Chapter 15

Forest Soil: Characterization, Sampling, Physical, and Chemical Analyses

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Chapter Outline

Developments in Environmental Science, Vol. 12. http://dx.doi.org/10.1016/B978-0-08-098222-9.00015-7

15.1 INTRODUCTION

Forest soil variability is enormous, even within a single monitoring plot. A soil profile, consisting of different layers and genetic horizons, is the result of a developing process, which continuously evolves and never stops. It is function of time, parent material (e.g., peat, sandy, or clayey deposits), type of bedrock (e.g., acid plutonic rocks, calcareous lime stones), (micro-)topography, climate, tree species composition and understory vegetation, soil biological activity, and natural (e.g., windthrow, fire) and anthropogenic disturbances (e.g., forest management). Moreover, air pollution (Driscoll et al., 2006; Gundersen et al., 2006) and climate change (Bytnerowicz et al., 2007; Polglase and Paul, 2011; Rennenberg et al., 2009) directly or indirectly affect forest soils.

The objective of soil sampling in forest monitoring is to obtain reliable information about the soil condition in a particular plot and its change over time. When repeating a soil survey, it is essential to avoid measuring spatial variation instead of temporal variation. Therefore, the sampling procedures should aim at minimizing the spatial variability and maximizing the accuracy of the mean plot values. The laboratory results depend on effective sampling: careless or inappropriate sampling in the field can never be solved by careful measurement in the laboratory (Bates, 1993). This chapter outlines both field sampling and laboratory analytical methods for soil properties which are relevant for long-term forest soil monitoring, for large-scale as well as intensive monitoring plots.

On the large scale, the soil component of a forest health monitoring program may assess those soil properties that determine the forest soil's sensitivity to environmental factors such as air pollution. This concerns mainly acidification and eutrophication (Cools and De Vos, 2011; Herman et al., 2001), though trace metals and persistent organic pollutants are also monitored at the regional or national level (Belis et al., 2009). Both the physical and the chemical status of the soil are considered. Soil biological properties and soil biodiversity are generally not yet addressed in international forest monitoring programs, although soil biodiversity indicators have been integrated into national programs, for example, in France (Cluzeau et al., 2012), the United Kingdom (Black et al., 2003), and The Netherlands (Rutgers et al., 2009). In addition to the study of atmospheric deposition effects, forest soil surveys may serve other environmental purposes, such as the inventory of carbon (C) stocks and sinks related to climate change (Palmer et al., 2002; Saby et al., 2008) and sustainable forest management (Burger and Kelting, 1999; Page-Dumroese et al., 2000).

Within the International Co-operative Programme on Assessment and Monitoring of Air Pollution Effects on Forests (ICP Forests), more intensive soil studies are conducted on permanent monitoring plots, where they are essential for understanding the role of forest soils in cause–effect relationships and in ecosystem functions and services. Intensive soil studies involve soil

characterization, an evaluation of the current soil condition and the study of the long-term soil processes and dynamics of, for example, nutrients (De Vries et al. 2003), carbon (Hilli et al., 2008; Jochheim et al., 2004), and the water balance (Hoermann and Meesenburg, 2000). Methods for measuring the composition of the soil solution and the soil water content are described in the related chapters (see Chapters 16 and 17).

For the sake of data comparability among the different partners in the monitoring program, it is a prerequisite that the same methods for soil sampling and analysis are used throughout the whole network. This chapter provides a consistent methodology for collecting high quality, harmonized, and comparable forest soil data across European forests. This will allow (i) the proper characterization and description of soil condition and (ii) the periodic monitoring of changes in soil properties (e.g., on a 10-year basis). Concerning field observations and sampling, the aim is to provide a set of minimum requirements in order to arrive at a harmonized approach. In terms of laboratory analyses, all laboratories should apply the same reference methods, which are usually based on International Organization for Standardization (ISO standards). The relevance of the key soil variables in forest soil monitoring is given in Table 15.1.

At the time of the plot installation, a detailed pedological characterization should be conducted. This comprises a detailed soil profile pit description complemented by sampling and laboratory analyses according to the genetic horizons. The humus and soil profiles should be classified according to internationally agreed classification systems. This information cannot be derived directly from national soil maps as: (i) these are generally based on national soil classification systems, (ii) they have often been made for agricultural purposes, usually with a lower survey intensity in forests, and (iii) map generalization and scale do not allow an accurate derivation of soil type at the plot level. This is illustrated by the disparity in the distributions of the WRB reference soil groups (IUSS Working Group WRB, 2007) described based on the second European forest soil condition survey (2004–2008) compared to those derived from the Soil Geographical Database of Europe (SGDBE version 4 beta, EC, 2004) for identical plots (Table 15.2).

After pedological characterization, the chemical and physical status of the forest soil is assessed through composite and core sampling at fixed depths. This sampling forms the baseline for repeated soil monitoring campaigns. In monitoring programs that focus on chemical changes to the soil, both the organic and the mineral soil layers are sampled and analyzed in the laboratory at regular time intervals (e.g., every 10 years). Measuring of the physical soil condition (bulk density, coarse fragment content, particle-size distribution, and soil water retention curve) at a single time is generally accepted.

In Figure 15.1 the soil sampling and analytical methods are described in chronological order, starting from the design of the soil survey and soil monitoring and moving up to the data reporting.

TABLE 15.1 Relevance of Key Soil Variables

Type of variable	Key soil variable	Layer	Relevance
Carbon and nitrogen	Total organic C, total N, and carbonates	Organic	Forest nutrition, atmospheric N deposition, and climate change
		Mineral	Forest nutrition (0–20 cm), C, and N sinks
Nutrients	Total P, Ca, Mg, K, Mn, and S	Organic	Atmospheric deposition of basic cations and stock of macronutrients
		Mineral	Weathering rates, critical loads of acidity, and stock of macronutrients
Acidity, cation exchange characteristics	pH, carbonates, cation exchange capacity, base saturation, and exchangeable cations	Organic and mineral	Buffering acid input and acidification status
	Acid ammonium oxalate extractable Al and Fe	Mineral	
Heavy metals	Pb, Cu, Zn, Cd, Cr, Ni, and Hg	Organic	Atmospheric metal deposition
		Mineral	Atmospheric metal deposition, calculation critical loads (0–20 cm), and deficiency of oligo elements
Physical soil variables	Particle-size distribution and soil texture	Mineral	Soil classification, estimation of plant available water, and nutrient exchange capacity
	Organic layer mass	Organic	Calculation of stocks
	Bulk density of the fine earth, coarse fragment content, and soil depth	Mineral	Calculation of stocks, nutrient supply to plants, index for compaction, and rootable soil volume
	Soil water retention characteristic	Organic Mineral	Water balance models, nutrient fluxes, and estimation of soil porosity

Source: Cools and De Vos (2010).

TABLE 15.2 The Relative Distribution of the Major Reference Soil Groups According to the World Reference Base for Soil Resources (WRB) on the Level I Forest Monitoring Network of ICP Forests: (1) Based on the Field Survey and (2) Derived from the Soil Map of Europe (1998)

WRB reference soil group	Distribution (%) based on field survey	Distribution (%) based on Soil Geographical Database of Europe (EC, 2004)
Cambisols	22.9	30.4
Regosols	15.5	4.6
Podzols	14.0	29.3
Arenosols	11.5	2.4
Leptosols	7.2	10.5
Luvisols	5.9	8.4
Histosols	5.2	5.4
Gleysols	3.4	2.3
Umbrisols	3.4	Not available
Stagnosols	2.9	Not available
Phaeozems	1.9	0.3
Alisols	1.2	0.0
Fluvisols	0.6	2.3
Albeluvisols	0.6	1.7
Other	3.7	2.5

15.2 FIELD SAMPLING AND FIELD MEASUREMENTS

15.2.1 Sampling Design

Ideally, the soil profile(s) described should be located in such a way that the dominant soil type, tree species, ground vegetation, slope, and surface stoniness in the sampling area are represented. The profile should be minimally affected by strong natural (e.g., windthrow) or anthropogenic disturbances. An exploratory bore register based on augering may help provide insight into the local pedodiversity. On slopes, the profile is best oriented with its longest axe in the slope direction. On flat terrain, it is recommended to orient the profile in such a way as to avoid unequal light distributions on the profile wall.

