2. Soil carbon monitoring based on repeated measurements

DESIGNING A SOIL CARBON SURVEY

The objective of the nationwide soil carbon inventory is to provide unbiased estimates of the soil carbon stock and stock changes. In designing soil carbon monitoring, relevant issues to be considered are:

- sampling intensity (number of study sites) and rules to locate the study sites
- sampling interval (e.g. decadal or five-year interval)
- soil layers to be studied
- location of sample points and their number at the study sites

Constructing a sampling design is an optimization problem, where the trade-off is between required resources and reliability of resulting estimates. When planning sampling to detect a potential change, it is necessary to know how many samples need to be taken in order to estimate a statistically significant change. Appropriate sampling intensity (sample size) and sampling interval (time between consecutive measurements) can be designed after collecting information on between-site variation and estimation of the rate of potential change. Efficiency of the sampling can be improved by stratification, if prior information on the variation of the target variable is available.

Selection of the soil layers to be studied can be guided by the known sensitivity of different layers to human-induced disturbances. The 2006 IPCC guidelines recommend using a default 0–30 cm layer. Within this layer, the influence of management practices is more pronounced than in the deeper soil layers (Bationo *et al.*, 2007). Knowledge of within-site variation of soil properties can be used to determine the number of subsamples per plot.

Soil samples that are taken and prepared for soil organic carbon analyses can also be used for the measurements of other soil variables (e.g. soil nutrients, pH and grain size distribution). Since many soil properties are related, for example, to site productivity and site protection from erosion, an evaluation should be made as to which measurements are feasible and how/where samples can be stored for additional analyses in the future.

IMPROVING THE EFFICIENCY OF THE SAMPLING EFFORT

The efficiency of soil carbon monitoring can be improved and costs reduced by stratified sampling. Stratification increases efficiency if a subdivision of the population is made so that within-stratum variability is lower than the variability within the entire population.

Stratification by land-use category, soil type, etc., i.e. by groups where rates of soil carbon change are similar, will improve efficiency of the sampling targeted for soil carbon monitoring. A target variable in the soil carbon monitoring is a change in the soil carbon stock, but there is far less information on spatial variation of soil carbon changes than on soil carbon stock (Häkkinen, Heikkinen and Mäkipää, 2011). Therefore, information on the variation of carbon stock (or other measured soil variables) may be used for sampling design, but such information on stocks cannot be used for stratification since large soil carbon stocks do not necessarily indicate major changes.

It has been estimated that, by grouping similar sites (in terms of model-predicted change in the soil carbon stock) into the same stratum, and optimally allocating samples to these strata (according to the size and variance of the strata), the number of plots can be decreased by 25 percent without a reduction in the precision of the estimates (Peltoniemi, Heikkinen and Mäkipää, 2007). The gain provided by stratification is, however, sensitive to uncertainties in the predicted changes (e.g. those resulting from future thinning activities) and precision of the measurements. Furthermore, stratification with the target of soil carbon monitoring may result in an allocation of the sampling effort that is less effective for assessment of other soil parameters than simple random sampling (Peltoniemi, Heikkinen and Mäkipää, 2007).

INFORMATION NEEDED TO DESIGN A REPEATED SOIL CARBON INVENTORY

Between-site variation in soil carbon stock and number of plots to be measured

In designing soil monitoring, information on between-site variation of soil properties can help decide upon the total number of plots to be measured. The first round of a nationwide soil carbon inventory will yield information on the between-site variation of soil carbon stock that can be used to estimate the number of plots allowing detection of change after a repeated inventory.

The number of plots needed for detection of a change in the soil carbon stock is estimated using the equation:

