfer equations associated with each LOI protocol was evaluated. For each model, the intercepts of the linear regression

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equations were tested for statistical significance relative to zero. We chose a p-value less than 0.05 to indicate that the null hypothesis of the intercept equaling zero should be rejected. It is important to note that a positive intercept indicated incomplete removal of SOM, whereas a negative intercept indicated that processes such as dehydration, dehydroxylation, or carbonate removal likely occurred (Pribyl, 2010).

Separate method transfer equations were developed by stratifying the samples by IC content and landform region, and those models were compared to investigate how these two factors affected the precision and accuracy of SOC predictions. Evaluation of region and genetic horizon served as proxies for differences in clay mineralogy, where differences in the precision and accuracy of method transfer equations for the respective subsets were assumed to be driven by sample characteristics. Climate and vegetation were not considered sufficiently variable across the Iowa landform regions to be likely factors. Comparison between the performance of region-specific and generalized method transfer equations (i.e., based on the complete data set) served as a test of whether localized method transfer equations could compensate for confounding factors (e.g., non-SOM mass loss).

To compare the intrinsic measurement errors of the different methods (i.e., repeatability), we calculated the coefficient of variation (CV), defined as the ratio of the standard deviation to the mean of replicate measurements. For LOI, triplicate samples were analyzed by different operators to capture both analytical and operator-related variability. For DC measurements, CVs were derived from duplicate quality-control samples included in each analytical batch.

## 3 Results and discussion

### 3.1. Assessment of relationships between DC and different LOI protocols

#### 3.1.1. Which LOI protocol has the highest precision?

The regression analysis on all sample points demonstrated a reasonable relationship between LOI-SOM measurements and DC-SOC, with $R^2$ values of 0.67 and 0.65 when using LOI360 and LOI400, respectively. In contrast, when using LOI550, $R^2$ decreased to 0.47 (Figure 4). This decline in precision suggested that variability in mass loss with LOI550 may not be suitable for a generalized method transfer equation. The breakdown in the relationship between LOI-SOM at 550 °C and DC-SOC was most likely due to greater mass losses associated with dehydroxylation or $CO_2$ volatilization of minerals at the higher temperature. The smectite, vermiculate, chlorite, illite, and kaolinite content in the soil samples was susceptible to some mass loss at this higher temperature. Kaolinite could be especially impactful on the mass lost, with a mass loss of up to 14% (Table 1). Such impacts are difficult to predict quantitatively without prior quantification of those soil minerals.

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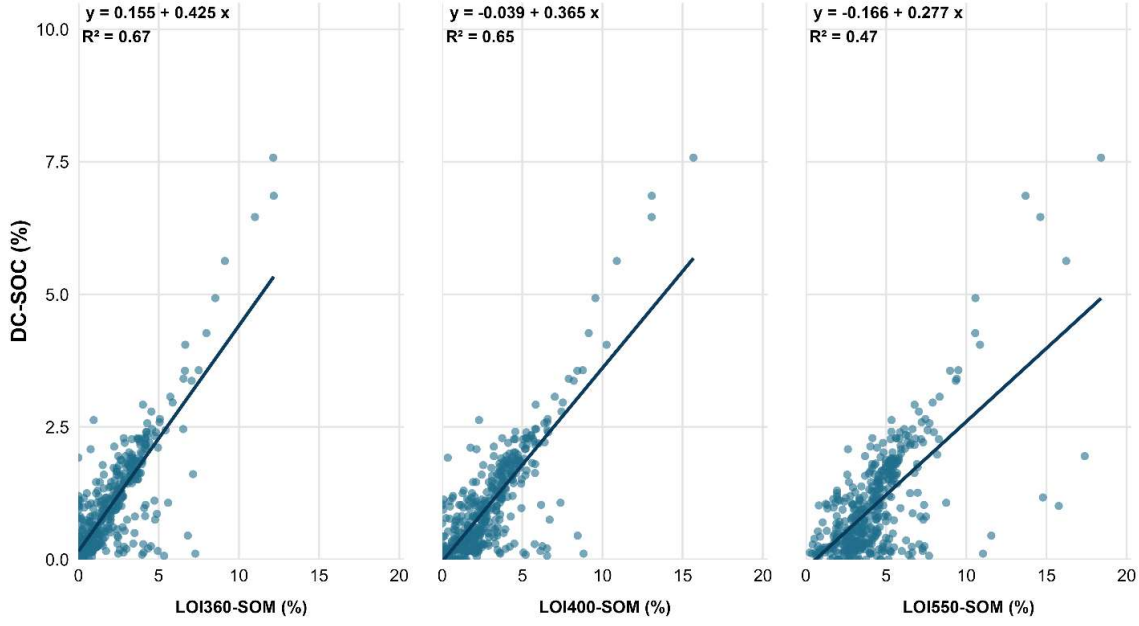


**Figure 4: Relationship between LOI-SOM (%) and DC-SOC (%) for the three LOI protocols tested for the whole data set. The solid blue line shows the fitted linear regression. LOI360 and LOI400 showed stronger relationships with DC-SOC than LOI550, indicating that higher ignition temperature reduced the consistency of the relationship between LOI-SOM and DC-SOC. Notice that the slope of the method transfer equations decreased as the LOI protocol temperature increased.**

### 3.1.2. Which method transfer equation provides the highest accuracy?

The lowest MAE and RMSE values were obtained for LOI360 and LOI400, indicating smaller mean and extreme deviations between predicted LOI-SOC and observed DC-SOC values. In contrast, LOI550 produced the largest residuals (Figure 5). The MedAE values were consistently smaller than RMSE and MAE, underscoring that most predictions were close to the observed values and that the larger deviations penalized by RMSE occurred only occasionally. The slight differences between RMSE, MAE, and MedAE for LOI360 and LOI400 suggest comparable predictive accuracy between these two protocols, whereas LOI550 displayed both larger typical and extreme errors (Figure 5).