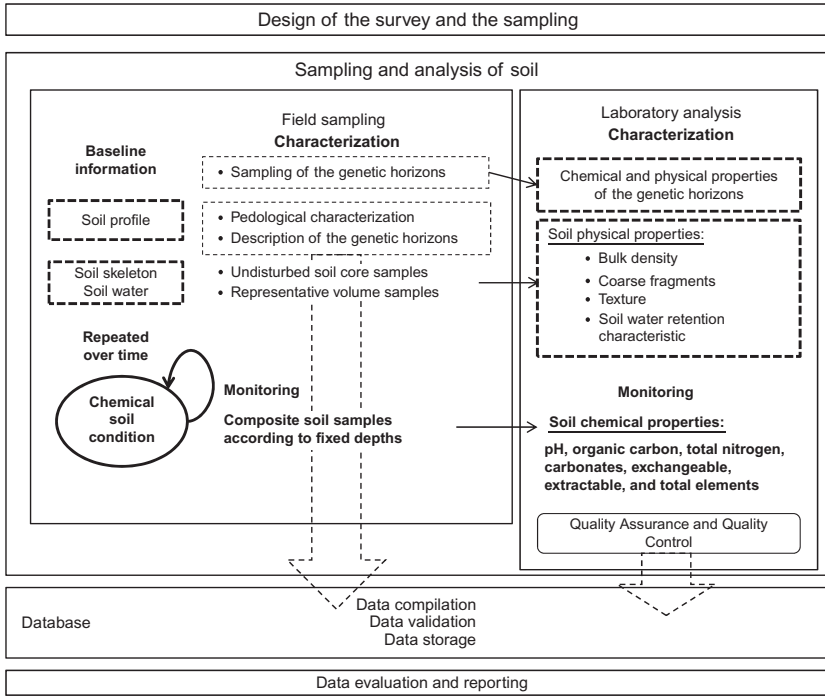

FIGURE 15.1 Overview of the stages in forest soil monitoring.

Note that observations made while digging can be very informative. These include rock fragment abundance and size, presence of compaction and cementation, number and dimensions of roots, and total rooting depth. In order not to interfere with the other observations or installations on the plot, the soil profile may be located in the buffer zone surrounding the plot, as long as this area has similar characteristics as the area within the plot.

Sampling sites should be avoided in areas around tree trunks (1 m) (e.g., Riha et al., 1986) and animal holes, as well as disturbances like wind-thrown trees and trails. A record of the sampling location should be kept for later reference, either electronically with detailed GPS georeferencing of all sampling activities on the plot or just by drawing a simple map of the plot.

The number of sampling sites required to determine the soil moisture retention characteristics depends on the spatial soil variability of the monitoring site. For example, within the ICP Forests programme, at least three separate profiles are sampled. The location of these profiles within the plot may be chosen freely, as long as their spatial design meets the following requirements:

1. The individual profile pits are typical for the soil conditions within the plot.
2. The profiles are not located in one single profile pit (i.e., profiles are at least some meters apart).

3. The profiles should be situated as closely as possible to the installed soil moisture sensors (see Chapter 17) and lysimeters (see Chapter 16).
4. The exact coordinates of each profile location should be determined and logged for internal records.

Table 15.3 provides an overview on the soil sampling design of the main components for the large-scale plots and the intensive monitoring plots in the ICP Forests programme.

Figure 15.2 gives an example of the spatial sampling design of a typical, large-scale forest monitoring plot. Traditionally, for most plots four satellites of six trees each are assessed within the crown condition inventory (this design has been recently revised—see Chapter 6 for details). In each of these satellites, one sample of the organic layer, three samples of the mineral soil layers, and one set of undisturbed soil samples are taken. Additionally, a fifth organic layer sample and a fifth set of core samples are randomly chosen from among the four satellites.

15.2.2 Pedological Characterization

A pedological characterization consists of two main components: a site description and a soil profile characterization by its genetic horizons. Information in the site description will partially overlap with the site information that is recorded for other forest monitoring surveys, such as the plot coordinates and the elevation (m a.s.l.), the main tree species, and the tree species mixture. Additionally, it covers soil-related information such as the parent material, the depth of water table, and the effective rooting depth of the soil profile pit. The humus and soil profiles should be classified according to an internationally agreed classification system such as Zanella et al. (2011) for the humus forms and the World Reference Base for Soil Resources (IUSS Working Group WRB, 2007) for the soil types. When the WRB is the agreed system, it is recommended that the full name is reported, including all relevant qualifiers. The classification version applied needs to be correctly referenced, as soil taxonomic groups are prone to change.

The parent material is an important soil characteristic. It refers to unconsolidated organic and mineral materials in which the soil profile is formed. The underlying bedrock is the geological material, which should not be confused with the parent material.

The effective rooting depth of the profile can be restricted by a number of factors such as (i) the groundwater table, (ii) bedrock, either continuous or discontinuous rock preventing further digging, or (iii) cementation of any kind. When the required sampling depth cannot be reached, the underlying reason should be reported, as this is important when calculating element stocks. The depth of the profile is measured from the top of the mineral soil. According to the ICP Forests approach for conducting European forest soil surveys, this rule

TABLE 15.3 Overview of Sampling Designs on ICP Forests Large Scale (Level I) and Intensive Monitoring (Level II) Plots

Level	Sampling locations within plot area	Sampling design	No. of sampling points (=subsamples)	No. of soil layers per sampling point	No. of samples per layer and point
(1) Pedological characterization					
Level I	Representative for dominant soil type within the plot area	By expert judgment	≥1	=No. of horizons	≥1
Level II	Representative for dominant soil type within the plot area; can be in the buffer zone	By expert judgment	≥1	=No. of horizons	≥1
(2) Composite sampling at fixed depth					
Level I	Within the plot area	By expert judgment	≥5 (stony soils ≥3)	3–8	1
Level II	Within the plot area or in the buffer zone	Random (or systematic with random component)	≥24	5–8	1
(3) Sampling at fixed depth for soil bulk density					
Level I	Within the plot area	By expert judgment	1–5	1–5	1
Level II	Within the plot area or buffer zone	By expert judgment	5	3–5	1
(4) Sampling for soil water measurements					
Level II	Within the plot area	In vicinity of soil moisture probes	3	3–7	≥1

Source: Cools and De Vos (2010).

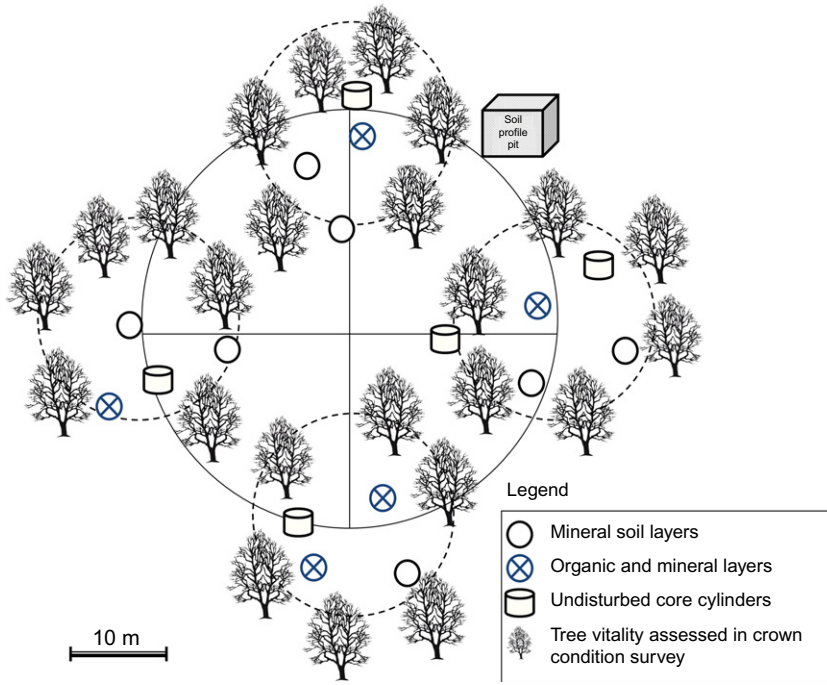

FIGURE 15.2 Example of soil sampling design on a large-scale monitoring plot. Reference is made to the cross-cluster plot design (see Chapter 6).

is maintained even when there are thick organic layers (up to 40 cm), in contrast to the Soil Survey Manual of the USDA (2004) or the FAO (2006), the latter having been developed primarily for agricultural land evaluation.

