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MANUAL

on

methods and criteria for harmonized sampling, assessment,
monitoring and analysis of the effects of air pollution on forests

Part X

Sampling and Analysis of Soil

Version 2025-1

Prepared by:

ICP Forests Forest Soil Co-ordinating Centre and the Expert Panel on Soil and Soil Solution

(Nathalie Cools and Bruno De Vos)

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Application for such permission should be addressed to:

Programme Co-ordinating Centre of ICP Forests
Thünen Institute of Forests Ecosystems
Alfred-Möller-Str. 1, Haus 42/43
16225 Eberswalde
Germany

Email: pcc-icpforests@thuenen.de

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1 Objectives

This Part of the Manual outlines the sampling, analysis and reporting procedures for the set of soil parameters measured in the ICP Forests programme. Investigating forest soils is important on both Level I and Level II plots of the monitoring scheme.

The purpose of the large-scale soil survey (Level I) is first of all the assessment of basic information on the chemical soil status and its changes over time, and secondly the assessment of soil properties which determine the forest soil's sensitivity to air pollution (e.g. acidification status). Besides providing soil data for the study of atmospheric deposition effects at the broader scale, the soil survey will serve other purposes, as supporting studies related to climate change (e.g. inventory of carbon storage) and sustainable forest management (e.g. nutrient and water balances studies).

A third major objective of the large-scale representative soil survey (Level I) is to allow the evaluation of the forest soil condition across Europe. For the sake of data comparability among countries, a prerequisite is that the same methods for soil sampling and analysis are used throughout the network. As such, analytical results obtained by national methods, different from those described in this manual, cannot directly be compared with analytical results obtained by the international reference methods of this manual. Notwithstanding, the participating countries are encouraged to make efforts (where necessary and possible) to allow the comparison over time of the data obtained over the different surveys.

The intensive soil studies are conducted on permanent plots (Level II) where other measurements and assessments for the analysis of the forest ecosystem are performed. Intensive soil measurements are essential in understanding the role of forest soils in cause-effect relationships and in ecosystem functions and services. The intensive soil study involves the soil characterisation, the evaluation of the soil condition and the study of the soil processes and dynamics on the long-term. Methods for the short-term soil dynamics are described in the Part XI on Soil Solution Collection and Analysis and partly in Part IX on Meteorological Measurements (soil temperature and soil water dynamics).

2 Scope and application

Soil analyses are relevant to many environmental applications such as studies on acidification, eutrophication, C stock assessment, nutrient fluxes, water balances, biodiversity assessments and impact of climate change. This Part presents all the soil related field and laboratory parameters that are required for these studies within the ICP Forests programme. Concerning the field observations and sampling, the aim is to provide a set of minimum requirements which need to be met to come to a harmonised approach. Related to the analyses in the laboratory, all laboratories have to use the reference methods, which mainly follow ISO standards.

The relevance of the key soil parameters is given in Table 1. Table 2 provides an overview on the mandatory and optional soil surveys. An overview on the mandatory and optional parameters and sampling depths is given in Table 11 for the set of soil physical and soil chemical parameters and in Table 7 and 9 for the soil moisture measurements.

Table 1: Relevance of the key soil parameters

Type of parameter	Key parameters	Layer	Relevance
Carbon and nitrogen	C _{tot} , N _{tot} , (Carbonates)	Organic	Forest nutrition, atmospheric N deposition, climate change
		Mineral	Forest nutrition (0-20 cm), C- & N stocks (0-80 cm)
Nutrients	P, Ca, Mg, K, S, Mn	Organic	Atmospheric deposition of basic cations, stock of macronutrients
		Mineral	Weathering rates, critical loads of acidity, stock of macronutrients
Acidity, Exchange characteristics	pH, Carbonates, CEC, BS, Exchangeable cations, Exchangeable Acidity	Organic	Buffering acid input, acidification status
	pH, Carbonates, CEC, BS, Exchangeable cations, Exchangeable Acidity, Al _{ox} , Fe _{ox}	Mineral	
Heavy metals	Pb, Cu, Zn, Cd, Cr, Ni, Hg	Organic	Atmospheric metal deposition
		Mineral	Atmospheric metal deposition, calculation critical loads (0-20 cm), deficiency of oligo elements
Physical soil parameters	Particle size distribution and soil texture	Mineral	Profile description and soil classification, estimation of plant available water, nutrient exchange capacity
	Organic layer mass	Organic	Calculation of stocks
	Bulk density of the fine earth (BD _{fe}) and the coarse fragment content	Mineral	Calculation of stocks, nutrient supply to plants, index for compaction
	Soil Water Retention Characteristic (SWRC)	Organic Mineral	Water balance models, nutrient fluxes, estimation of soil porosity

Table 2: Overview of soil survey at the Level I, II and the Level II core plots

Soil survey	Level I	Level II	Level II core
Pedological characterisation	Mandatory (once at installation of plot)		
Soil sampling at fixed depths	Mandatory (every 10 - 20 years) *		
Soil sampling for bulk density at fixed depths	Mandatory		
Sampling for measurement of SWRC	Optional	Optional	Mandatory

* Pan-European synchronisation within a period of 3 years is essential

3 Objectives

This Manual is designed to provide a consistent methodology to collect high quality, harmonised and comparable forest soil data across Europe. This will allow (i) the proper characterization and description of the soil condition; and (ii) to monitor changes in soil properties periodically (e.g. on a 10 years basis).

The soil survey comprises three main pillars (Table 2):

1 Pedological characterisation. At the plot installation a detailed soil profile pit description complemented by sampling according to genetic horizons should lead to a detailed soil classification following the World Reference Base for Soil Resources (IUSS Working Group

WRB, 2022). WRB classifies the soil based on a vertical description of the profile wall starting below the litter layer (OL) up to a depth of 2 m.

2 Monitoring of the soil condition. Both the organic and the mineral soil layers are sampled and analysed in the laboratory at regular time intervals (e.g. every 10 years). For this purpose, composite samples are taken at fixed depth layers.

3 Determination of the soil water retention characteristic (SWRC). The assessment of the forest water budget is essential to study the nutrient fluxes in the forest ecosystem on the permanent monitoring plots. For the parameterisation of various water balance models meteorological data, stand characteristics and soil physical data are essential. For the validation of the models soil temperature, soil moisture and stand precipitation measurements are needed. To characterise the soil water retention, a series of undisturbed soil samples need to be taken and analysed in the laboratory. This survey is mandatory on all (core) plots where water budgets are assessed.

4 Location of measurements and sampling

4.1 Sampling design at plot level

Table 3 provides an overview of the sampling design on the Level I and Level II plots.