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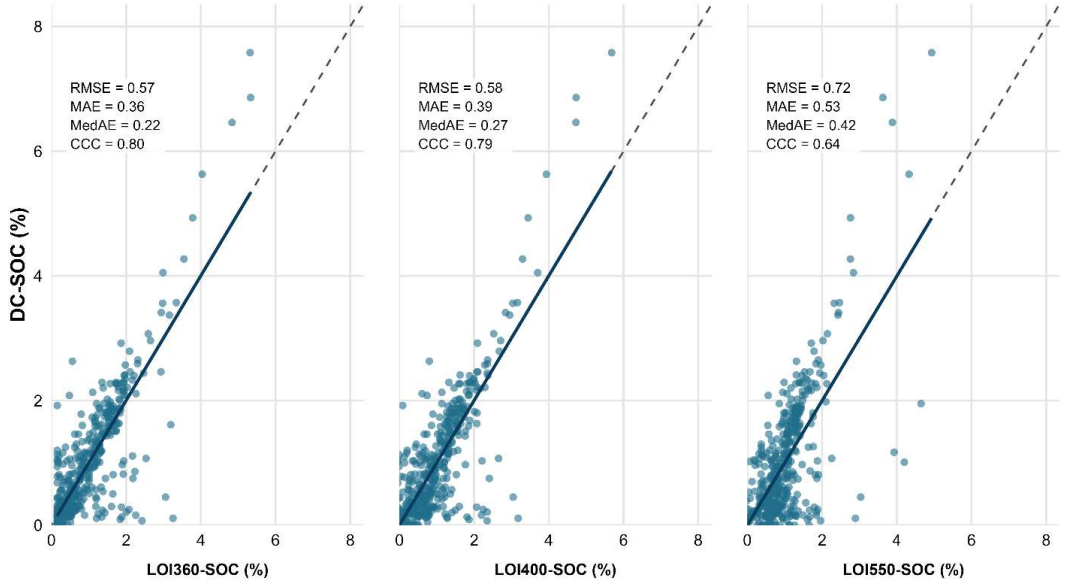


**Figure 5: Performance of conversion equations based on the whole data set for the three LOI protocols. The predicted LOI-SOC (%) is compared with the observed DC-SOC (%). Each panel shows the 1:1 reference (dashed line), where accurate predictions should align. RMSE, MAE, MedAE, and CCC are comparable for LOI360 and LOI400, while performance declines for LOI550.**

The residual diagnostics revealed substantial deviations from normality (Shapiro-Wilk $p < 0.001$), with heavy tails (kurtosis ranging from 6 to 8) and near-zero skewness for all LOI protocols (Supplemental Table S1). In the presence of non-Gaussian, heavy-tailed errors, MedAE provides more robust summaries of the errors. The slight differences in RMSE, MAE, and MedAE between LOI360 and LOI400 suggest comparable predictive accuracy, whereas LOI550 exhibits larger typical and extreme errors.

This behavior is consistent with additional mass loss due to dehydroxylation of minerals such as goethite, smectite, illite, and, particularly, kaolinite. Without knowledge of the concentrations of these potential additional sources of mass loss, the accuracy of the method transfer equations at higher ignition temperatures weakens. CCC, as a summary statistic for precision and accuracy, shows the same pattern, with moderately good agreement for LOI360 and LOI400 and a decline for LOI550 (Figure 5).

### 3.2. Examination of variability and sources of error

#### 3.2.1. Effect of excluding IC

Comparison of models for samples without IC to those for the entire data set showed simultaneous improvements in both precision and accuracy. For this subset, $R^2$ increased by 0.11 and 0.16 units over the full set regression for LOI360 and LOI400, respectively, despite the decrease in variance. At the same time, RMSE dropped by 0.13 (LOI360) and 0.17 (LOI400), MAE dropped by 0.06 (LOI360) and 0.09 (LOI400), while MedAE showed minor improvements for

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both LOI360 and LOI400 (Figure 6). Although none of the error statistics are large, the pattern of higher error for samples with IC suggests that either IC or something correlated with IC reduced the precision and accuracy of the method transfer equations.