The detailed profile characterization is a description of the horizons/layers according to standardized field guidelines. We recommend referring to the ICP Forests Manual, Part X, for more specifically forest-orientated guidelines (Cools and De Vos, 2010). An overview of essential horizon characteristics is given in Table 15.4.

The profile description needs to be carried out only once. It provides all the necessary information for the soil classification. For practical reasons, it is not recommended to describe soil profiles during the winter season (in areas with snow cover), when temperatures fall below 0 °C or during periods with very shallow water tables. Describing the soil profile wall should be avoided when the (summer) sun causes big contrasts between illuminated and shaded parts of the wall.

The identification of soil horizons depends on, among other aspects, the required level of detail. While a more extensive survey might only delineate master horizons A, E, B, and C (and/or R), another one might split the

TABLE 15.4 Overview of Essential Horizon Characteristics for the Pedological Characterization of the Profile

Horizon characterization	Description and/or unit
Horizon number	Horizon sequence
Date laboratory analysis	Date of analysis
Horizon name	FAO (2006) definitions and symbols for master horizon, subordinate symbol, indication of discontinuity, and vertical subdivision
Upper and lower limit horizon	cm from mineral soil surface (negative for organic layer)
Horizon distinctness and topography	FAO (2006) definitions and symbols
Structure	FAO (2006) definitions and symbols
Moist and dry color of soil matrix	Description of hue, value, and chroma according to the Munsell soil color charts (Munsell, 2000), for example, 7.5 YR 3/4
Textural class	FAO (2006) definitions and symbols
Clay (0–2 μm fraction)	% of the fine earth fraction
Silt (2–63 μm fraction)	% of the fine earth fraction
Sand (63–2000 μm fraction)	% of the fine earth fraction
Coarse fragments	Volume% of the bulk soil
Total organic C content	g total organic C kg^{-1} mass of the dry fine earth
Total N	g total organic N kg^{-1} mass of the dry fine earth
Total $CaCO_3$	g $CaCO_3$ kg^{-1} mass of the dry fine earth (if present)
Gypsum content	g $CaSO_4 \cdot 2H_2O$ kg^{-1} mass of the dry fine earth (if present)
pH	With specification of the soil:volume ratio and extract
Electrical conductivity	dS m^{-1}
Exchangeable Ca, Mg, K, and Na	Base exchangeable cations ($cmol_{(+)}kg^{-1}$)
Cation exchange capacity	$cmol_{(+)}kg^{-1}$ (sum of the acid and base exchangeable cations)
Base saturation	% (proportion of sum of base exchangeable cations to the cation exchange capacity)
Porosity	Expressed as a percentage or coded classes (FAO, 2006)
Bulk density	kg m^{-3}
Root abundance and distribution	Expressed as abundance classes of (very) fine, medium, and coarse roots (FAO, 2006)

Source: Adapted from Cools and De Vos (2010).

same B horizon into several subhorizons, for example, B_t, B_{tg1}, and B_{tg2}. While the first subdivision may provide sufficient information on the large scale, research on intensive monitoring plots will benefit from the more detailed approach. Each described layer or horizon is sampled, with samples being taken to the laboratory for further chemical and physical analysis.

Horizon sampling is done with a blade (knife or trowel); the soil is gently loosened from the respective horizon and collected in a tray. Using the tray allows any material that is accidentally included in the sampled material to be easily removed before the material is transferred to a sampling bag. In general, it is best to sample after the horizons have been delineated and described. The deepest horizons should be sampled first to avoid contamination. As the main purpose of the profile pit sampling is to enable a correct soil classification, the "middle" sampling strategy is recommended. Here, a sample is taken from more or less the middle of the horizon, where the characteristics of the horizon are best developed.

As a general rule, and for taxonomic purposes, at least one sample per horizon should be taken. If a horizon is very heterogeneous, for example, due to strong mottling, it may be necessary to take several subsamples.

15.2.3 Composite Sampling at Fixed Depths

15.2.3.1 Sampling Time

While the pedological description is a one-time assessment, the composited fixed-depth samples are the core of the soil monitoring program. In order to minimize temporal changes due to seasonal variations, especially in the organic layer, sampling activities should be confined to periods with low biological activity, for example, winter or the dry season. Temporal repetitions of the sampling campaign should be carried out in the same season and sampling dates should always be recorded.

15.2.3.2 Organic Layer Sampling

Both the organic layer at the soil surface (i.e., the forest floor) and the underlying mineral soil are sampled. Buried organic layers are sampled in the same way as mineral layers. Care should be taken to correctly separate the organic layer from the mineral soil material (Bélanger and Van Rees, 2007; Federer, 1982). Separation is done in the field based on morphological properties (Zanella et al., 2011) but needs to be checked in the laboratory, following the internationally accepted criteria (FAO, 2006). Organic C determination in the laboratory must be used to check whether the separation in the field was done correctly. If the separation was not done correctly, a new sample must be taken. Organic and mineral layers should be sampled at exactly the same location. The mineral soil probes may be done at spots where the organic layer has already been removed for sampling.

A distinction has to be made between an organic layer that is water saturated and one that is not. The organic layer in aerated conditions may consist of one or more of the following organic subhorizons (Zanella et al., 2011): litter (OL), fragmentation horizon (OF) and/or humus (OH). In water saturated organic layers, a distinction is made between Hf (fibric), Hm (mesic), and Hs (sapric) horizons.

In the field, the total fresh mass of each organic (sub)layer (OL, OF, and OH, or Hf, Hm, and Hs) has to be determined, preferably together with the thickness of the layer concerned. The use of a metal frame to sample the forest floor is recommended. The frame is carefully pressed into the forest floor (Bélanger and Van Rees, 2007), and the organic subhorizons are cut separately along the frame using a sharp knife. Living material (such as mosses, roots, etc.) and objects exceeding 2 cm in diameter are removed from the sample, but smaller twigs and fruits remain to determine the mass of the sample. A subsample is collected from each layer for the determination of moisture content (mass%), which is in turn necessary to calculate the total dry mass (kg m^{-2}).

The description of the humus form should be carried out simultaneously with the sampling of the organic layer. Either all subsamples coming from within the frame or from the auger are taken individually to the laboratory to determine the dry mass (kg m^{-2}), or the subsamples are bulked and homogenized in the field, and a subsample is taken to the laboratory for further measurements. In the latter case, it is absolutely necessary that the fresh mass (kg m^{-2}) of each subsample and each organic subhorizon is measured in the field using an electronic field balance. The total surface of each subhorizon (surface of the frame or auger times the number of subsamples) must be recorded in order to allow later nutrient stock calculations.

15.2.3.3 Mineral Layer Sampling

Most forest health monitoring programs apply a fixed-depth sampling protocol (Cools and De Vos, 2010; EANET, 2000; Palmer et al., 2002), in which the top of the mineral soil corresponds to the zero reference level for depth measurements. The thickness of the depth layers depends on the objectives of the monitoring program. Since greater changes are expected in the topsoil compared to the deeper mineral soil, there is a tendency to sample the upper layers more intensively compared to the deeper ones. Within the ICP Forests, the depth intervals of 0–10 cm (or 0–5 and 5–10 cm separately), 10–20, 20–40, and 40–80 cm are sampled. Note that the entire thickness of the predefined depth needs to be sampled. For the sampling of mineral layers, augering is preferred in order to (i) guarantee a minimal disturbance of the monitoring plot and (ii) collect a large number of samples that adequately capture the variations in soil properties. Pits are possible as well, especially for stony soils where augering is difficult or impossible. Notwithstanding the fact that

horizon-based sampling is fundamental in pedogenetic studies, there are numerous reasons that favor fixed-depth sampling in forest soil monitoring:

1. The general sequence of A–E–B–C/R is not present in each profile. For example, below a mor humus, the A_h is often lacking; on the other hand, it can be well developed below a mull humus. A well-developed texture B horizon in a Luvisol might be very thick, while in young profiles (e.g., Regosols) the B horizon will be absent. This makes a transnational evaluation of forest soil properties by horizon very complicated.
2. Sampling at fixed depth is easier for field staff, especially when nonpedologists are employed, and facilitates repetitive sampling over time.
3. A large-scale survey involves many field crews. In the description of the soil profiles in the 22 countries that participated in the second European forest soil survey, the number of horizons described at each location varied from 1 to 13 (De Vos and Cools, 2011). Such "splitter" versus "lumper" approaches cause a big variation in the level of detail in profile descriptions (Boone et al., 1999).
4. In stock calculations, analysis of the full vertical soil is required. In this respect, it is important that the organic and mineral soil samples are taken from exactly the same location. When sampling by horizons, it is easy to tend to sample the central part of the horizon, discounting the transitional zones between horizons from the sample.
5. Preparing composite samples with subsamples of equal weight is easier when they are composed of fixed-depth samples. Within the spatial variability of a plot, one specific genetic horizon might be present in one sampling point, but might be completely missing at another sampling point.