$$n = (t \times s / E)^2 \quad (1)$$

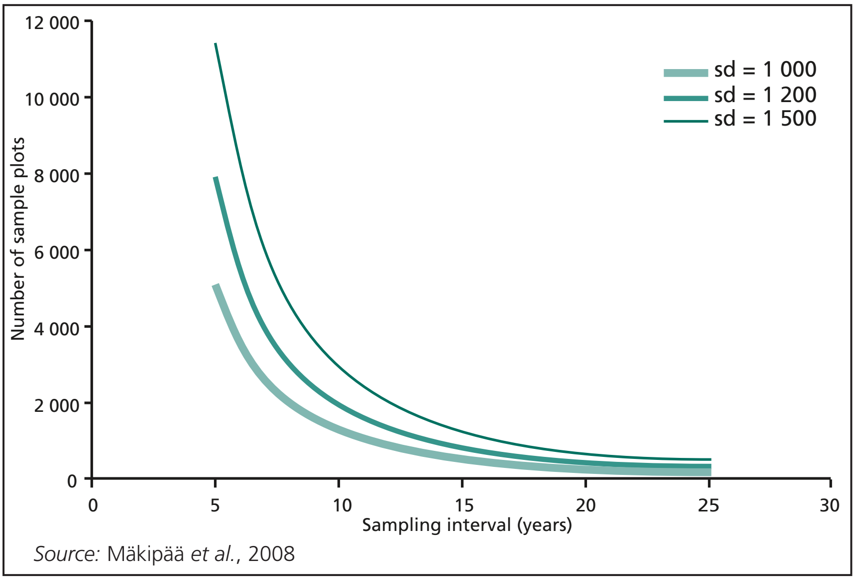
where n is the number of sample plots required, t is a value taken from the student's t distribution table for a given number of degrees of freedom and desired confidence interval, s is the estimated standard deviation of the measured values (assuming independency of the observations), and E is the desired half of the confidence interval. This is a general formula that applies when soil carbon stock measurements have a normal distribution.

The desired confidence interval of a carbon stock estimate should allow detection of the expected change in the soil carbon stock. In general, the rate of soil carbon change is not known, but can be estimated with soil modelling (e.g. Liski *et al.*, 2006) or generalized from CO_2 flux measurements of selected sites (e.g. Malhi and Grace, 2000). If we assume that the average rate of change is 11 g C m^{-2} yr^{-1} (as has been predicted by soil modelling in a country-scale assessment by Liski *et al.* [2006]), the desired half of the confidence interval of the carbon stock estimate (E) is 27.5 g C m^{-2} with a sampling interval of five years, increasing to 137 g C m^{-2} with extension of the sampling interval to 25 years (assuming that the carbon change increases linearly over time) (Mäkipää *et al.*, 2008). In disturbed forest soil under high anthropogenic pressure, temporal variation of soil carbon stock might be high and linear trends non-existent. In such cases, detection of the change with repeated measurements is possible only with very short sampling intervals.

We can assume a standard deviation of 1 000 g C m^{-2} (which is the value reported for the mean carbon stock of the uppermost soil layers by Peltoniemi *et al.* [2004]), and also apply values of 1 250 and 1 500 g C m^{-2} in order to determine the sensitivity of the results to this assumption. With these assumptions, the approximate estimate for the minimum number of sample plots required to detect a change in this soil layer is 3 000 plots with a sampling interval of ten years (Figure 1). This sampling intensity allows detection of an expected change of 110 g C m^{-2} per ten years if the standard deviation of the measured carbon stock is less than 1 500 g C m^{-2}. With a local representative estimate for variation of soil carbon stock (standard deviation of mean carbon stock) and prediction of the rate of soil carbon change, this calculation can be updated for a target region or country.

FIGURE 1

Sample size required with different sampling intervals calculated on the assumption that the change to be detected is 11 g C m^{-2} per year. Standard deviation (sd) of measured carbon stock was assumed to be 1 000 g C m^{-2}. Larger values (1 250 and 1 500 g C m^{-2}) were applied to show the sensitivity of the results to this assumption