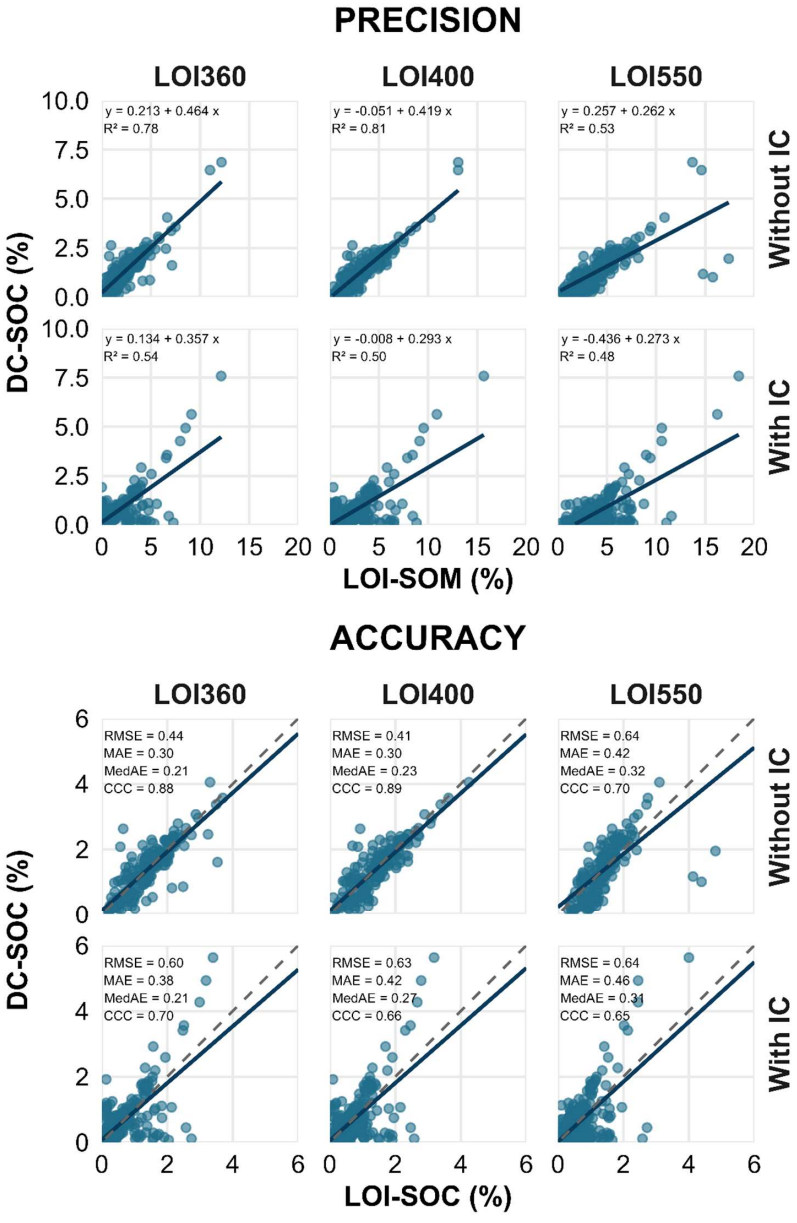


**Figure 6: Comparison of the precision of the relationship between DC-SOC (%) and LOI-SOM (%), and accuracy of the predicted LOI-SOC and observed DC-SOC for samples with IC, and without IC. Essentially, samples without IC are less noisy.**

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The most substantial effect of omitting samples with IC was on precision (Figure 6). The $R^2$ for LOI550 was relatively low with or without IC, but the difference in $R^2$ between with and without IC for LOI360 and LOI400 was large. LOI360 and LOI400 were highly precise ($R^2 > 0.78$) for samples without IC; however, the $R^2$ values dropped below 0.54 for these protocols on samples with IC. The SOC variance for samples with IC (0.80) is only slightly lower than that for samples without IC (0.88), making the difference in variance an unlikely explanation for the large reduction in $R^2$.

The exclusion of IC samples resulted in some improvement in the accuracy of the LOI-SOC predictions of DC-SOC (Figure 6), although not as pronounced as the difference in precision. This result suggests that the method transfer equations compensate well for the additional sources of mass loss. Residual diagnostics continued to show substantial deviations from normality in all cases (Shapiro–Wilk $p < 0.001$) and heavy-tailed residual distributions (kurtosis 5–9), with near-zero or mild skewness (Supplemental Table S1). However, excluding samples with IC reduced both kurtosis and error magnitudes. Nonetheless, the gain in accuracy was never more than 0.22% SOC in terms of RMSE, and even less for MAE and MedAE.

To understand the drivers of reduced model performance, it is essential to consider the known interactions between soil materials and temperature. None of the carbonate forms are known to volatilize $CO_2$ below 425 °C, making the presence of IC an unlikely direct cause of reduced precision in LOI360 and LOI400. However, given the observed relationship between IC and LOI's reduced performance in predicting SOC, IC indicates a likely lower precision and accuracy.

### 3.2.2. Effect of stratification by region

Comparing precision among regions and the data set as a whole becomes problematic because of the range of variances across the sample sets (Table 3). However, the decreasing variance trend does not fully account for the differences in $R^2$ across regions (Figure 7). The method transfer equations for DML and IES exhibited precision comparable to that of the overall data set. The $R^2$ for the SIDP was higher than expected, and for the NWP, lower than expected. For the SIDP, the method transfer equations appear to compensate well for a consistent excess of mass loss with increasing LOI temperature. The y-intercept decreases from -0.35 to -1.5 considerably between LOI360 and LOI550, but across all three methods, the $R^2$ remains high. For the NWP, the low $R^2$ values indicate low precision, likely driven by greater variability in soil components contributing to mass loss beyond SOM. Here, the higher variability in mass loss is more noticeable in the lower-variance case. These results suggest that localized method transfer equations can be beneficial when the additional sources of mass loss are consistent; however, they can be challenging when the causes of these additional mass losses are variable and unmeasured.

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**Table 3: LOI-SOC (%) descriptive statistics for all samples compared to the sample sets stratified by landform region. Although not the case for the combined data set, variance decreased as the quantity of samples decreased.**

| | All | DML | IES | NWP | SIDP |
|---|---|---|---|---|---|
| n | 423 | 168 | 134 | 92 | 29 |
| Mean | 1.02 | 0.97 | 1.27 | 0.81 | 0.71 |
| Variance | 0.96 | 1.4 | 0.71 | 0.6 | 0.35 |
| Min | 0.00 | 0.00 | 0.00 | 0.00 | 0.16 |
| Max | 7.58 | 7.58 | 4.93 | 3.56 | 2.08 |

Although the localized method transfer equations reduced error compared to the generalized method transfer equation, the magnitude of these improvements remained modest. The maximum reductions were 0.46%, 0.32%, and 0.24% SOM for RMSE, MAE, and MedAE, respectively. These values are all below 0.5 percentage units of SOC, which can be considered the approximate threshold at which differences begin to acquire practical significance. These reductions are relatively small in operational terms. Nevertheless, even minor improvements in accuracy may influence SOC detection over time, and the implications of these reduced errors for detecting SOC changes are examined further in Section 3.3.