If the upper edge of an indurated horizon (e.g., parent rock) is above the lower limit of the sampled soil, the soil is sampled down to the limiting horizon. For example, at a location where continuous rock starts at 65 cm below the soil surface, the 40–80 cm mineral layer will be sampled down to 65 cm. The upper limit of the indurated horizon or continuous rock is reported as the effective rooting depth in the soil profile description. The reason for a reduced sampling depth should be mentioned (e.g., physically impossible, choice made when designing the survey, etc.).

15.2.3.4 Sampling of Peat Layers

The proposed sampling design for peatlands provided below is based on the WRB definition of Histosols (IUSS Working Group WRB, 2007). As long as the thickness of a peat layer is less than 40 cm, the sampling design for mineral forest soils is applied, which implies separate sampling of the organic layers and mineral soil according to the fixed-depth layers. In the event that

the peat layer is 40 cm or thicker, the peat is sampled according to same fixed depths used for mineral soils. However, in order to distinguish them from the mineral soil layer, the peat layers should be coded differently. The required soil variables are the same as for the forest floor layers. If the conditions allow (lower water table), the mineral soil below the peat soil (>40 cm) can be further sampled down to a depth of 80 cm (where the 0 cm reference line remains at the top of the peat layer). The standard sampling depths should be followed as much as possible. It is important that the depth in centimeters is always reported along with the depth codes, for cross-checking.

15.2.3.5 Sampling of Stony Soils

Only the fine earth is sampled and analyzed. Therefore, an estimate of the volumetric stone content is to be made in the field. Additionally, a sample is to be taken to the laboratory for a more exact measurement of the volumetric or mass stone content. However, it should be noted that, as chemical analyses only concern the fine earth fractions and ignore nutrient content inside the gravel and stones, underestimations of C, and other nutrient stocks may be expected (Harrison et al., 2003).

15.2.3.6 Number of Subsamples in the Composite

If data are normally distributed, then the number of subsamples (n) for each layer that is necessary for a given level of precision can be determined by using Eq. (15.1) (Boone et al., 1999):

$$\mathrm{n} = \frac{\mathrm{t}^2\left(1-\frac{\alpha}{2};\mathrm{n}-1\right)\mathrm{CV}^2}{\mathrm{E}^2}, \tag{15.1}$$

where n is the number of subsamples per layer to be collected, t is the Student's t statistic appropriate for the level of confidence $(1-\alpha/2)$ and the number of samples minus 1 $(n-1)$ being collected, α is the significance level (e.g., 0.05), CV is the coefficient of variation (standard deviation divided by the mean), and E is the acceptable error as a proportion of the mean.

The number of subsamples in a composite sample depends on the forest soil layer that is being sampled and the variable that is being measured. This is illustrated in Table 15.5 for two intensive forest monitoring plots in Belgium, where 36 subsamples were collected and analyzed separately. By fixing the acceptable error at 10% of the true population mean with a 95% confidence interval ($\alpha = 0.05$), the minimum number of subsamples ranges from 7, in case of nitrogen (N), up to 83, in the case of organic carbon (OC) in the subsoil. It is therefore recommended to conduct a similar prestudy on a limited number of plots in order to set the required number of subsamples.

TABLE 15.5 Minimum Number of Subsamples Required to Obtain an Estimate of the True Mean with a Maximum Acceptable Error of 10% from the True Mean for the Analysis of Pb and P in the Forest Floor and for Organic Carbon and Kjeldahl Nitrogen in the Mineral Soil (CV, Coefficient of Variation)

	Zoniën forest—loess soil		Ravels forest—cover sand	
Organic layer	*Pb (ppm)*	*P (ppm)*	*Pb (ppm)*	*P (ppm)*
Mean	136.4	822.1	138.9	394.3
CV	21%	14%	26%	20%
Min. no. samples	18	8	27	17
	Zoniën forest - loess soil		**Ravels forest—cover sand**	
Mineral soil	*OC* ($g\ kg^{-1}$)	*Kjeldahl N* ($g\ kg^{-1}$)	*OC* ($g\ kg^{-1}$)	*Kjeldahl N* ($g\ kg^{-1}$)
	0–5 cm		**0–12 cm**	
Mean	53.3	3.0	31.1	0.86
CV	35%	30%	26%	31%
Min. no. samples	48	34	28	39
	5–8 cm		**12–25 cm**	
Mean	20.2	1.1	27.1	0.66
CV	35%	27%	43%	17%
Min. no. samples	52	30	76	13
	12–18 cm		**30–60 cm**	
Mean	9.8	0.6	8.6	0.33
CV	37%	22%	23%	21%
Min. no. samples	58	20	22	18
	31–97 cm		**60–100 cm**	
Mean	2.3	0.3	2.1	0.12
CV	45%	13%	29%	14%
Min. no. samples	83	7	36	8

However, within an international forest monitoring program it is not feasible to take 80 subsamples per soil layer in order to obtain this degree of precision. For example, within the ICP Forests programme, it was agreed to include a minimum of 24 subsamples for each composite sample at the intensive monitoring plots, down to a depth of 40 cm.

The subsamples must be of equal mass, except in situations with a variable lower-depth limit. In such a case (e.g., an indurated horizon within the depth range of the sampled layer), the mass of each subsample should be proportional to the thickness of the currently sampled layer. In the example mentioned above, the mass of the subsample taken should be equal to (65–40)/(80–40) of the standard sample mass.

15.2.3.7 Quantity of Samples

The minimum mass of each representative sample for chemical analysis should be large enough for all laboratory analyses, possible repetitions, and reanalyses over time. The absolute minimum mass of the composite samples (field mass) with no or little gravel should be 500 g, but 1 kg is recommended for important (reference) samples. Material not included in the composite sample can be used to refill bore holes or pits.

15.2.4 Soil Bulk Density and Coarse Fragments

15.2.4.1 Definition

The bulk density is defined as the mass of an oven-dried sample of undisturbed soil per unit of bulk volume. This volume includes both solids and pores. Since most biogeochemical processes are confined to the fine earth in between the coarse fragments (equivalent diameter >2 mm), the separate assessment of the bulk density of the fine earth is crucial. In mineral soils without coarse fragments, the bulk density of the total mineral soil is equal to the bulk density of the fine earth.

15.2.4.2 Measurement by the Core Method

The core method is applicable for stone-less or slightly stony soils. The samples should be taken vertically using steal core cylinders. This sampling procedure for undisturbed soil sampling is the same when samples are taken for soil water retention measurements and is as follows:

1. Soil cores are carefully taken to ensure minimal compaction and disturbance of the soil structure.
2. In a soil pit, undisturbed samples can be taken directly with hand pressure, using the sampling cylinder, without any additional equipment.
3. Alternatively, an open ring holder may be used. In such a holder, the ring is locked by means of a rubber or lever. On the outside of the ring, some space is left to allow for taking an oversized sample. This prevents sample compaction during sampling.

4. In hard soil layers, an impact-absorbing hammer may be used for hammering the ring holder into the soil.
5. When sampling in a borehole, a closed ring holder is recommended. This type of ring holder holds the cylinder in a cutting shoe. The ring is clamped inside the cutting shoe and no water or soil can penetrate into the ring from the top. Moreover, the sample ring is protected, the sample is oversized on both sides and there is no risk of losing or damaging the sample ring. In hard layers, an impact-absorbing hammer may be used with care.
6. The ring sample is taken vertically, with its cutting edge pointing downwards.
7. The cylinder should be dug out carefully with a trowel. If necessary, the sample should be adjusted within the cylinder before any uneven parts sticking out of the cylinder are trimmed. The two faces of the cylinder should be roughly trimmed using a small frame saw. A spatula or knife may be used, but care has to be taken to avoid smearing the surface (closing macro- and mesopores).
8. Both sides of the cylinders should be closed using suitable lids.
9. The following should be recorded: sampling date, sample plot reference and location within the plot, horizon encompassing the center of the core, and the exact sampling depths (depth of top and bottom of the cylinder with respect to the top of the mineral horizon).
10. The cylinder must be labeled clearly on the lid, with sampling date, sample plot reference, sampling location, horizon code, and sample depth.
11. The ring samples are to be wrapped in plastic bags or a plastic or aluminum foil to prevent drying.