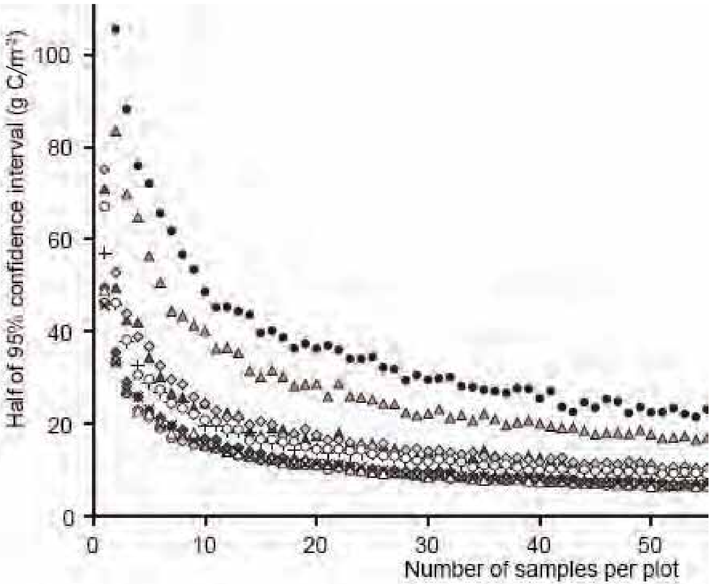
Source: Mäkipää *et al.*, 2008

Within-site variation and determination of number of subsamples per plot

Knowledge of the within-site variation in the soil carbon stock is used to determine the number of subsamples per plot yielding estimates that are accurate and precise enough for monitoring purposes. The accuracy of the mean estimate increases with the number of samples per plot. For instance, in a study focused on within-site spatial variation of the carbon stock of the organic layer, 73–116 samples were taken with a soil corer at ten study sites (Muukkonen, Häkkinen and Mäkipää, 2009). The authors' results indicate that required accuracy is usually obtained with 10–20 samples per plot, after which increases in the sample size do not significantly increase accuracy (Figure 2).

FIGURE 2

Confidence intervals of the carbon stock of the organic layer according to sample size for ten different study sites



Source: Mäkipää *et al.*, 2008

The appropriate number of subsamples can be determined in a case study where plots are sampled in more detail and each of the collected samples is analysed separately. Thereafter, determined sample size per plot can be adopted for an operationalized soil inventory, where soil samples taken from the same plot and same soil layer can be pooled to reduce the costs of laboratory analyses. However, the use of pooled samples (one sample analysed per layer per plot) means that information on the within-site variation is lost and it is difficult to determine when the difference between two measurements is significant at plot scale. Despite these drawbacks, the pooling is efficient and accepted in regional and national soil surveys aimed at assessing change in the mean carbon stock.

UNCERTAINTIES IN SOIL CARBON SURVEYS

Sources of uncertainty

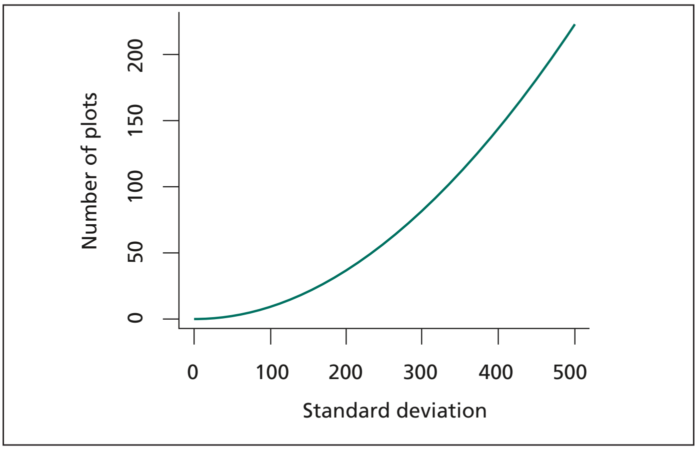
The possibility of deriving uncertainty estimates based on a formal statistical procedure is an important advantage in applying a sample-based inventory. This section describes the different sources of error in soil surveys and their effects on overall uncertainty in soil carbon estimates. The major types of error in a soil carbon inventory are those of sampling and measurement. In addition, model errors, classification errors in upscaling (e.g. plots recorded as afforested grassland misclassified to grassland during the processing of the data) as well as registration and calculation errors may be potential sources of uncertainty in a sample-based inventory (IPCC, 2003). Furthermore, inclusion of data from outside the population (e.g. plots located on land that is not among the target land-use categories) or sampling that does not include some elements of the population (e.g. ignoring the organic layer) may cause errors.

Sampling errors

Sampling is a practice that is used to select a subset of objectives within a population in order to yield some knowledge (e.g. soil carbon amount) about the population of concern, since it is not feasible to measure the entire population. In soil carbon inventories, systematic sampling is most commonly applied, since it is effective and easy to implement. However, efficiency of the sampling can be improved and sampling errors reduced with stratification if a priori information on target variable is available. Using proper sampling design (random or systematic, stratified or non-stratified), sampling errors are only random and can be reduced by increasing the sample size, which results in greater precision. On the other hand, heterogeneous soils require a larger sample size to reach a degree of precision that is adequate for detection of a change by repeated measurements (Figure 3).