While differences in variance make comparing precision between sample sets unreliable, the accuracy metrics indicate little benefit for creating localized conversion equations by region. The summary statistic CCC suggests some improvements with the localized method transfer equations for SIDP and DML. However, except for LOI550, the maximum increase in CCC was 0.15. That increase in CCC observed in the smaller SIDP subset of 29 samples may not be sustained at larger sample sizes.

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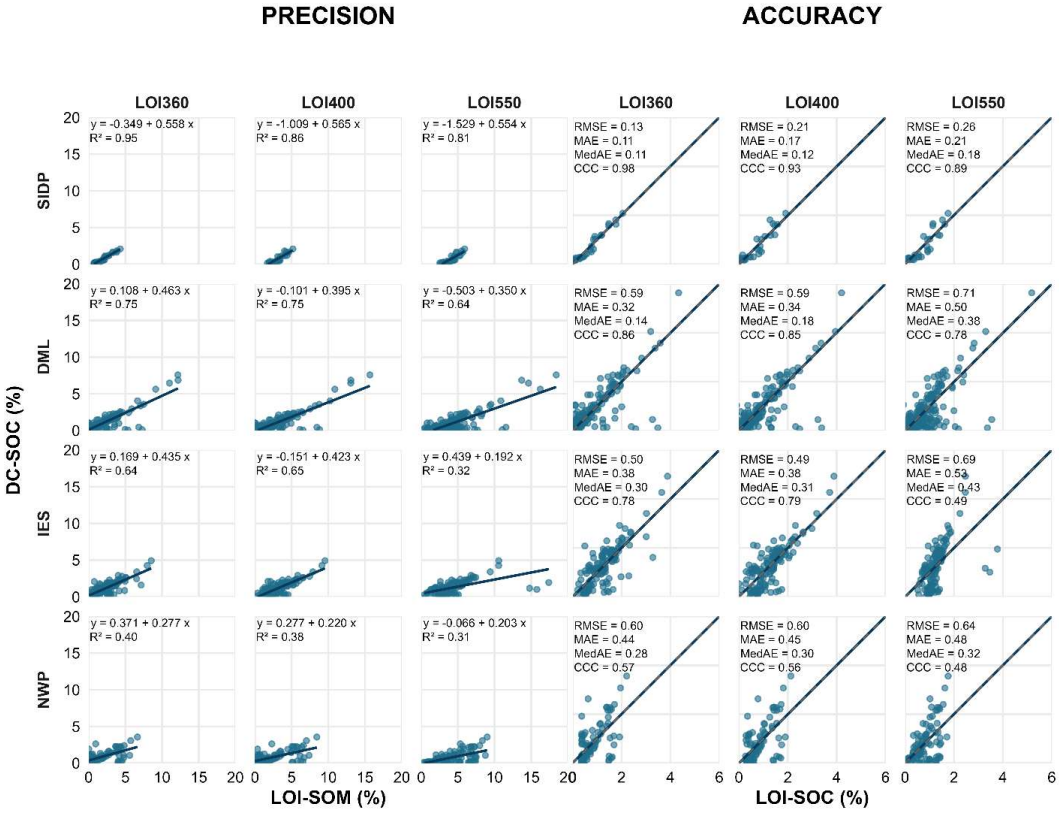


**Figure 7: Comparison of the precision (left three columns) for the relationship between DC-SOC (%) and LOI-SOM (%) and the accuracy (right three columns) for the prediction of DC-SOC (%) by LOI-SOC (%) across the four landform regions (SIDP, DML, IES, and NWP) and three LOI protocols. The results for the DML and IES generally align with those for the entire data set. The SIDP stands out with reduced variance, increased precision, and increased accuracy. The smaller sample size for SIDP likely led to a more consistent mass loss from non-SOM sources, which the method transfer equation could better accommodate. The poor performance of the IES method transfer equations across the board suggests the presence of more variable sources of mass loss in that localized sample set that are more difficult to account for in a simple method transfer equation.**

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The mineralogical differences between soils across landform regions in this study were insufficient to substantially affect the precision or accuracy of converting LOI-SOM measurements to DC-SOC. While the SIDP samples benefited from a localized method transfer equation, the similar NWP samples did not. Because soil parent materials in the SIDP and NWP regions both contain smectite and illite, the difference in model performance between these two regions is unlikely to be explained by their clay mineralogies alone. This analysis suggests that the variability of clay mineral concentrations is a more significant factor in localized method transfer equations than the type of clay minerals alone. That is, if the relative contributions of clay minerals to mass loss are consistent, then a method transfer equation can compensate. Although precision and accuracy appear to improve with localization for the SIDP, this improvement may be offset if additional samples reveal greater variability in the region's clay minerals.

### 3.2.3. Generalized versus localized method transfer equations

In summarizing this analysis, the focus is on CCC, a metric designed to compare two measurement methods, and on prediction accuracy, as these are most closely connected to the practical implications of measurement error. Across the three LOI protocols, localizing the method transfer equation showed some improvement in model performance in nearly half of the cases (Figures 8 and 9). However, there were more improvements for the protocols that tended to either underestimate (i.e., LOI360) or overestimate (i.e., LOI550) SOM. This pattern suggests that localized method transfer equations compensate for the interactions between protocols and local soil characteristics. For example, the greatest improvements occurred for LOI550, where high temperatures interact with varying concentrations of clay minerals susceptible to mass loss.

Segmenting the data set into increasingly fine local conditions highlighted the challenge of obtaining sufficient sample quantities for increasingly specific method transfer equations. Many localized method transfer equations that outperformed the generalized method transfer equation (red dashed line) tended to have small sample sizes ($n < 10$), which can lead to overfitting that would decline in performance if more samples were analyzed. This interpretation is reinforced by the fact that in many cases, where CCC increases, MedAE does not decrease proportionally.