15.2.4.3 Measurement by the Excavation Method

An alternative to the core sampling method for bulk density is sampling by the excavation method (ISO, 1993a). Sampling of bulk density in stony soils is a much more delicate procedure, and generally more time consuming than sampling in stone-less soils. First, a carefully leveled horizontal section is prepared. A soil volume is then excavated. The volume required depends on the general coarse fraction content. For example, if the coarse fraction makes up about 30% of the soil volume, a sample of 20 dm^3 should be sufficient. While excavating the sample, compaction of the sides should be avoided. The sample can be stored in a plastic bag. The excavation hole should be lined with a thin, strong, plastic film, and the hole filled to excess with a known volume of sand, using a funnel kept 5 cm above the ground. The surface is then leveled, avoiding compaction. The excess sand is removed into a graduated measuring cylinder, and the volume read. Finally, the total volume of sand used to fill the excavation hole is calculated.

15.2.4.4 Number of Samples

In the ICP Forests, at least five samples with a minimal volume of 100 cm^3 must be taken per plot and per depth layer. Based on a pilot study on monitoring plots with no or with very low stone content (Table 15.6), the expected error with five core samples is 16% (expressed as a percentage relative to the mean) in the upper 5 cm of the mineral soil and tends to decrease with depth.

Bulk density may also be estimated using pedo-transfer functions. In this case, regional calibration and validation are necessary. Information on how to determine the usefulness and predictive quality of bulk density pedo-transfer functions for forest soils can be found in De Vos et al. (2005).

15.2.4.5 Coarse Fragments Volume

A good estimation of the volume of the coarse fragments is essential when assessing element stocks (Eriksson and Holmgren, 1996; Throop et al., 2012). This is especially true for forest soils, which tend to be stonier, compared to agricultural land. The most accurate, though most labor-intensive, method is to dig a pit of sufficiently large volume, followed by sieving and volumetric measurement or weighing (and converting to volume by approximating the stone bulk density by the bulk density of granite of 2650 kg m^{-3}). An additional disadvantage is that this method is destructive and disturbs the monitoring plot (Eriksson and Holmgren, 1996). Therefore, it is recommended to assess the coarse fragment content while digging the soil profile pit for the pedological characterization. Within the ICP Forests, a visual estimation of the stones and boulders in the profile wall of the soil pit dug for pedological characterization is recommended. This method is acceptable

TABLE 15.6 The Acceptable Error as a Percentage of the Mean Bulk Density of the Soil Related to the Number of Soil Cores Calculated on Belgian Forest Monitoring Plots with Content of Coarse Fragments <5%

Number of test sites	67	10	9	14
Number of core samples	3	4	5	6
0–5 cm	31%	39%	16%	12%
5–10 cm	21%	22%	7.6%	8.9%
10–20 cm	18%	12%	6.9%	7.6%
20–40 cm	19%		7.7%	7.5%
40–80 cm	15%		9.7%	7.7%

for large stones (>76 mm) and boulders. However, it tends to underestimate the content of smaller stones (<76 mm) (Alexander, 1981; Baize and Jabiol, 1995).

A nondestructive method has been described by Viro (1952) and Eriksson and Holmgren (1996); it is often referred to as the "rod penetration method" or the "Finnish method." By pushing a graduated metal rod (diameter 1 cm) down through the organic layer and as far as possible into the mineral soil, the proportion (volume%) of coarse fragments larger than 2 cm in the upper 10, 20, or 30 cm of glacial till soil is estimated. Regional calibration (e.g., Rytter, 2012) of the soil function is indispensable to obtain a reasonable accuracy, as the relationship between the stones and boulders and the penetration depth is dependent on the geology (Stendahl et al., 2009).

15.2.4.6 Combined Approach for Bulk Density and Volume of Coarse Fragments

Bulk density should always be assessed in combination with the volume percentage of the coarse fragments. A combined field and laboratory approach can improve the determination of the bulk density and fine earth mass in stony soils, and lead to a better approximation of the real coarse fragments content. The field component consists of a volumetric estimation of the coarse fragments in the profile for each sampling depth and for each of the size classes: fine gravel (2–6 mm), medium gravel (6–20 mm), coarse gravel (20–60 mm), stones (60–200 mm), boulders (200–600 mm), and large boulders (>600 mm). The laboratory component entails the drying and weighing of the different disturbed and undisturbed samples. The appropriate procedure should be selected according to the prevailing conditions (i.e., coarse fragment content and size). Detailed descriptions and calculation formulas are given in König et al. (2009) (English summary in the ICP Forests soil manual, Cools and De Vos, 2010).

15.2.5 Sampling for Soil Water Retention Characteristic Measurements

If soil moisture content is monitored on the plot, undisturbed core samples should be taken to determine the soil water retention characteristic (SWRC) (pF). The same core samples can be used to measure the bulk density. The samples should be taken when the soil is near field capacity, which is often toward the end of winter. Soil should not be sampled when frozen. Ideally, the undisturbed cores should be collected at the time of the installation of the soil moisture probes to assure (i) minimal soil disturbance and (ii) that the cores are taken from the same layer and horizon where the soil moisture sensors are installed. The exact depth range of the soil core (top to bottom

of core) should be reported, along with the ring ID information and the name of the pedogenetic horizon containing the center of the sampling cylinder.

15.2.6 Sampling Equipment

Detailed lists of field equipment for profile description, sampling at fixed depth, and sampling of undisturbed core samples are provided by Cools and De Vos (2010). It is recommended to sample the organic layer with a 25×25 cm frame, but alternatives with a minimum total surface of 500 cm^2 are acceptable; for mor humus, an auger with a diameter of 8 cm can be used. Sampling of the organic layer can be done by hand, supported by a trowel, knife, spatula, and/or brush. For sampling of the mineral soil by auger, different types of soil augers are recommended, depending on the soil texture and moisture conditions. Undisturbed soil cores are collected using dedicated steal cylinders (sleeves), with a volume between 100 and 400 cm^3. The same steel cylinders can be used for the soil water measurements and for determining bulk density. The sample ring dimensions should be appropriate for representing the natural soil variability and structure. The most frequent dimensions of cylinders for forest soil sampling are (height $\times$ diameter in mm): 53×50 (100 cm^3), 40.6×56 (100 cm^3), and 50×79.8 (250 cm^3). It is important to verify that the laboratory processing the undisturbed samples is equipped for the type of sample rings used. The bottom of the sample rings should have a cutting edge. Plastic lids should fit perfectly to both ends of the steel cylinder.

15.2.7 Sample Packing and Transport

It is essential that the sample recipient is properly labeled with a comprehensive code that includes location name, plot number, profile number, horizon number or layer name, depth of sample, and sampling date. In the field, samples (whether in bags, boxes, metal rings, or other storage containers) should never be left exposed to the open air or sun. This would cause water to evaporate from the sample and condense in the bag or recipient, with a risk of ultradesiccation. The warming-up of the sample would also activate the biological activity within it. Samples for standard soil laboratory analyses are mostly kept in either plastic bags or boxes that can be sealed and labeled properly. In the laboratory, the content of the bags should be transferred into open (plastic) boxes for drying. Double bags may be required to reduce the possibility of tears or punctures when samples contain a large number of rocks, sticks, or other sharp objects.

The undisturbed core samples are transported in plastic boxes or aluminum cases, protecting the samples from heat, humidity, and dust. If transported in vehicles over long distances, shockproof materials should be used to avoid shocks to the samples. Undisturbed soil samples need to be protected from

freezing. Samples should be stored at 1–2 °C to reduce water loss and to suppress biological activity until analysis. It is recommended to avoid long storage (e.g., 1 week) of undisturbed soil samples. Ideally, these samples are analyzed in the laboratory immediately after sampling.