FIGURE 3

Number of sample plots needed for detection of soil carbon change by variation (sd of soil carbon stock change g C/m^2) when the amount of change is assumed to be 400 g C/m^2 between initial and second sampling of a soil layer



The uncertainty estimates derived with sampling principles for sample-based variables can be combined with the uncertainties of other data sources and models using error propagation equations (e.g. IPCC, 2003).

Measurement errors

Measurement errors can lead to substantial systematic errors, especially where changes are estimated by repeated measurements and the systematic error levels and direction vary over time. The size of measurement errors can only be estimated by careful control measurements on a subsample of the plots (e.g. 1/20 of the sites/samples/elements re-measured with careful control measurements). Furthermore, in some cases it is possible to obtain true measurement values through highly accurate control procedures, e.g. potential systematic errors in the carbon content derived with wet combustion can be checked by more accurate carbon analysis of the very same samples with a dry combustion analyser (where products of combustion are measured using a thermal conductivity detector). Potential sources of measurement errors considered in this report are soil density measurement; determination of depth of soil sample collection; errors introduced by incorrectly calibrated laboratory instruments; and systematic errors introduced by incorrectly calibrated methods of carbon analysis in participating laboratories.

In soil carbon stock and stock change measurements, errors in soil density measurements are typical sources of uncertainty. Since both carbon content and soil density vary across soil profiles, volume-specific soil samples need to be taken from all soil layers that are accounted for. A volume-specific cylinder is the recommended sampling device and it is important that sampling be carried out extremely carefully, i.e. the cylinder is filled properly. In the case of challenging soil texture such as stone-packed coarse soil or compact clay soil, it may be necessary to use a smaller cylinder, but the volume of the cylinder should be recorded to allow accurate estimates of the soil carbon amount (g C/m^2).

In general, soil carbon concentration decreases with soil depth and for detection of the soil carbon stock change it is important that soil samples of consecutive inventories represent the same soil layers. If the soil sampling layer is determined to be, for example, the topmost 30 cm, the soil samples may cover the entire profile down to 30 cm (e.g. samples from the organic layer and 0–10 cm, 10–20 cm, and 20–30 cm mineral soil layers) or they may be point samples and the change in the soil carbon concentration between sample points is interpolated or modelled. The soil sampling procedure in the first sampling and in consecutive samplings should be as similar as practicable.

Laboratory measurements may be a major source of error, if necessary calibration and testing of the methods are not carried out. A key instrument is balance, which needs to be calibrated according to the guidelines and calibration sets provided by the manufacturer. Since precision of the balance has to be high enough for the intended measurements, it is recommended recording precision as given in the documentation of the used balance in data files.

For the determination of soil organic carbon various methods can be used. A common method is to determine organic matter content by loss of ignition and to convert that into carbon concentration by using the van Bemmelen conversion factor, which assumes that soil organic matter is 58 percent carbon. Another option for the determination of organic matter is the wet-chemical oxidation technique (Walkley-Black, or a modification), but this requires the use of potassium dichromate, and disposal of the waste is problematic. Furthermore, it may not measure all the organic carbon, but only an easily oxidized part. A more accurate carbon estimate can be obtained with a dry combustion analyser that gives carbon (and nitrogen) concentrations directly. High-temperature dry combustion can be used as a standard against which other techniques are calibrated. All methods have potential for major errors in sample preparation, in the determination of moisture content of samples and in the performance of the method itself. To avoid biased results, it is necessary to calibrate the method and to check the consistency of the analysis performed in the different laboratories and at different times. This can be done by analysing samples with known carbon concentration as test samples. Any significant deviation of reference concentration will lead to necessary repair or calibration of the instrument.

QUALITY CONTROL AND QUALITY ASSURANCE IN SOIL CARBON INVENTORIES

Quality control (QC) is a system of routine technical activities to assess and maintain the quality of the inventory as it is being implemented. It is carried out by personnel implementing the inventory. Quality assurance (QA) is a planned system of review procedures conducted by personnel not directly involved in the inventory. Reviews, preferably by independent third parties, are undertaken upon a completed inventory following the implementation of QC procedures (IPCC, 2006). The objective of the QC/QA process is to increase and ensure the credibility of the soil carbon inventories.

Need for consistency control

Differences in soil sampling, sample preparation and procedures of chemical analyses can introduce considerable systematic errors in soil carbon estimates. The resulting soil carbon stock estimates may be biased and change estimates may be invalid if level and direction of error vary over the monitoring period. Major systematic errors can occur in cases where an estimate is based on a population that is different from the population where the estimate is to be applied (i.e. biased or statistically non-representative sampling).