The contrast between the performance of localized method transfer equations for SIDP and NWP illustrates the interaction between sample set size, CCC, and MedAE. The SIDP showed improvements in CCC for the majority of the data segmentations, but the entire region had only 29 samples, and subdivisions of those samples were even fewer. In contrast, the NWP showed minimal to no improvements in CCC from localization, as only two of the segmentation sets had fewer than 20 samples. Although the MedAE decreased below the MedAE for the generalized method transfer equation in most of the SIDP's localized method transfer equations, the difference was a maximum of 0.1% SOC, excluding localized method transfer equations with fewer than five samples and a variance of 0.01 or less. While generalized method transfer equations are broadly applicable regardless of the presence of IC, landform regions, or horizons, localized method transfer equations should be used only when the number of samples is large, and there is confidence that the sample variance represents the population.

The overall results indicate that localized calibrations rarely outperform the generalized method transfer equations in a substantial or robust manner. Because of this, we can assume that the generalized method transfer equations remain broadly applicable, and specialized method transfer equations from narrowly focused sample sets should be used only

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when an adequately large sample size and consistent reductions in error metrics are observed. If other factors contributing to mass loss (e.g., clay mineralogy) are accounted for, the precision and accuracy of predicting DC-SOC from LOI-SOM could be improved. However, while landform regions, horizons, and the presence of IC showed some correspondence with these confounding factors, they were insufficient proxies for achieving strong improvements.

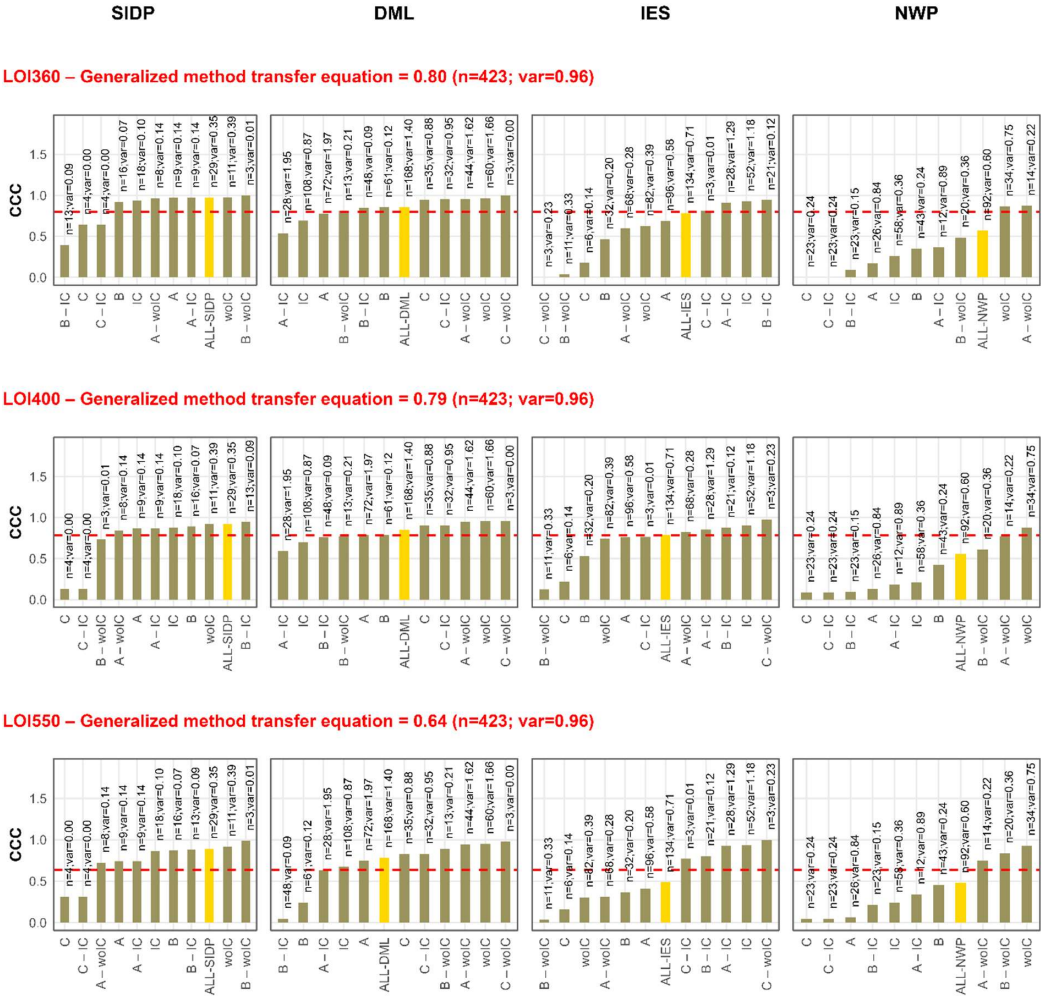


**Figure 8: Comparison of CCC values between generalized and localized LOI-SOC method transfer equations for LOI360, LOI400, and LOI550 across the four landform regions evaluated. The red dashed line represents the CCC of the generalized method transfer equation using all samples (ALL) from this study, which serves as the reference for evaluation. Bars exceeding this line represent localized method transfer equations with improved concordance relative to the generalized method transfer equation. Yellow bars correspond to the method transfer equations localized to the respective landform regions only (i.e., models built using all samples within each landform region), while olive bars represent method transfer equations created by segmenting the data to more specific factors (IC content, horizon, or their combinations). Segmentations involving samples with IC are labeled "IC", whereas segmentations involving samples without IC are labeled "woIC". Horizon-**

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based segmentations are denoted by A, B, and C. Although localized improvements are possible, the maximum improvement was a CCC change from 0.64 to 0.95, achieved by the lowest-performing LOI550 protocol. Observed improvements greater than this are unlikely to be reliable due to the limited number of available samples.