Table 3: Overview of sampling design on the Level I and Level II plots

Objective	Location of sampling regards the plot area	Sampling design	N° of sampling points	N° of soil layers per point	N° of soil samples per layer and point
Pedological characterization					
Level I	Representative for dominant soil type within the plot area	Judgemental	≥1	= N° of horizons	≥1
Level II	Buffer zone	Judgemental	≥1	= N° of horizons	≥1
Soil sampling at fixed depth					
Level I	Sampling sites should be located within the plot area.	Judgemental	≥ 5 (but on stony soils for optional depth layers ≥ 3)	3 to 8	1
Level II	Sampling sites should be located within the plot area or if not feasible, in the buffer zone of the plot.	Random design or systematic design with a random component.	≥24	5 to 8	1
Sampling at fixed depth for soil bulk density					
Level I	Not specified	Not specified	0 to 5	0 to 5	0 to 1
Level II	Not specified	Not specified	≥ 5	3 to 5	1
Sampling for soil water measurements					
Level II core	Within the plot	In vicinity of field soil moisture probes	3	3 to 7	≥1

4.1.1 Pedological characterization of the plot

4.1.1.1 Allocation of the soil sampling sites

The pedological characterization:

- Is mandatory for Level I and Level II plots but has to be carried out only once;
- Includes a detailed profile characterisation with information on soil parent material and at least one profile description with characterisation by horizons according to the Field Guide in Annex 1 of IUSS Working Group WRB (2022). The soils should then be classified according to the most recent official version of the World Reference Base of Soil Resources (WRB)-classification system. It is recommended to report all qualifiers. In addition, the correct reference needs to be made to the applied WRB reference system. An overview of the mandatory and optional parameters for the pedological characterisation is given in Table 4.
- Includes the identification of the dominant humus form on the observation plot according to the standardized morpho-functional classification of Zanella *et al.* (2022) up to the first hierarchical level. This is the distinction between Mull, Moder, Mor, Amphi, Tangel, Fibrimoor, Mesimoor, Amphimoor, Saprimoor and Anmoor.
- The described soil profile(s) should be located at locations representative for the dominant soil type in the actual sampling area. For Level II this should be in the buffer zone of the plot. More detailed information on the location and orientation of the soil profile and on the required observations which need to be made while digging the profile are given in Annex II.
- The parameter '**effective soil depth**' is MANDATORY to report (it replaces the previous parameters 'root' 'rock' and 'obstacle depth'. The 'effective soil depth' is defined as the depth to the 'continuous rock' (IUSS Working Group WRB, 2015). In case the 'continuous rock' is found at a depth of more than 1 meter, it is sufficient to report 999'Units are centimeters.

Continuous rock is consolidated material underlying the soil, exclusive of cemented or indurated pedogenetic horizons such as petrocalcic, petroduric, petrogypsic and petroplinthic horizons.

Continuous rock is sufficiently consolidated to remain intact when an air-dried specimen, 25-30 mm on one side, is submerged in water for 1 hour.

The material is considered continuous only if cracks into which roots can enter are on average ≥ 10 cm apart and occupy $< 20\%$ (by volume) of the continuous rock, with no significant displacement of the rock having taken place (IUSS Working Group WRB, 2015).

- The depth to the mean highest and mean lowest groundwater table (HGWT, LGWT) is provided in PRF file according to 50 cm depth classes.

4.1.1.2 Sampling time

The pedological characterisation has to be carried out only once to make sure that all necessary information is available for soil classification according to WRB. See Annex 1 of IUSS Working Group WRB (2022).

Table 4: Overview of mandatory and optional parameters for the pedological characterisation of the plot on Level I and Level II

Parameter	Unit	Dec.	Mandatory / Optional	
Profile characterisation				
Coordinates of the profile pit (Lat/Long)	+/-DDMMSS	0	M	
Date of profile description	DDMMYY		M	
Elevation of profile pit	Metres asl	0	O	
WRB Reference Soil Group (see IUSS WG on WRB, 2022)	Code		M	
WRB qualifiers and specifiers (see IUSS WG on WRB, 2022)	Code		O	
Definition of diagnostic horizons, properties and materials (see IUSS WG on WRB, 2022)	Code		O	
Upper depth limit of diagnostic horizons, properties and materials	cm from mineral soil surface ¹	0	O	
WRB reference publication	Code		M	
Parent material (European Soil Database)	Code		M	
Mean highest and mean lowest groundwater table depth	Code		O ²	
Type of water table	Code		O ²	
Humus form (Zanella et al. 2022)	Code		M ³	
Effective soil depth	cm from mineral soil surface	0	M	
Horizon characterisation			Org. Layer	Min. Layer
Horizon number	Integer		M	M
Date laboratory analysis	DDMMYY		M	M
Horizon name (symbols for master horizon, subordinate symbol, indication of discontinuity, vertical subdivision)	Code		M ⁴	M ³
Upper and lower limit horizon	cm from mineral soil surface	0	M	M
Horizon distinctness and topography	Code		O	O
Structure	Code		O	M
Moist and dry colour of the soil matrix	Munsell colour code		O	M
Textural class	FAO (2006) code			M
Clay (0 – 2 µm fraction)	%	1		O
Silt (2 – 63 µm fraction)	%	1		O
Sand (63 – 2000 µm fraction)	%	1		O
Code coarse fragments	Code based on vol %		O	O
Coarse fragments	weight %	0	O	O
Total Organic Carbon content	g/kg	1	O	O
Total Nitrogen	g/kg	1	O	O
Total Calcium Carbonate	g/kg	0	O	O
Gypsum content	g/kg	0	O	O
pH		2	O	O
Electrical conductivity	dS.m ⁻¹	0	O	O
Exchangeable Ca, Mg, K, Na	cmol(+)/kg	3	O	O
Cation Exchange Capacity	cmol(+)/kg	2	O	O
Code Porosity	Code		O	O
Bulk Density	kg/m ³	0	O	O
Method to determine Bulk Density	Text/code		O	O
Abundance classes of very fine, fine, medium and coarse roots	Code		O ⁵	O ⁴

¹ Note that the ICP Forests approach deviates here from WRB, which means that for soil classification purposes only the WRB reference depth (0-cm level below OL horizon) should be used, while depths of diagnostic horizons should be reported according to ICP Forests depths (0-cm level below forest floor).

² In hydromorphic soils, this parameter is mandatory

³ On both Level I and Level II plots, it is mandatory to report at the humus system level of Zanella et al. (2022). On Level II it is optional to report up to the humus form level, so the second, more detailed level.

⁴ Master symbol is always mandatory. Subordinate symbol, indication of discontinuity, vertical subdivision only when it is defined.

⁵ Mandatory on Level II core plots

4.1.1.3 Sampled layers

- Each pedological characterisation needs to be accompanied by sampling of the identified horizons.
- Note that for the mineral horizon designations, the FAO (2006) definitions are applied whereas for the organic horizons the European horizon symbols (OL, OF and OH, HF (fibric), HM (mesic) and HS (sapric))(see Annex II).
- The analytical data required for soil classification should be reported in the PFH-file.