15.3 LABORATORY MEASUREMENTS

15.3.1 Sample Preparation

Collected samples should be transported to the laboratory as soon as possible and should then be air-dried or dried at a temperature of 40 °C (ISO 11464) (ISO, 1994c) and stored until analysis. To recalculate the analysis results on mass basis, the moisture content of the sample is determined by oven drying a subsample once at 105 °C (ISO 11465) (ISO, 1993a). All analytical results expressed on mass basis need to be corrected by applying a moisture correction factor (MCF):

$$\mathrm{MCF} = \frac{100 + \text{moisture content}(\%)}{100}. \tag{15.2}$$

Living macroscopic roots and all particles (mineral and organic) with a diameter exceeding 2 mm should be removed from the samples by dry sieving. Roots and organic matter particles less than 2 mm in diameter can be left in the sample, unless it is explicitly stated otherwise in the experiment design. Mineral particles that do not pass through the 2 mm sieve are weighed separately for the determination of the coarse fragments content. To guarantee a harmonized approach, samples should not be further milled or ground. However, for the total analyses for which finely ground material is required (e.g., carbonate, total OC, total N) further milling or grinding is allowed.

15.3.2 Selection of Key Soil Analytical Variables

Within a forest monitoring program, two sets of laboratory soil variables can be distinguished. A first set of soil variables concerns the characterization of the soil and should only be measured once, at the time the plot is installed (Table 15.4). A second set of soil variables is monitored regularly, that is, repeated measurements over time. This second set of variables depends on the objectives of the monitoring program. For the assessment of the effects of transboundary air pollution, it is essential to monitor soil pH, exchangeable elements (Ca, K, Mg, Na, Al, Fe, Mn, and free H^+), total N, and macronutrients (aqua regia extractable P, Ca, Mg, K, Mn, and S). In order to arrive at a compromise between what is scientifically required and what is feasible in an international program—including in financial terms—a distinction can be made between top priority and secondary priority variables and layers. As temporal changes are—in the first place—expected in the upper layers,

primarily in the chemical soil variables, a reassessment of the physical soil variables in the mineral layers deeper than 20 cm is not considered necessary. In order to be able to calculate C stocks and assess their changes over time down to a reference depth of 30 cm, the ICP Forests programme included a repetition of C and bulk density measurements in the 20–40 cm layer.

For the determination of pH, measurement in a $CaCl_2$ extract is of higher priority than measurement in water, although the latter can be useful for comparison with results in literature. The nutrients, extracted using aqua regia, are of major importance for the organic layers, and less essential for the mineral soil. This extraction method results in a semitotal, ecologically relevant concentration, which can be useful as an estimate of the nutrient stocks. Additional costs and work are minimal, as these nutrients can be measured during the same extraction as the trace metals. For the determination of the "real" total amounts, with the complete digestion of the soil matrix (i.e., using hydrofluoric acid) required for studying weathering rates and other soil genetic processes, more specialized materials and skills are required, and not routinely applied in a transnational context.

15.3.3 Physical Soil Variables

The laboratory determination of the soil granulometry according to the sieving and sedimentation method, more specifically the pipette method (ISO 11277) (ISO, 1998c) and classification according to the USDA–FAO textural classes (FAO, 1990), is the most widely agreed reference method. The textural classes of the fine earth fraction (<2 mm) are based on the particle-size fractions of clay, silt, and sand, which follow the limits of 2, 63, and 2000 μm equivalent spherical diameter. Alternatively, although it is not recommended, estimations of the particle-size fraction can be based on an in-field finger test on one composite of each layer, together with an estimate of the clay content (FAO, 2006). Repetition of the determination of the granulometry during follow-up monitoring surveys is not required.

The soil cores taken in the field for bulk density measurements are placed in their holders in an oven at 105 °C until constant mass is reached and weighed on a balance according ISO method 11272 (ISO, 1993a).

Although field methods are recommended, alternatively the coarse fragment volume can be measured in the laboratory. During the preparation of the soil samples for chemical analyses, the coarse fragments are separated from the fine earth fraction. The mass of coarse fragments is determined by weighing the residue on a 2 mm sieve after washing and drying. In order to convert the mass percentage into a volume percentage, the bulk density of the coarse fragments and of the total soil should be known.

In order to determine the SWRC, the volumetric water content (θ in proportional volume, $m^3\ m^{-3}$) is determined at predefined matric potentials

TABLE 15.7 Overview of Matric Heads Used for the Determination of the Soil Water Retention Characteristic with Recommended Instruments

Matric potential Ψ			Recommended instrument	Estimator
cm H_2O	*pF*	*kPa*		
1	Infinitely small	0	Pycnometer	$\approx\theta$ sat = water-holding capacity = total porosity
10	1.0	−1	Sand suction table	
51	1.7	−5		
102	2.0	−10		Field capacity sand
337	2.5	−33	Kaolin suction table	Field capacity silt loam
1022	3.0	−100	Pressure plate extractor or pressure membrane cells	Field capacity clay
2555	3.4	−250		
15330	4.2	−1500		Permanent wilting point
10^7	7.0	-10^6	Oven	Dry bulk density

Source: Cools and De Vos (2010).

(Ψ in kPa). It is recommended to measure and report the water content at nine matric heads per plot (Table 15.7).

Some matric heads immediately provide information on the SWRC variables. At 0 kPa, the maximum water-holding capacity of the saturated soil sample is determined. Depending on definitions and soil texture, field capacity may be inferred from −10 to −100 kPa. The permanent wilting point is attained at a matric pressure of −1500 kPa. The dry bulk density (lowest pressure at about -10^6 kPa) is calculated based on the oven dry mass at 105 °C. The standard instruments required for each determination are listed in Table 15.7. The reference methods used within the ICP Forests programme for all physical variables are also listed in Table 15.8.

15.3.4 Chemical Soil Variables

Table 15.9 gives an overview of a set of recommended reference methods for the laboratory analysis of chemical soil variables in forest soil monitoring. The variables are grouped according to the analytical method. Thus, elements are grouped which can be measured in the same run, without additional costs

TABLE 15.8 Overview of Reference Methods Used for the Determination of Physical Soil Variables

Variable	Reference method	Reference	Unit
Soil moisture content	Determination of dry matter and water content on a mass basis (gravimetrically)	ISO 11465 (ISO, 1993b)	Mass%
Particle-size distribution (sand, silt, clay fractions)	Sieving and sedimentation method (pipette method)	ISO 11277 (ISO, 1998c)	%
	Finger test method	FAO (2006)	
Coarse fragments	Laboratory measurement	ISO 11277 (ISO, 1998c)	Volume%
	Field estimate during soil profile description	FAO (2006)	
Soil water retention characteristic	0 kPa: Pycnometer measurement −1 till −10 kPa: Sand suction table −33 kPa: Kaolin suction table −100 till −1500 kPa: Pressure plate extractor or pressure membrane cells	ISO 11274 (ISO, 1998b)	$m^3\ m^{-3}$
Bulk density Bulk density in stony soil	Oven drying at 105 °C	ISO 11272 (ISO, 1993a) König et al. (2009)	$kg\ m^{-3}$
Volume dry mass of organic layer	(1) Field measurement of total fresh mass (2) Field measurement of the horizon thickness (3) Determination of moisture content in the laboratory		(1) $kg\ m^{-2}$ (2) cm (3) Mass%

Source: Adapted from Cools and De Vos (2010).

and with almost no extra work involved. A number of variables can be determined using alternative methods.

The pH of the mineral and the organic soil samples, including peat samples, is potentiometrically measured in a supernatant suspension of 1:5 (volume fraction). The liquid is made up of a 0.01 mol L^{-1} solution of calcium chloride in water for pH ($CaCl_2$) or deionized water for pH (H_2O).

Carbonates are measured after treating the soil sample with hydrochloric acid (4 mol L^{-1}). The volume of the CO_2 produced is measured by using a calcimeter (Scheibler unit) and is compared with the volume of CO_2 produced by pure calcium carbonate.