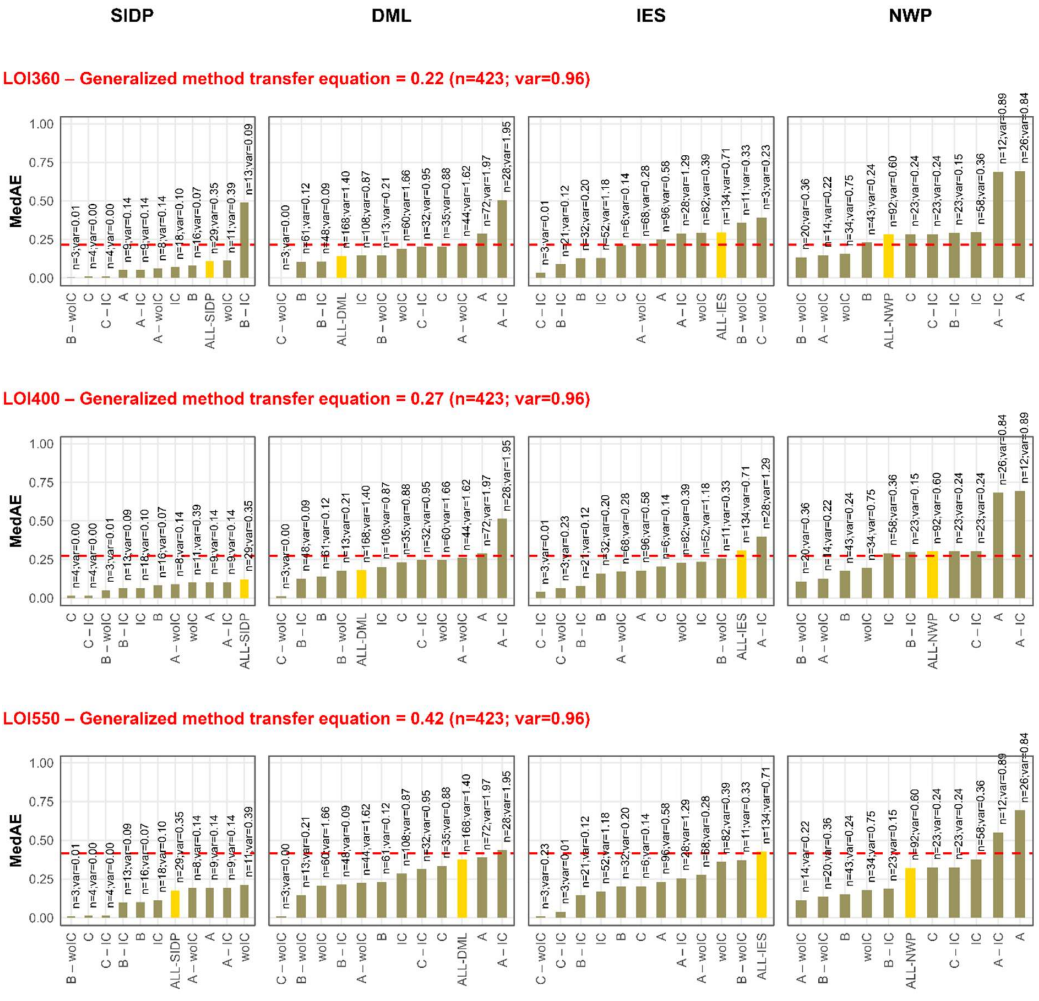


Figure 9: Comparison of MedAE values between generalized and localized LOI-SOC method transfer equations for LOI360, LOI400, and LOI550 across the four landform regions evaluated. The red dashed line represents the MedAE of the generalized method transfer equation trained on all samples (ALL) from this study, serving as the reference for evaluation. Bars below this line represent segmented method transfer equations with lower error than the generalized method transfer equation. Yellow bars correspond to the method transfer equations localized to the respective landform regions only (i.e., models built using all samples within each landform region), while olive bars represent method transfer equations created by segmenting the data to more specific factors (IC content, horizon, or their combinations). Segmentations involving samples with IC are labeled "IC", whereas segmentations involving samples without IC are labeled "woIC". Horizon-based segmentations are denoted by A, B, and C. Although localized improvements are possible, the maximum

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**improvement was 0.37% SOC, achieved with the least accurate LOI550 protocol. Observed improvements greater than this are unlikely to be reliable due to the limited number of available samples.**

### 3.3. Repeatability and evaluation of measurement error

Multiple measurements of the same samples demonstrated that DC had the highest repeatability, as indicated by the lowest CV. Among the LOI protocols, LOI400 had the highest repeatability, with a CV of 5.3%, followed by LOI360 (7.6%) and LOI550 (26.9%) (Table 4). Although the different LOI protocols had lower repeatability than DC, LOI400 and LOI360 still maintained suitable repeatability for many applications. These results are consistent with Konen et al. (2002), who measured SOC using DC and SOM using LOI360. They reported a CV of 2.6% for DC-SOC, identical to our findings, and a CV of 4.1% for LOI360, which is 3.5 percentage points lower than our results. However, a higher CV in our study is expected, as we included samples from multiple landform regions, including soil with and without IC, and from A, B, and C horizons. In contrast, Konen et al. (2002) evaluated samples from different landform regions separately and did not include any samples from C horizons.