4.1.1.4 Number of samples

One sample for each identified horizon is sufficient. In case more than one sample for each horizon is analysed, the average value should be reported.

4.1.2 Soil sampling at fixed depths

4.1.2.1 Allocation of soil sampling sites

Sites that should be avoided are areas around tree stems (1m) and animal holes, disturbances like wind-thrown trees and trails. A record of the places sampled should be kept.

4.1.2.2 Sampling time

In order to reduce temporal variations, especially in the forest floor, sampling activities should be confined to periods with low biological activity, e.g., winter or dry season, based on expert judgement. However, when repeating the soil survey, one should stick to the same period (season) as for the first survey. The sampling dates have to be reported in the reduced plot file (*.PLS file).

4.1.2.3 Sampled layers

The “forest floor” or organic layer at the soil surface is sampled separately from the underlying mineral soil. **Buried** organic (including peat layers) layers are sampled in the same way as mineral layers.

Care should be taken to correctly separate the forest floor from the mineral soil material. Separation will be done in the field, but will be checked in the laboratory, following the internationally accepted criterium (at least 20% organic carbon) to make a distinction between both layers. According to this criterium, organic carbon determination (which is mandatory for both Levels of the survey) has to be used to check whether the separation has been done correctly. If the separation was not done correctly, a new sample has to be taken.

Where possible, the organic and mineral soil should be sampled at exactly the same locations, e.g., sample the mineral soil where the organic layer has already been removed for sampling.

A distinction has to be made between an organic layer that is saturated (H) for more than 30 consecutive days in most years or drained, or not saturated (O) or saturated for less than 30 consecutive days in most years and not drained (FAO, 2006; IUSS WG on WRB 2022). The organic layer in aerated conditions may consist of one or more of the following organic subhorizons (Zanella *et al.* 2022): litter (OL), fragmentation horizon (OF) and/or humus (OH). In water-saturated organic layers a distinction has to be made between HF, HM or HS horizon. Detailed definition and descriptions can be consulted in Annex II.

For the submission of data, these horizons are designated as OL, OF, OFH and OH for the aerated organic (O) layers and as HF, HM, HS for the saturated H-layers. The thickness of the different horizons has to be measured (perpendicularly to the local slope gradient) and reported.

If OL-horizon is sampled, it should be sampled separately. The OH-horizon has to be sampled separately only if it is thicker than 1 cm; otherwise, it may be sampled together with the OF-horizon. Optionally, the individual horizons (OL, OF, OH) may be sampled and analysed separately.

In the mineral soil, sampling should be done by fixed depth. **The top of the mineral soil corresponds with the zero reference level for depth measurements.** Depths below the zero reference level should always be measured in a plumb-vertical way (in contrast to the forest floor where the depth is measured perpendicular to the local slope).

Mineral soil layers are designated as 'Mij', where i is the first number of the upper depth limit and j is the first number of the lower depth limit (e.g., M01 corresponds to the 0-10 cm layer). Table 5 shows the layers that should be sampled.

Table 5: Status of layers to be sampled in both levels

Level I ⁽¹⁾		Level II ⁽¹⁾	
Mandatory	Optional	Mandatory	Optional
OF+OH, H layer 0-10 cm 10-20 cm 20-40 cm	OL layer 0-5 cm 5-10 cm 40-80 cm ⁽²⁾	OF+OH, H layer 0-10 cm 10-20 cm 20-40 cm 40-80 cm ⁽³⁾	OL layer 0-5 cm 5-10 cm

¹ Note that the entire thickness of the predetermined depth should be sampled and not the central part of the layer only.

² Optional, but recommended if big changes between topsoil and subsoil are to be expected

³ Only mandatory for a first assessment, not to be repeated (optional) for a next survey if all mandatory parameters were determined with the reference method, see also par. 5.2.1., key soil parameters

If the upper surface of an indurated horizon (e.g. parent rock) is above the lower limit of sampled soil (40 cm for Level I; 80 cm for Level II), the soil is to be sampled till the depth of the limiting horizon. For example, a M48 layer subsample taken at a location where the rock surface reaches up to 65 cm below the soil surface is composed of material from the mineral soil between 40 and 65 cm depth. The depth range of the upper limit of the indurated horizon is reported under 'Effective soil depth' in the PRF file.

Material discarded for the representative sample can be used to refill bore holes or pits.

Sampling of peatlands

The sampling design is based on the WRB definition of Histosols (= peat soils) which is based on the 40 cm boundary. As long as the peatlayer is less than 40 cm the existing sampling design for mineral forest soils shall be applied (separate sampling of the organic layers and mineral soil according to the fixed depth layers). From the moment the peat is \geq 40 cm, the peatlayer shall be sampled according to the PEATLAND SAMPLING DESIGN:

This means that the peatlayer is sampled at fixed depths, mandatory 0 – 10 and 10 – 20 cm and optionally at 20 – 40 and 40 – 80 cm. In the reporting forms a separate name for the peatlayers shall be used, namely H01, H12, H24 and H48 in the records for the organic layers. The list of the analytical parameters (mandatory and optional) follows the rules for the OF, OH or OFH layer (see Table 11).

If the conditions allow (lower water table), the mineral soil below the peat soil (> 40 cm) can be further sampled till a depth of 80 cm (where the 0 cm reference remains at the top of the peat layer). The standard sampling depths and layer codes should be followed as much as possible, and determined based on the dominant material within the given depth range. For example, if the 40 – 80 cm depth range (measured from the top of the peat or mineral soil as the 0-cm reference depth) contains both peat and mineral layers, but has an overall organic carbon content below 20 %, it should be assigned the code "M48".

Peat soil can be covered by a forest floor (OL, OF, OH layers), especially when the peat is drained. In case the forest floor is saturated for most days of the year and HF, HM or HS layers can be distinguished, these layers will become part of the fixed depth peat layers (M05, M51, M12, M12, M24, M48).

4.1.2.4 Number of samples

Level I: For every layer, mandatory 5 subsamples have to be taken (a composite of 5 is allowed) (e.g., if taken with an auger ≥ 8 cm diameter), but more subsamples are required according to the variability of the site. Mandatory 1 composite sample has to be analysed and reported, more can be analysed optionally to determine the variability of the site. In case of very stony soils where sampling by auger is not possible, 1 composite of at least 3 subsamples can be accepted for the optional depth layers (M24 and M48) only.