TABLE 15.9 Example of Reference Methods for Chemical Soil Variables

Variable		Reference analytical method				
		ISO reference	*Extractant*	*Measurement method(s)*		
pH ($CaCl_2$)		ISO 10390 (ISO, 2005)	0.01 mol L^{-1} $CaCl_2$	pH-electrode		
pH (H_2O)			H_2O	pH-electrode		
Carbonates		ISO 10693 (ISO, 1994a)	HCl	Calcimeter		
Total organic C		ISO 10694 (ISO, 1995a)	–	Dry combustion at $\geq$ 900 °C		
Total N		ISO 13878 (ISO, 1998d)	–	Dry combustion		
		ISO 11261 (ISO, 1995b)	–	Modified Kjeldahl		
Free acidity (or sum of acid cations Al, Fe, Mn, and free H^+)		ISO 14254 (ISO, 1994d)	0.1 mol L^{-1} $BaCl_2$	titration to pH 7.8 or "German" method (König et al., 2009)		
Exchangeable cations	Al, Fe, Mn	ISO 11260 (ISO, 1994b)	0.1 mol L^{-1} $BaCl_2$	ICP	AAS	–
	K, Ca, Mg, Na					FES
P		ISO 11466 (ISO, 1995c) ISO 11047 (ISO, 1998a)	Aqua regia by reflux digestion	ICP	Colorimetry	
K, Ca, Mg, and Mn					AAS	
Heavy metals: Cu, Cd, Pb, and Zn						
Other: Al, Fe, Cr, Ni, and Na						
Hg				ICP	Cold vapor AAS	
S				ICP		
		ISO 15178 (ISO, 2000)	CNS analyzer			
Reactive Fe and Al		ISRIC (2002)	Acid ammonium oxalate	AAS	ICP	

AAS, atomic absorption spectrometer; FES, flame emission spectrometer; ICP, inductively coupled plasma spectrometer.
Source: Adapted from Cools and De Vos (2010).

To determine the total OC, the C present in the soil is oxidized to CO_2 by heating the soil to at least 900 °C in a flow of oxygen-containing gas that is free from CO_2. The amount of CO_2 released is then measured using titrimetry, gravimetry, conductometry, gas chromatography, or an infrared detection method, depending on the total analyzer used. When the soil is heated to a temperature of at least 900 °C, most carbonates present in the sample are completely decomposed. Total organic C can be determined directly or indirectly. Direct determination consists of the previous removal of any carbonates present by treating the soil with hydrochloric acid (HCl). Indirect determination consists of a correction of the total C content for the carbonates (inorganic C) present.

Total N is determined by dry combustion when heating the sample to a temperature of at least 900 °C in the presence of oxygen gas. Mineral and organic N compounds are oxidized and/or volatized. The combustion products are oxides of nitrogen (NO_x) and molecular nitrogen (N_2). After transforming all N forms into N_2, the content of total N is measured using thermal conductivity. In the modified Kjeldahl method, total N is measured by Kjeldahl digestion; but instead of selenium (Se), titanium dioxide (TiO_2) is used as the catalyst.

To determine the exchangeable cations, the soil is saturated with barium (Ba) by treating the sample with a 0.1 mol L^{-1} barium chloride ($BaCl_2$) solution. Concentrations of the exchangeable basic cations sodium (Na), potassium (K), calcium (Ca), and magnesium (Mg), and the exchangeable acid cations iron (Fe), manganese (Mn), and aluminum (Al) are determined in the 0.1 mol L^{-1} barium chloride soil extract using spectrometry (ISO 11260 and ISO 14254) (ISO, 1994b,d).

To measure the exchangeable acidity, the 0.1 mol L^{-1} extract is titrated with a 0.05 mol L^{-1} NaOH solution up to pH 7.8. Determination of the free H^+ acidity is carried out by adding sodium fluoride to the soil extract prior to titration. The aluminum ions are complexed, and only the free H^+ acidity is detected during the titration process. Alternatively, the free H^+ acidity can be determined using the "German calculation method" based on the pH of the $BaCl_2$ solution before and after extraction (König et al., 2009). The exchangeable acidity is subsequently calculated based on the sum of the acid cations and the free H^+.

The aqua regia extractable elements are measured after extracting the dried sample with a hydrochloric/nitric acid (HCl/HNO_3) (3:1 volume fraction) mixture by standing for 16 h at room temperature, followed by boiling under reflux for 2 h. The extract is then clarified and filled up to the required volume with HNO_3. Elements are determined using spectrometry.

In the acid ammonium oxalate extraction of Fe and Al, the sample is shaken with this complexing solution, thereby dissolving the "active" or "amorphous" Fe, Al (and Si) compounds, and a (variable) amount of organically complexed Fe and Al, which is determined in the extract by means of an atomic absorption or an inductive coupled plasma spectrometer (ISRIC, 2002).

15.3.5 Quality Control

It is essential that the quality of the laboratory analytical results is controlled. Chapter 22 provides a full account of the Quality Assurance (QA) and Quality Control (QC) measures undertaken within the framework of ICP Forests.

15.3.6 Long-Term Storage of Soil Samples

It is very useful to have a sample archive: for future reanalysis and to rule out the possible role of shifts in analytical techniques in determining artifacts, for example, trends in concentration levels (Mol et al., 1998). The samples should be stored at least until the next campaign. The sample material for long-term storage should be kept without preservative under normal room conditions with minimal temperature and humidity fluctuations, shielded from incidental light. If the humidity in the storage room cannot be controlled, dried soil samples should be kept in airtight containers. In addition to storing the (composite) samples for the monitoring program, it is also recommended to store the samples of the genetic horizons of the soil profile pit description, as soil classification systems are also prone to change and development. Soil core sample material can be disposed off after the measurement of the soil moisture retention characteristics and the bulk density, as it is no longer suitable for chemical analysis. Most changes in the chemical properties of air-dried, archived soil samples are smaller than those caused by management, atmospheric, and other inputs (Blake et al., 2000). However, a number of studies show that pH in samples stored at room temperature tends to decrease over time, especially when measured in water (Falkengren-Grerup, 1995; Ogner et al., 2001). The constant progress of analytical methods and technical innovation, and a lack of cross-checking with standard samples after changes in techniques, remain major problems, which only can be solved through storage and reassessment of samples. Therefore, any investigation reporting time changes in, for example, soil chemistry should be able to demonstrate continuity of techniques and/or clear procedures in order to monitor and account for changes caused by new analytical techniques.

15.4 DATA COMPILATION AND VALIDATION

After the completion of the laboratory analyses, all field and laboratory data need to be compiled within one database. Once brought together, cross-checks should be performed between the field and the laboratory data. For example, in reference to the soil classification, Arenosols are defined as soils with the weighted average texture of loamy sand or coarser (IUSS Working Group WRB, 2007). The textural triangle in Figure 15.3 shows the reported textures of more than 500 profiles classified as Arenosols in the ICP Forests large-scale plots, where indeed sand and loamy sand are found to be the dominant textural classes.

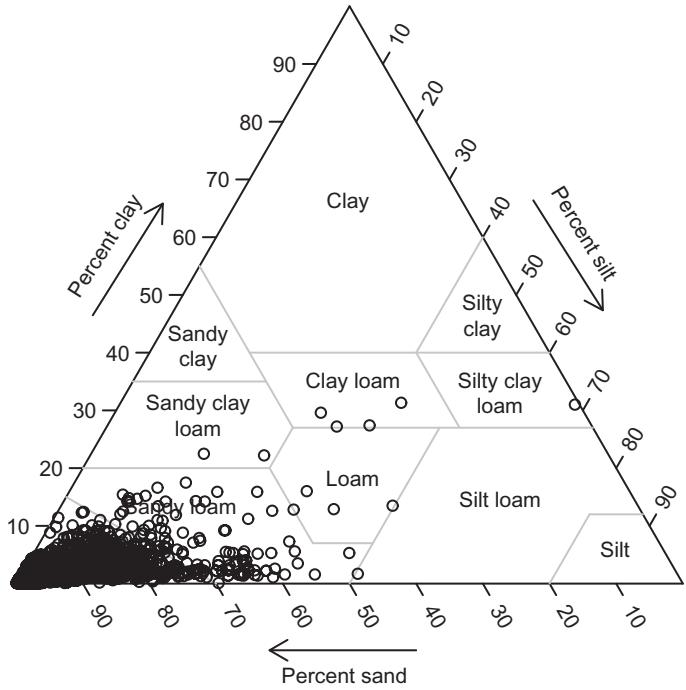

FIGURE 15.3 Reported textures of more than 500 profiles classified as Arenosols on the ICP Forests large-scale monitoring plots. *Source: De Vos and Cools (2011).*

15.5 SUBMISSION OF THE DATA TO AND STORAGE IN THE CENTRAL DATABASE

In transnational surveys, all validated data are collected primarily at the national level, and then stored in a central database. Submission to the central database needs to be coordinated, and detailed time schedules should be provided by the relevant bodies (see Chapter 23). Predefined formats are required for data reporting. Examples of such data submission forms can be found on the ICP Forests website (www.icp-forests.net). Soil data are best reported using separate forms, for example, one for the site description, one for the profile horizon description, and one for the composite samples analyses. Furthermore, the data structure should make it possible to discern between analytical results originating from individual subsamples versus composite samples, or from mean results from the measurement of a number of individual samples.