**Table 4: Descriptive statistics and CV as a measure of repeatability (analytical precision) for DC and three protocols of LOI. DC-SOC had the highest repeatability, but lower temperature LOI protocols were not substantially less precise.**

| | DC-SOC | LOI360-SOM | LOI400-SOM | LOI550-SOM |
|---|---|---|---|---|
| Mean (%) | 1.70 | 2.08 | 2.88 | 4.25 |
| Minimum (%) | 0.24 | 0.10 | 0.35 | 1.33 |
| Maximum (%) | 4.90 | 5.41 | 6.71 | 7.10 |
| n | 73 | 45 | 45 | 45 |
| CV (%) | 2.60 | 7.60 | 5.30 | 26.90 |

n = number of samples, CV = coefficient of variation expressed in percentage.

Under emerging soil C market scenarios, uncertainty in SOC measurements is an important limitation that, for example, contributed to the collapse of the Chicago Climate Exchange voluntary carbon market (Gosnell et al., 2011). Estimates of SOC stock accuracy are influenced by soil spatial variability, sampling design, and the laboratory method implemented (Stanley, 2023). Laboratory methods must be able to detect small changes under heterogeneous conditions (Lehmann et al., 2007).

Some authors have argued that if the goal is to measure SOC, carbon should be measured directly using methods such as DC (e.g., Pribyl, 2010). However, others have expressed concerns about the analytical variability of DC (Chatterjee et al., 2009; O'Rourke and Holden, 2011). The present study found that DC indeed offers superior replicability compared to LOI. However, LOI, particularly LOI400 and LOI360, also demonstrate satisfactory repeatability. For instance, based on the obtained CVs for SOC measurement, when analyzing a sample with 3% SOC, DC exhibited a variability of approximately ±0.078%. In contrast, LOI400 and LOI360 exhibited variability of ±0.16% and ±0.23 %, respectively.

Although DC demonstrates the highest repeatability, accounting for spatial variability is crucial in detecting changes in SOC. In addition to equipment costs, cumulative analysis costs must be considered, as greater spatial variability requires larger sample sizes. For measuring SOC by DC, the price in a commercial laboratory in the Midwest USA is around $25 per sample, while the cost of SOM measurement using LOI is around $2 per sample. At a sampling rate

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of one sample per hectare (Mallarino and Sawyer, 2023), a 65-ha field would cost $1,625 using DC and $130 for LOI (Figure 10). Conversely, if the goal were to stay within a budget or to maximize the spatial support for characterizing SOC in that 65-ha field, $1,625 would provide either 65 DC samples or 812 LOI samples. While the CV for LOI400 may be double that of DC, the cost of LOI would enable the analysis of 12 times as many samples as DC.

Consider the relationship between the sample standard deviation (**s**) and the number of samples (**n**) in the paired t-test. The denominator for calculating **t** is **s** divided by the square root of **n**. If we assume that measurement error is the primary source of variability represented by s, then increases in **s** could be compensated for by increasing **n**. The **s** for the SOC samples analyzed by LOI400 (1.52%) was about three times that of the s for DC (0.442%). For LOI400 to have the same denominator in the t-test as DC does with 100 samples, 1,183 samples would be required. The costs become similar with these sample quantities and the additional labor required to collect more samples. However, this exercise assumed that the variability in the samples was due solely to measurement error. Any real spatial variability in the samples collected would also increase s, meaning that the more SOC varies within a field, the more the DC scenario would be at a disadvantage relative to the LOI400 scenario. For example, if field variability added one unit of standard deviation to the current scenarios, the denominator for the t-test of DC would be twice that of LOI400, halving the t-value and greatly increasing the threshold for statistically significant differences with the t-test.

Based on these results, certifiers now have the necessary information to make an informed decision. DC is a method with low measurement error but a higher cost. The cost of DC limits the number of samples available for statistical significance tests, which is especially critical in the presence of high spatial variability. In contrast, LOI400 has a higher measurement error but a substantially lower cost. By sacrificing accuracy only to an RMSE of 0.69 g $kg^{-1}$, the cost savings can be invested in collecting more samples to better assess spatial variation.

Together with the lower cost and, consequently, better assessment of spatial variation, collecting more samples with LOI could also support preliminary SOC screening across spatially variable areas. In this role, LOI400 could be used to identify zones of contrasting SOC and guide the selection of representative samples or targeted locations for subsequent DC analysis while also facilitating the generation of expanded SOC training data sets that support emerging remote sensing and machine learning approaches for SOC estimation. Modelers' SOC prediction frameworks increasingly rely on algorithms calibrated with ground samples to capture spatial variability across contrasting landscapes. Because LOI is inexpensive, fast, and with good repeatability, it would enable researchers to assemble much denser calibration sample sets than would be economically feasible using DC alone. Having larger sample sets could be particularly valuable for training multi-sensor SOC models that use different indices (e.g., NDVI, SAVI, EVI), topographic attributes, and other covariates to create robust prediction models for mapping SOC and evaluating its spatial and temporal variation.