Level II: For every layer, mandatory a MINIMUM of 24 subsamples has to be taken, to be combined in at least three composite samples (e.g., at least 3 composites of each 8 subsamples or 4 composite samples of each 6 subsamples). Each composite sample should be spatially clustered. Mandatory at least 3 values have to be reported (1 from each composite), to obtain information on the sampling variability among clusters (composites). The samples should be representative for the whole plot area. The distance between sampling clusters (composites) should be at least 5 meter in order to avoid autocorrelation.

The subsamples have to be of equal mass, except for 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 is function of the thickness of the actually sampled layer. In the above example (section 4.1.2.3. last part), the mass of the subsample taken should be a proportion equal to $(65-40)/(80-40)$ of the standard sample mass.

4.1.3 Sampling at fixed depth for soil bulk density

4.1.3.1 Allocation of the soil sampling sites

Not specified except when done in association with soil water measurements (see 4.1.4.1).

Determination of bulk density by measurement is mandatory for Level II, but if this measurement has been done according to the reference methods once, it has not to be repeated. For Level I, bulk density is a mandatory parameter too, but it can be estimated using pedotransfer functions (PTFs). If pedotransfer functions are used, regional calibration and validation are necessary. Information on how to determine the usefulness and predictive quality of bulk density PTFs for forest soils can be found in De Vos *et al.* (2005).

4.1.3.2 Sampling time

Not specified.

4.1.3.3 Sampled layers

The determination of the bulk density is mandatory on 3 depth layers (0-10 cm, 10-20 cm and 20-40 cm) on non-stony soils and optional on the 4th depth layer (40-80 cm). At least 5 replicates need to be taken for each depth layer.

4.1.3.4 Number of samples

Per plot, five samples with a minimal volume of 100 cm³ have to be taken.

4.1.4 Sampling for soil water measurements

4.1.4.1 Allocation of the soil sampling sites

On each plot at least 3 profiles are sampled separately. The location of these profiles within the plot may be chosen freely, as long as their spatial design meets following requirements:

- The individual profiles are representative for the soil condition within the plot;
- The profiles are not located in one single profile pit (e.g., profiles are at least some meters apart);
- The profiles should be situated as close as possible to the location of the soil moisture measurement sensors;

The exact coordinates of each profile location should be determined and kept for internal record.

4.1.4.2 Sampling time

The samples should be taken when the soil is close to field capacity, which is often towards the end of the winter. Do not sample the soils when it is freezing. Ideally the undisturbed cores are taken at the time of the installation of the soil moisture probes to assure 1) minimal soil disturbance and 2) that the cores are taken in the same layer and horizon as the soil moisture sensors.

4.1.4.3 Sampled layers and number of samples

At each location, adequate undisturbed soil sampling within the soil profile is done according to the sampling scheme in Table 7. At least one undisturbed core is taken within the fixed depth intervals 0 - 20, 20 - 40 and 40 - 80 cm, preferentially at the same depth as the soil moisture measurements. See also the Manual Part IX on Meteorological Measurements. The exact depth range of the soil core (top to bottom of core) is reported, along with the ring ID information.

When forest floor thickness (OF + OH layer) is > 5 cm, the OF+OH layer should be sampled also with a suitable cylinder or frame. Optionally, extra mineral soil layers or horizons could be sampled that are considered relevant for the hydrological regime of the soil profile.

Table 7: Sampling scheme for core samples to determine soil water retention characteristic

Matrix	Depth interval (cm)	Minimum number of replicates		Requirements for Level II core plots
		per profile	per plot	
Organic Layer	OF+OH > 5 cm thick	1	3	Mandatory
	OF+OH ≤ 5 cm thick	-	-	Not required
Mineral layer	0 - 20 cm	1*	3	Mandatory
	20 - 40 cm	1*	3	Mandatory
	40 - 80 cm	1*	3	Mandatory
	> 80 cm	-	-	Optional
	Extra (specific) layer	-	-	Optional

(*) if the mineral layer is difficult to sample (e.g. caused by higher gravel content) a higher number of samples are strongly recommended.

Concluding from Table 7, on each plot at least 9 undisturbed and representative samples should be taken if the forest floor is less than 5 cm thick and 12 samples if the forest floor is more than 5 cm thick.

For each undisturbed sample, the pedogenetic horizon according to the designations given in Annex II, should be reported that contains the centre of the sampling cylinder. The pedogenetic horizon may be deduced from the soil profile description of the sampled plot.

Hence for each undisturbed core sample following information is reported:

- The exact depth range of the core cylinder in cm by reporting the depth of the upper and lower end of the cylinder (e.g. 10 -15 cm for a cylinder of 5 cm in height);
- Pedogenetic horizon containing the centre of the undisturbed sample (e.g. 12.5 cm is located in E horizon)

Use the Soil Water Content (SWC) form of the Soil Water (SW) survey to upload this information to the central database of the Programme Co-ordinating Centre (PCC).

4.2 Sampling equipment

4.2.1 Pedological characterisation of the plot

A list of field equipment for profile description is provided in Annex II.

4.2.2 Soil sampling at fixed depths

It is recommended to sample the **organic** layer with a **frame** of 25 by 25 cm, but alternatives with a minimum total surface of 500 cm² 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 trowel, knife, spatula and/or brush.

For sampling of the **mineral** soil by **auger**, Annex II provides a list with recommended soil augers according to the soil texture type and moisture conditions.

Further following equipment is essential:

- Field forms, pencils and permanent marker
- Folding meter
- Knife
- Spade
- Impact absorbing hammer
- Spatula
- Electronic field balance and spare batteries (only when subsamples are taken)
- Recipients for transporting the samples plus labels
- Sampling tray for mixing the subsamples of the composite samples

4.2.3 Sampling of undisturbed soil core cylinders

Undisturbed soil cores are taken in dedicated metal cylinders (sleeves) with a volume between 100 and 400 cm³. Plastic cylinders are dissuaded. The same steel cylinders can be used for the soil water measurements (Annex I, method SA14) as for determination of bulk density (Annex I, method SA04). The sample ring dimensions should be representative of the natural soil variability and structure. The most frequently met dimensions (height x diameter in mm) of cylinders for forest soil sampling are: 50 x 53 (100 cm³), 40.6 x 56 (100 cm³) and 50 x 79.8 (250 cm³). It is important to verify that the laboratory that will process the undisturbed samples is equipped for the type of sample rings used. The bottom of the sample ring should have a cutting edge. Plastic lids should perfectly fit to both ends of the steel cylinder.

In a soil profile pit, undisturbed samples can be taken directly using the sample ring, without extra material. When sampling is done in a bore-hole, a closed ring holder is recommended.