Data should be accompanied by information on the field and laboratory methods and the quality of the reported data (metadata). Data quality forms should include all details on sampling and analytical procedures in an easily accessible (digital) format. In addition, irregularities in sampling and analytical procedures, missing data, estimated values, and errors encountered during validation, should be documented as well. Separate forms may be needed for field and laboratory methods.

All details on how data were treated and how calculations were made must be documented and related information linked to the results. If values are below the quantification limit (not the detection limit), this should be mentioned explicitly, together with the limit, which may vary among matrices (forest floor, peat, or mineral soils), surveys, and laboratories. Such metadata are essential, as data from international monitoring programs are often used by many external researchers and modelers, who are not necessarily familiar with the applied sampling and analysis methods.

For the sake of harmonization, it is preferable that soil characteristics derived by computation or classification are generated from the individual soil properties based on the centralized data, rather than computed and directly reported by the individual partners of the monitoring program. Examples of derived chemical soil properties include the cation exchange capacity, base saturation, C:N, and C:P ratios. Typical examples of derived physical soil characteristics are the available water capacity, field capacity, wilting point, and total porosity, which are derived from the SWRC.

15.6 DATA EVALUATION

The following figures present examples of results based on data generated by the ICP Forests. Figure 15.4 shows the change in forest soil pH ($CaCl_2$) between the first (Vanmechelen et al., 1997) and the second (De Vos and Cools, 2011) forest soil condition surveys of more than 2000 common

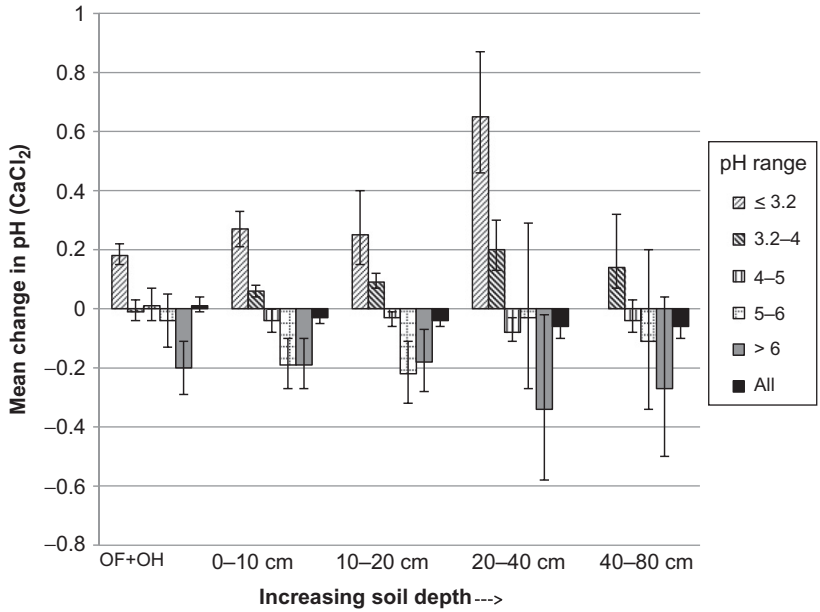

FIGURE 15.4 Mean change in soil pH ($CaCl_2$) between two forest soil inventories on more than 2000 large-scale monitoring plots of ICP Forests.

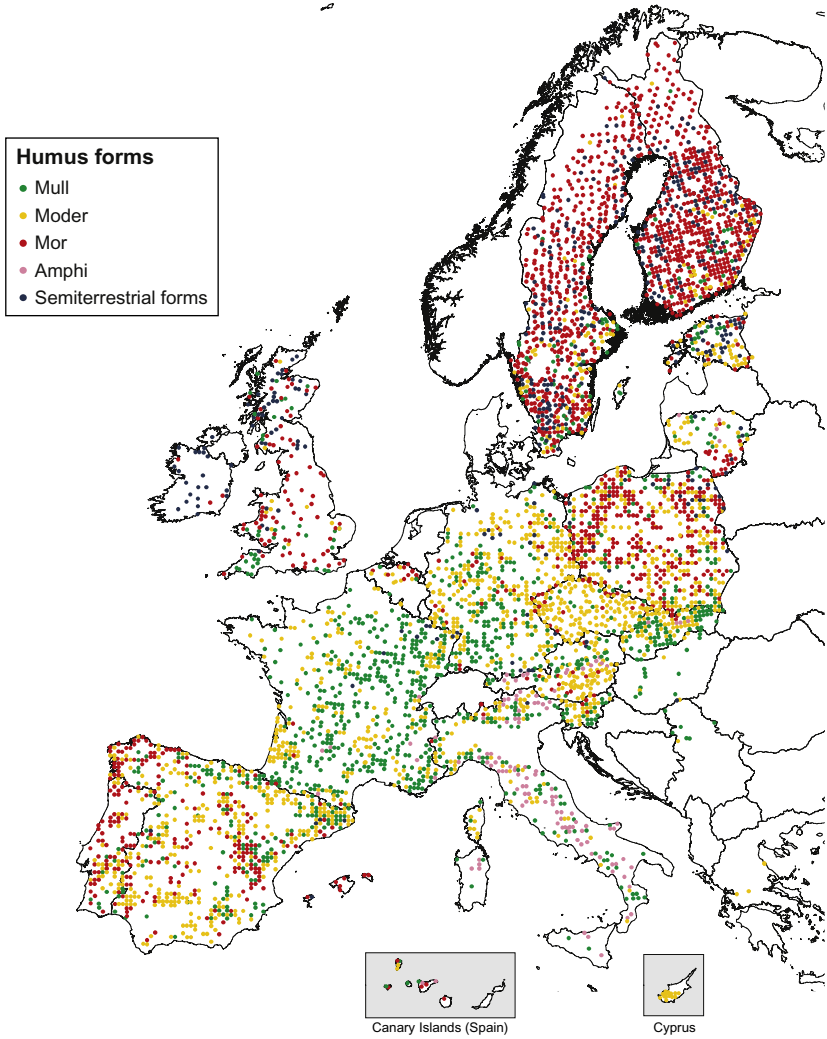

FIGURE 15.5 Humus forms on 4807 large-scale plots of the ICP Forests network (survey period 2004–2008). *Source: Adapted from De Vos and Cools (2011).*

European plots, in the forest floor and in the mineral soil layers down to 80 cm, by pairwise comparisons between identical plots in both surveys. Positive changes indicate an increase in soil pH, while negative changes indicate a decrease. Error bars represent the 95% confidence intervals of the bootstrapped mean differences for each pH class (<3.2, 3.2–4, 4–5, 5–6, and >6). Changes are statistically significant when the error bars do not cross the 0 line. The results show that the pH in all layers increased in the lowest

pH ranges (below 4.0) but decreased when the pH was above 4. The changes are most explicit and statistically significant in the upper 20 cm of the mineral soil. These results seem to show a recovery after acidification in the most acidic forest soils, in contrast to a further acidification in forest soils with pH ($CaCl_2$) above 4.0. The results, however, indicate the need for more in-depth investigations to fully understand these changes.

Figure 15.5 maps the humus forms in 4807 ICP Forests network plots (survey period 2004–2008). Mor is the most dominant humus forms, not only in northern Europe but also in the Iberian peninsula. Moder and mull humus forms are found throughout the whole of Europe. Amphi humus is a typical Mediterranean humus form. Semiterrestrial humus forms are mainly confined to northern Europe. Traditionally, it has been assumed that mor humus forms generally occur in cold, moist climates under temperate, and boreal vegetation. Recent data show a high frequency of mor humus forms in Portugal and Spain, as well. Such mapping exercises provide a better understanding not only of the geographic distribution of the main humus forms across Europe, but of the various chemical and physical soil properties using common definitions as well. Harmonized soil data provide a basis for cause–effect studies to analyze the impact of environmental changes to forest ecosystem across the continent.

ACKNOWLEDGMENTS

This work is mainly based on the work done by the ICP Forests Expert Panel on Soil and Soil Solution and on Part X "*Sampling and Analysis of Soil*" of the ICP Forests Manual. We thank all participants in the Expert Panel for their active discussions and contributions, which created a fertile ground for this chapter.

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