Soil sampling

At the time of establishment of a network of monitoring plots, it is essential that recording of land-use category, human activities, natural vegetation type and soil type as well as procedures to select plots to be sampled are consistent across the sampled population. In consecutive inventories, the same rules and delineations of the sampled population (e.g. land-use categories to be included) must be followed. For instance, if urban areas are excluded from the soil inventory and a potential sample plot is located in land that is turning from urban into forest land, which is accounted for in the sampling population, a new plot needs to be established in the second inventory.

Delineation of soil layers (organic versus mineral soil) and determination of soil sampling locations in the soil profile (depth from soil surface) have to be consistent across the entire country in nationwide inventories. Since the topmost layer has a higher carbon content than the deeper soil layers, slight differences in sampling depth may introduce high systematic errors. This is a considerable source of uncertainty in soil carbon monitoring and can be minimized only by careful fieldwork. A continuous soil sample, which starts from the soil surface and reaches down to the selected depth, would be optimal to avoid errors in the location of the sampling layer, but such samples are extremely difficult to obtain from compact or stony soils. Therefore, an approach where representative samples from different soil depths are taken is recommended.

In cases where it is not possible to follow a sampling protocol, deviations should be recorded and documented with necessary details on data forms in order to ensure correct calculations. Consistency of the sampling can be tested by repeated measurements, i.e. cross-checking by another field team repeating all measurements (without knowing the data collected by the first team), but it is not possible to obtain duplicated soil samples for comparison from the very same location.

Checklist for QC/QA activities of field measurements

- Check for consistency of procedures to select plots across sampled population; follow the same rules during entire inventories and consecutive inventories.
- Check that plots have been installed and measured correctly, by having approximately 10 percent of all plots re-measured by an independent team. If the deviation between measurement and re-measurement is greater than 5 percent, investigate the source of the error, record and correct.
- Check for consistency of the soil layers to be sampled.

Laboratory analyses

Consistency checking between laboratories before they start processing soil samples

A detailed protocol of sample preparation and laboratory analyses needs to be prepared in order to obtain consistent results if analyses are carried out in different laboratories. In addition, it is necessary to analyse a set of test samples in all participating laboratories. In practice, soil samples that represent a range of different soil types are taken and homogenized in one laboratory. A homogenized soil sample is then divided into identical subsamples, which are analysed in participating laboratories according to a common protocol. Results of the analyses are reported to the coordinating laboratory where the consistency of the participating laboratories is checked. Should the results submitted by a laboratory deviate from the accepted mean value, they will fail the test and cannot participate in analyses of the real samples until they are able to reach an acceptable level of consistency with other laboratories.

Should laboratories apply different methods in carbon analysis, calibration of the methods is necessary and the dry combustion technique should be used as a standard against which other techniques are calibrated.

Test/calibration samples to be analysed during processing of soil samples

After initial checks of the participating laboratories, QC of the laboratories needs to be continued by regular analyses of the test samples. These samples can be identical subsamples of a large soil sample with known carbon content. The samples distributed to each of the participating laboratories are analysed at regular intervals with other soil samples. Should results deviate from earlier analyses and from "known values", the procedure must be checked and sources of the errors eliminated.

All measurements should be conducted in laboratories where QC practices are well understood and followed. Standard laboratory practices include documentation of sample handling and preparation before analyses, procedures for all analyses, sample storage after analysis, and calibration of instruments. In addition, participating laboratories are expected to report implementation of QC techniques, including replicate samples of standard analyses, internal QC samples, and reference samples of the nationwide soil survey.

Checklist of QC/QA activities of laboratory analyses

- Adhere to protocol of sample preparation and laboratory analyses at all times.
- Conduct laboratory analyses for replicate samples and check results.
- Cross-check results of analyses by another laboratory and explain any differences identified.
- Calibrate methods if methods vary across laboratories.

Checklist of QC/QA activities of data entry and analyses after laboratory measurements

- Cross-check a sample of input data for transcription errors.
- Ensure that all files are named appropriately.
- Check that units are properly labelled.
- Check for consistency among time-series data; identify outliers as soon after the actual measurement as possible and investigate the causes of the outlying observation, and correct if needed.
- Compare estimates from field measurements and laboratory analyses with literature values.
- Review the entry of data into the data analyses spreadsheets by an independent verification.