In conclusion, the sample material consists of:

- Steel cylinders (sample rings) with lids
- Open ring holder (optional)
- Closed ring holder (needed when sampling in boreholes)
- Spade and/or trowel for digging out the cylinder
- Impact absorbing hammer (for hard soil layers only)

- Small frame saw
- Spatula or knife
- Waterproof marker for labelling
- Plastic bags or foil for wrapping the rings

4.3 Sample collection

4.3.1 Pedological characterisation and profile pit sampling

By profile sampling, using a knife and a tray the soil is gently loosened from the respective horizon. By using the tray any material that accidentally is included in the sampled material can easily be removed before the material is brought into the bag.

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

Samples for chemical analyses can be collected in various ways. The mode of sampling should be recorded, as for example on a sample list and by means of either a simple sketch or by special photos. The chosen sampling procedure should reflect the soil variability within the horizon and naturally the purpose of the prospection.

- The “composite” sample: several soil samples are collected throughout the horizon. These samples can be kept separate. If, as for example, from profile number 12 the 4 subsamples a, b, c and d are collected in horizon 2, this can be labelled P12H2a, P12H2b, P12H2c, P12H2d, or they can be mixed together in one bag and labelled e.g. P12H2.
- The “massed average” sample: is a sample taken throughout the whole (vertical) thickness of the horizon.
- The “middle” sample: is a sample taken more or less in the middle of the horizon, there where the characteristics of the horizon are best developed. For classification purposes, the “middle” sampling strategy is recommended.

4.3.2 Sampling at fixed depths

4.3.2.1 Organic layer sampling

It is strongly recommended to make the description of the humus form simultaneously with the sampling of the forest floor.

Either all subsamples coming from the inside of the frame or from the auger are taken individually to the lab to determine the dry mass (kg/m^2), or the subsamples are first bulked in the field and subsequently a subsample is taken to the lab 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.

Record the total surface of each subhorizon (surface of the frame/auger * N° of subsamples) to allow stock calculations later on.

The frame is pushed carefully in the forest floor. Then the organic subhorizons are separately cut out along the frame using a sharp knife. Be careful not to include any mineral soil material in the OH sample. Living material (such as mosses, roots, etc.) and objects > 2 cm in diameter are removed from the sample but smaller twigs, fruits remain to determine the mass of the sample.

4.3.2.2 Mineral soil sampling

Augering is preferred but pits are allowed, especially in case of stony soils where augerings are difficult or impossible.

4.3.2.3 Size of samples

The minimum mass of each representative sample for chemical analysis should be large enough for all laboratory analyses (mandatory and optional parameters) and possible repetitions or reanalyses in time. It is also advisable to keep the sample in a storeroom. The absolute minimum mass of samples (field mass) with no or little gravel should be 500 grams but 1 kg is recommended for important (reference) samples.

4.3.3 Cores for bulk density and soil water retention measurements

The core method is applicable for stone-less and slightly stony soils. The samples are taken with core cylinders on horizontal sections.

The sampling procedure for undisturbed soil sampling (core sampling in steel rings) is as follows:

- Take soil cores carefully to ensure minimal compaction and disturbance to the soil structure:
- In a soil pit, undisturbed samples can be taken by hand pressure directly using the sampling ring.
- Alternatively, an open ring holder may be used. In such a holder, the ring is locked by means of a rubber or lever. Over the ring some space headroom is left allowing for taking an oversize sample. This prevents the sample for compaction during sampling.
- In hard soil layers, an impact absorbing hammer may be used for hammering the ring holder into the soil.
- When sampling in a bore hole, 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 come 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.
- The ring sample is taken vertically with its cutting edge downwards;
- Dig out the cylinder carefully with a trowel, if necessary, adjust the sample within the cylinder before trimming flush, trim rough the two faces of the cylinder with 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).
- Close both sides of the cylinders with suitable lids.
- Record sampling date, sample grid reference, horizon encompassing the centre 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).
- Label the cylinder on the lid clearly with the sample plot reference, the sampling date, the horizon code and the sample depth;
- Wrap the ring samples in plastic bags or a plastic or aluminium foil to prevent from drying.

4.3.4 Excavation method for sampling for bulk density

An alternative to core samples for bulk density, is sampling by the excavation method (ISO 11272, 2017). Sampling of bulk density in stony soils is much more delicate, and surely much more time consuming than sampling in soils with none or little coarse fraction.

First a carefully levelled 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 dm³ should be sufficient. While excavating the sample, compaction of the sides should be avoided. The sample is stored in a plastic bag, avoiding any compaction. Line the excavation hole with a thin but strong plastic film, fill the hole to excess with a known volume of sand. The hole is filled using a funnel kept 5 cm above the ground, level the surface and avoid compaction. Remove the excess sand into a graduated measuring cylinder, and read the volume. Calculate the total volume of sand filled into the excavation hole (see also Annex I, SA04).

4.4 Sample storage and transport

The sample recipient should be properly labelled with a comprehensive code preferentially including location name, plot number, profile number, horizon number or layer name, depth of sample, and sampling date.

In the field all the samples either in bags, boxes, metal rings etc. should never be left exposed to the open air and sun. Otherwise, water will evaporate from the sample and condense in the same bag or recipient, and there is a risk of ultra-desiccation. The warming up of the sample will also activate the biologic activity within the sample.

Samples for standard soil laboratory analyses are mostly kept either in plastic bags or boxes. If using plastic bags, the bags with a closing zipper and with a special label for writing the sample code are recommended. Also feasible is sampling and transporting the samples in plastic bags and then transferring them into plastic boxes for drying and laboratory treatments.

The undisturbed samples are transported in plastic boxes or aluminium cases. They protect the samples from heat, humidity or dust. If transported in vehicles over long distances, shocking of samples should be avoided by using shockproof materials. Prevent undisturbed soil samples from freezing. Store the samples at 1 to 2 °C to reduce water loss and to suppress biological activity until analysis. It is recommended to avoid weeks of storage of undisturbed soil samples. Ideally, undisturbed soil samples are analysed in the lab immediately after sampling.

The indoor preparation of the soil samples for further laboratory work is based on the ISO 11464 (2006) method (Soil quality – pretreatment of samples for physico-chemical analysis). Collected samples should be transported to the laboratory as soon as possible and air dried or dried at a temperature of 40 °C (ISO 11464, 2006). They can then be stored until analysis. To recalculate the analysis results on mass basis, the moisture content of the sample has to be determined by oven-drying the sample once at 105°C (ISO 11465, 1993).

Living macroscopic roots and all particles, mineral and organic, with a diameter larger than 2 mm, should be removed from the samples by dry sieving as a preparation for analysis. The particles not passing the 2-mm sieve are weighed separately for the determination of the coarse fragments content (required for bulk density). To guarantee a harmonised approach, samples should not be further milled or ground. Though, for those analyses for which finely ground material is required [e.g. Carbonate content (SA07), Total Organic Carbon (SA08), Total Nitrogen (SA09), Total Elements (SA12),...] further milling or grounding is allowed.