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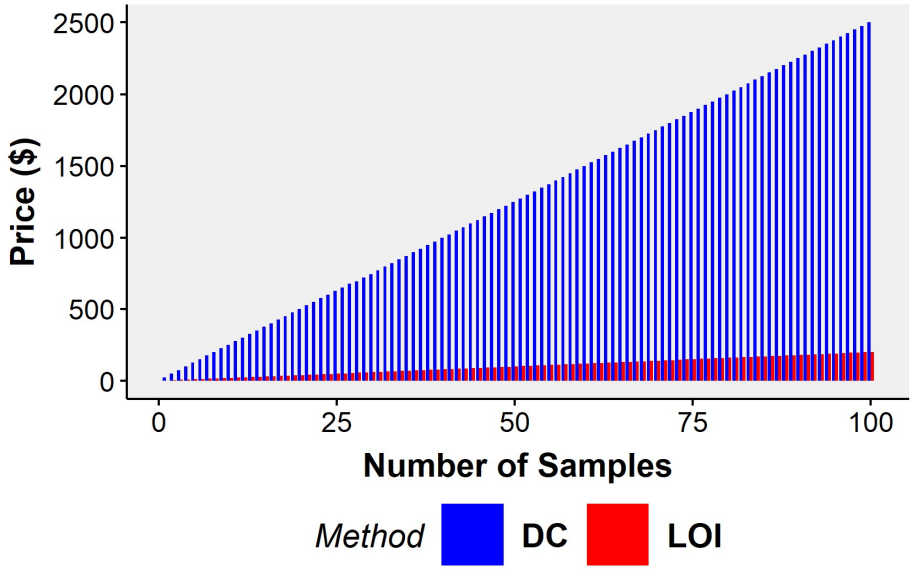


**Figure 10: Price comparison by method. Prices for DC and LOI were based on the analytical cost of a commercial lab in the USA Midwest in 2026, which was $25 and $2 per sample, respectively. The number of samples can be interpreted for either sampling larger areas or increasing sampling densities. With a focus on increasing statistical certainty for change detection, the lower repeatability of LOI can be compensated for by increasing the number of samples analyzed.**

## 4 Conclusions

This study shows that LOI should not be dismissed as an unreliable method for estimating SOC, but neither should it be used through fixed method transfer equations or interchangeable protocols. The results demonstrate that LOI400 (400°C for 16 hours) stands out as a reliable alternative to DC for estimating SOC. While DC remains the benchmark for repeatability (analytical precision), its high cost and operational complexity restrict the number of samples that can be feasibly analyzed, which is a critical limitation when addressing spatial variability in SOC stocks. In addition, SOM is relevant to other environmental and agronomic management questions, thereby increasing incentives to monitor it beyond carbon markets alone.

LOI400 and LOI360 (360°C for 2 hours) both deliver comparable precision and accuracy for predicting DC-SOC, with LOI400 exhibiting the highest repeatability among the LOI protocols. In contrast, LOI550 (550°C for 3 hours) is less suitable for generalized SOC estimation due to increased measurement error resulting from mineral mass loss, particularly in soils with variable clay minerals, such as kaolinite, smectite, and illite. Although those additional sources of mass loss could be measured and compensated for in the method transfer equation, such additional analyses would defeat the purpose of keeping costs lower than those of DC.

The method transfer equations evaluated in this research are not intended to estimate the concentration of C in SOM. They are the relationships between the methods of LOI and DC, which mathematically compensate for additional

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sources of mass loss in soil samples. A critical question is whether a generalized method transfer equation is sufficient or whether localized method transfer equations are needed. There is no simple answer to this question because it is difficult to compare model fitting and performance on sample sets with inherent differences in variance. Nonetheless, this research scrutinized method transfer equations based on practical strategies for localization, using samples with varying textures, clay mineral abundance, and SOM concentrations. Evidence for only minor improvements was found. The accuracy of estimating DC-SOC from LOI400 and LOI360 was high, with only minor opportunities for improvement.

Importantly, excluding samples with inorganic carbon (IC) from the analysis substantially improves the precision of LOI-based SOC predictions, but not necessarily the accuracy. Moreover, the development of localized method transfer equations, tailored to specific regions or soil horizons, provides additional benefits where soils in local areas have different clay mineralogies. For most practical applications, generalized method transfer equations are sufficient, simplifying SOC monitoring protocols and reducing the need for extensive mineralogical characterization.

A key advantage of the LOI method is its low cost, which enables much higher sampling densities. This increased sampling capacity can offset the slightly higher measurement and prediction errors relative to DC, enabling more robust detection of spatial patterns and changes in SOC stocks. Moreover, LOI400 can also serve as a preliminary screening tool to identify zones of contrasting SOC and guide the selection of representative locations for subsequent DC analysis. In scenarios where resources are limited or spatial variability is high, LOI400 provides a practical solution for assessing SOC across large areas and verifying carbon markets. In addition, the ability to generate denser, lower-cost SOC data sets supports emerging remote sensing and machine learning approaches that require extensive ground observations for model calibration and SOC digital mapping.

In summary, the choice between DC and LOI methods should be guided by the specific goals of SOC monitoring, available resources, and the degree of spatial variability in the study area. LOI400 is recommended for most applications, providing a balance between reliability and cost to achieve a suitable sampling density. Localized method transfer equations may be justified in regions with consistent mineralogical influences; however, generalized method transfer equations are generally adequate for broader use. These findings support adopting LOI400, per the USDA-NRCS Kellogg Soil Survey Laboratory Methods Manual (2022), as a practical and scalable approach for estimating SOC. The generalized equation for converting LOI400-SOM to the DC-SOC equivalent was LOI-SOC = 0.365 * LOI-SOM -0.039.

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## Data availability

The dataset analyzed in this study is available from the corresponding author upon reasonable request because it forms part of an ongoing research program and is subject to institutional data management policies.

## Supplement link

The link to the supplement will be included by Copernicus, if applicable.

## Author contributions

Luis Bentancor led the study and was responsible for conceptualization, study design, data collection, data curation, formal analysis, and writing the manuscript. Bradely Miller made substantial contributions to the development of the study, data interpretation, and manuscript revision. Michael Thompson and Lee Burras reviewed the manuscript and provided critical comments that improved the scientific quality and clarity of the work. Peter O'Brien contributed to manuscript review and revision and provided technical expertise and support for laboratory analyses related to dry combustion measurements. All authors reviewed and approved the final manuscript.

## Competing interests

The authors declare no competing interests.

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