4.5 Long-term storage of soil samples

The sample material for long-term storage should be kept without preservative under normal room conditions with minimal temperature and humidity fluctuations, shielded from incident light. When the humidity in the storage room cannot be controlled, the soil samples should be kept in air-tight containers. The samples should be stored at least till the next soil inventory.

5 Measurements

5.1 Physical characterization

5.1.1 Mass and thickness of organic layer

This is the determination of the mass (volume-dry mass, kg/m²) and the thickness (cm) of the forest floor. For the method of soil moisture content, see Annex I, SA02.

In the field, the total fresh mass of each layer (OL, OF and OH or HF, HM, and HS) has to be determined, together with the thickness of the concerning layer. Of each layer a subsample is collected for determination of moisture content (mass %) in the lab in order to calculate its total dry mass (kg/m²). It is mandatory to report both, thickness and dry mass of all forest floor layers (OL, OF, OH).

5.1.2 Particle size distribution

The determination of the soil granulometry and classification according to the USDA-FAO textural classes (Figure 1) is mandatory for the mineral layers for Level II, only if not already determined during a previous survey (no repetition required if this parameter was already measured). The particle size classes of the fine earth fraction (< 2 mm) are defined as follows (FAO, 2006):

Clay	< 2 μm
Silt	2 – 63 μm
Sand	63 – 2000 μm

For Level I, information on textural class for the mineral layers is mandatory too (though again only if not done in a previous survey). However, for Level I an estimate based on the finger test in the field on 1 composite of each layer can be accepted for classifying the soil texture according to the USDA-FAO textural classes. In addition an estimate of the clay content is mandatory as well. Practical guidelines can be consulted in Annex 1 of IUSS WG WRB (2022).

Method

Level I: finger test for estimation of soil texture classified according to USDA-FAO texture triangle (FAO, 1990), and for estimation of the clay content (%). Optional: reference method as described for Level II.

Level II: reference method as described in Annex I: SA03

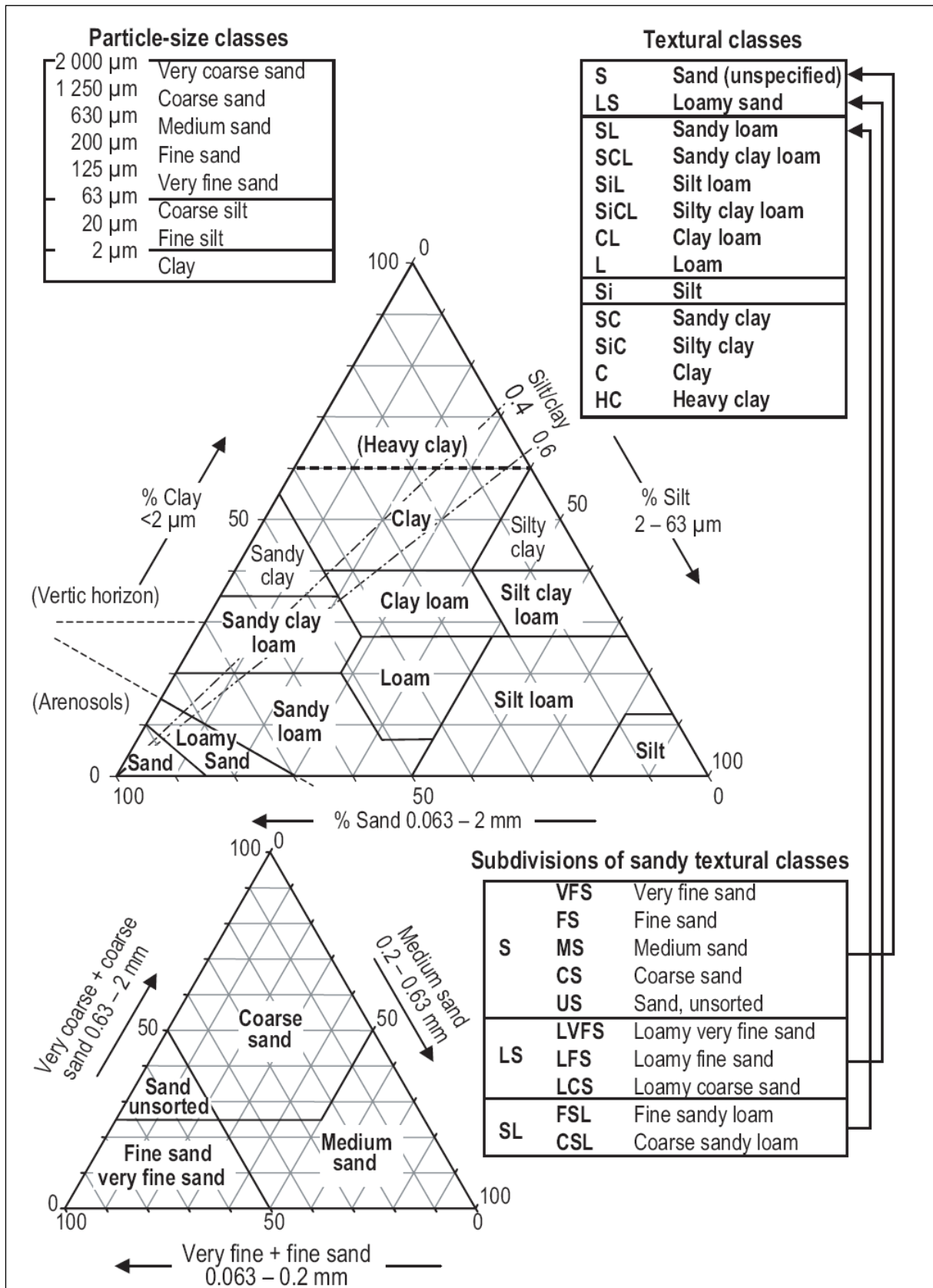


Figure 1: Relation of constituents of the fine earth by size defining textural classes and sand subclasses. Textural classes based on USDA (1951), adopted by FAO (1990) and refined by FAO (FAO, 2006)

5.1.3 Bulk density

Definition

The bulk density of the soil (BDs) is defined as the mass of a unit volume of oven dry soil. The volume includes both solids and pores.

The ICP Forests programme asks to report the bulk density of the fine earth (BDfe), to allow stock calculations.

In mineral soils without coarse fragments the bulk density of the total mineral soil (BDs) is equal to the bulk density of the fine earth (BDfe).

Optional and mandatory parameters

The mean bulk density of the fine earth (BDfe) has to be reported mandatory for three mineral soil layers (0-10 cm, 10-20 cm and 20 – 40 cm). For Level I, these values may be obtained either by estimation, pedotransfer functions or measurement. For Level II, the bulk density has to be measured. Determination of the bulk density of the 40-80 cm layer is optional for both Levels. No re-measurement is required if this parameter was determined according to the reference method for the first survey.

Methodology

For measurement: at least five samples have to be taken with a minimal volume of 100 cm³ per plot and per layer. In addition, the determination of bulk density requires estimation of coarse fragments according to the USDA-FAO classes (FAO, 1990, 2006, see Table 8). The latter can be measured or estimated in the soil profile. This estimation according to the fixed depths shall be done in addition to the profile description which follows the genetic layers. For detailed description of the method, see Annex I, Soil Analysis Method 4 (SA04).

5.1.4 Coarse fragments

Coarse fragments group all gravel, stones and boulders with a diameter larger than 2 mm. The size classes according to the greatest dimension of the individual gravels/stones are defined in Table 8.

Table 8: Size classes of the coarse fragments (FAO, 2006)

Size (cm)	Class name
0.2 – 0.6	Fine gravel
0.6 – 2.0	Medium gravel
2.0 – 6.0	Coarse gravel
6 – 20	Stones
20 – 60	Boulders
60 – 200	Large boulders

Report the amount of coarse fragments of the individual mineral layers in volume %. The abundance of coarse fragments can be measured in the laboratory, but is usually estimated during routine soil profile observations. In the case that very coarse materials are present (stones and boulders), the quantity of these materials has to be estimated in the field. Two methods are recommended: (i) the method established in Finland as described in Annex I, SA05 or (ii) the method used in Germany (see Annex I, SA04). Alternatively, the excavation method can be used, although it is an extremely labour-intensive method (ISO 11272, 2017).

The determination of coarse fragments is mandatory for the 0-10, 10-20 and 20-40 cm mineral layer and optional for 40 - 80 cm mineral layers in both Level I and Level II. In case of re-assessment (if this parameter was already measured according to the reference method in previous surveys) the parameter is optional. For Level I the parameter may be estimated, for Level II it must be measured using the methods described in Annex I, Soil Analysis Method 5 (SA05).

5.1.5 Combined approach to estimate bulk density of the fine earth and the content of coarse fragments

Riek and Wolff (2006) have revealed that the soil physical parameters (in this case bulk density and fine earth stock) can only be recorded with field methods at specific locations in an inadequate or scarcely reproducible manner. This applies to soils with a high content of coarse gravel and/or the presence of stones and boulders. Because of their low volume, the core samplers normally used in forest monitoring are unable to representatively collect stones or large portions of coarse fragments in the field. In these cases, the excavation method may produce good results but it may be too expensive, time-consuming and destructive in the framework of large-scale monitoring.

The combined approach can improve the determination of these parameters at locations with a high content of coarse gravel and/or the presence of stones and boulders and lead to a better approximation of the real coarse fragments content.

In the case of a high content of coarse gravel and/or the presence of stones and boulders, the quantity of both fine earth and coarse fragments has to be estimated/sampled in the field. Methods should be selected according to the prevailing conditions (e.g., coarse fragment content and size) at each individual sampling site.

In the analysis each method or each combined method leads to the determination of (partially) different parameters which means that different calculation formulas are needed.

A description of the different methods and/or combined methods, the related parameters and calculation methods are described in Annex I Soil Analysis Method 4 (SA04).

If the mineral soil contains no coarse fragments or the (estimated) coarse fragment portion is less than 5 % (**case 1**), then the bulk density of the fine earth (BD_{fe}) is approximately equivalent to the bulk density of the total mineral soil (BD_s) (see paragraph 5.1.3).

In case of mineral soils with a coarse fragment content of more than 5% which can be sampled with a core sampler or any other (representative) sampler for coarse fragments < 20 mm (**case 2**), a representative volume sampling with core sampler, root auger, AMS core sampler with liner or hollow stem auger is done. The % coarse fragments (either mass% or vol%) can be determined based on the coarse fragments present inside the sampling ring (assumed to be representative for the given layer). When calculating the bulk density of the fine earth, the volume of the coarse fragment content is subtracted from the total volume of the sampler and the mass of the coarse fragments subtracted from the mass of the total oven-dry material in the sampler.

In case the mineral soil cannot be representatively sampled with a core sampler or any other samples (coarse fragments > 20 mm) (**case 3**), there are two possibilities of taking the coarse fragments into account. The amount of coarse fragments has to be estimated in the field or be determined by additional sampling with a shovel or a spade (representative volume sampling).

When representative volume sampling is not possible, sampling with mini-core samplers and estimation at the profile (coarse fragments > 60 mm) is required (**case 4**). The bulk density of the fine earth in the spaces between the coarse material [soil skeleton] is determined with a mini-core sampler. In addition, a disturbed spade /shovel sample is taken in order to determine factor f (correction factor for a possible coarse fragment portion in the mini-core sampler). Furthermore, the coarse fraction portion > 60 mm is estimated at the profile.

5.1.6 Determination of the soil water retention characteristic (SWRC)

In order to determine the SWRC, the volumetric water content (θ in volume fraction, $m^3 m^{-3}$) is determined at predefined matric potentials (ψ , in kPa). As indicated in Table 9, six of these matric heads are mandatory to determine. Extra observations of the SWRC at pressures -10, -100 and -250 kPa are optional but they greatly improve fitting the SWRC.

Some matric heads immediately provide information on SWRC parameters: at 0 kPa the maximum water holding capacity (WHC) of the saturated soil sample is determined; depending on definitions and soil texture field capacity (FC) may be inferred from -10 till -100 kPa; permanent wilting point (PWP) is attained at a matric pressure of – 1500 kPa and dry bulk density (lowest pressure at about 10^{-6} kPa) derived in the oven at 105°C.

The standard instruments required for each determination are listed in Table 9. The reference methods for all physical parameters are listed in Table 10.

Table 9. Overview of matric heads to assess for the determination of the SWRC

Matric potential ψ			Recommended instrument	Estimator	Equivalent pore size diameter	M / O
cm H ₂ O	pF	kPa				
1	infinitely small	0	Pycnometer	$\approx \theta_{\text{sat}}$ = water holding capacity = Total porosity	Jurin's law, Hillel (1980) > 1 mm	M
10	1.0	-1	Sand suction table		300 μm	M
51	1.7	-5			60 μm	M
102	2.0	-10		Field capacity sand	30 μm	O
337	2.5	-33	Kaolin suction table	Field capacity siltloam	10 μm	M
1022	3.0	-100	Pressure plate extractor or pressure membrane cells	Field capacity clay	3 μm	O
2555	3.4	-250			1.2 μm	O
15330	4.2	-1500		Permanent wilting point	0.2 μm	M
10^7	7.0	-10^6	Oven	Dry BD	0.0003 μm	M

Where:

- 1) the pF is the logarithm of the absolute value of the matric potential expressed by the graduation of the water column (cm).
- 2) 1 kPa = 10.22 cm H₂O or 1 cm H₂O column = 0.097885 kPa
- 3) 100 kPa = 1 bar

Table 10: Overview of the reference methods for physical parameters

Parameter	Reference Method	Unit
Particle size distribution (sand, silt, clay fractions)	Pipette method	%
	Finger test method (only allowed on Level I)	
Coarse fragments	Laboratory measurement	vol%
	Field estimate during soil profile description	
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	m^3/m^3
Bulk density	Oven drying at 105°C	kg/m^3
Volume dry mass of organic layer	Field measurement of total fresh mass	kg/m^2
	Field measurement of the horizon thickness	cm
	Determination of moisture content in the laboratory	mass%

5.2 Chemical characterization of collected samples

5.2.1 Selected key soil parameters for the Level I and II Survey

An overview of the key parameters to be measured is presented in Table 11. Note that the minimum requirement for a number of the mandatory parameters, indicate that in the mineral

layers below 20 cm the parameters should be measured once and not necessarily be re-measured a second time.

With regard to the nutrients, the amount extracted by aqua regia is mandatory for the OF+OH horizons and H layers of the organic layer and optional for the mineral topsoil. While from this extraction not the real total content is obtained, it is useful as an estimate of the nutrient stock. Extra costs and work are minimal as it can be measured from the same extraction to be made for the heavy metals (mandatory for both the OF+OH horizons, H-layers and the mineral topsoil). For the determination of the 'real' total amounts, more specialised material and skill are required. As these 'real' total contents are important for the calculation of weathering rates and critical loads, they are optional for the mineral layers of Level II.

Note that the measurement of carbonates is required also for the correction of the organic carbon content if the $\text{pH}(\text{CaCl}_2) > 5.5$ in the organic and > 6 in the mineral layer.

For the determination of the pH, measurement on a CaCl_2 -extract is mandatory. $\text{pH}(\text{H}_2\text{O})$ has been made an optional parameter for reasons of comparability, as this is mostly used in literature.

Table 11: Chemical and physical key soil parameters on the samples taken at fixed depths⁽¹⁾

Parameter	Unit	Decimals	Level I						Level II					
			Organic Layer		Mineral Layer				Organic Layer		Mineral Layer			
			OL	OF+OH, H ⁽²⁾	0-10 cm	10-20 cm	20-40 cm	40-80 cm	OL	OF+OH, H ⁽²⁾	0-10 cm	10-20 cm	20-40 cm	40-80 cm
Physical soil parameter														
Organic layer mass	kg/m ²	2	O	M	-	-	-	-	O	M	-	-	-	-
Coarse fragments	vol %	0	-	-	M ^{(3), (4)}	M ^{(3), (4)}	M ^{(3), (4)}	O ⁽⁴⁾	-	-	M	M ^{(3), (4)}	M ^{(3), (4)}	O ⁽⁴⁾
Bulk density of the fine earth	kg/m ³	0	-	-	M ^{(3), (5), (6)}	M ^{(3), (5), (6)}	M ^{(3), (5), (6)}	O	-	-	M ^{(3), (5)}	M ^{(3), (5)}	M ^{(3), (5)}	O
Particle size distribution (FAO, 1990)	-	-	-	-	M ^{(3), (7)}	M ^{(3), (7)}	O	O	-	-	M ⁽³⁾	M ⁽³⁾	M ⁽³⁾	M ⁽³⁾
Clay content	%	1	-	-	M ^{(3), (7)}	M ^{(3), (7)}	O	O	-	-	M ⁽³⁾	M ⁽³⁾	M ⁽³⁾	M ⁽³⁾
Silt Content	%	1	-	-	O	O	O	O	-	-	M ⁽³⁾	M ⁽³⁾	M ⁽³⁾	M ⁽³⁾
Sand Content	%	1	-	-	O	O	O	O	-	-	M ⁽³⁾	M ⁽³⁾	M ⁽³⁾	M ⁽³⁾
Chemical soil parameter														
pH(CaCl ₂)	-	2	-	M	M	M	O	O	-	M	M	M	M ⁽³⁾	M ⁽³⁾
pH(H ₂ O)	-	2	-	O	O	O	O	O	-	O	O	O	O	O
Total organic carbon	g/kg	1	-	M	M	M	M	M	-	M	M	M	M	M
Total nitrogen	g/kg	1	-	M	M	M	M	M	-	M	M	M	M	M
Carbonates	g/kg	0	-	M ⁽⁸⁾	M ⁽⁸⁾	M ⁽⁸⁾	O	O	-	M ⁽⁸⁾	M ⁽⁸⁾	M ⁽⁸⁾	O	O
Aqua Regia extracted P, Ca, K, Mg, Mn	mg/kg	1	O	M	O	O	O	O	O	M	O	O	O	O
Aqua Regia extracted Cu, Pb, Cd, Zn	mg/kg	1	O	M	M	-	-	-	O	M	M	-	-	-
Aqua Regia extracted Al, Fe, Cr, Ni, S, Hg, Na	mg/kg	1	O	O	O	-	-	-	O	O	O	-	-	-
Exchangeable Acidity, Free H ⁺ , Exchangeable cations Al, Fe, Mn	cmol(+)/kg	2	-	M ⁽⁹⁾	M ⁽⁹⁾	M ⁽⁹⁾	O	O	-	M ⁽⁹⁾	M ⁽⁹⁾	M ⁽⁹⁾	M ^{(3), (9)}	M ^{(3), (9)}
Exchangeable cations Ca, Mg, K, Na	cmol(+)/kg	2	-	M ⁽⁹⁾	M	M	O	O	-	M ⁽⁹⁾	M	M	M ⁽³⁾	M ⁽³⁾
Total Elements: Ca, Mg, Na, K, Al, Fe, Mn	mg/kg	1	-	-	-	-	-	-	-	-	O	O	O	O
Oxalate extractable Fe, Al	mg/kg	1	-	O	O	O	O	O	-	O	M	M	M ⁽³⁾	M ⁽³⁾
Oxalate extractable P	mg/kg	1	-	O	O	O	O	O	-	O	O	O	O	O

¹ Abbreviations: M = mandatory parameter, O = optional parameter

² If the OH - horizon > 1 cm, the OF - and the OH - horizons should be analysed separately and each value has to be reported

³ In case of a re-assessment (if the parameter was already measured according to the reference method for the first survey) , the measurement is optional

⁴ May be obtained by estimation or measurement

⁵ Mandatory only in non-stony soils

⁶ May be obtained by estimation, pedo-transfer function or measurement

⁷ May be obtained by finger test, consists of texture classified according to USDA-FAO texture triangle

⁸ Only mandatory if pH(CaCl₂) > 5.5 or in calcareous soils

⁹ In calcareous soil, the measurement of this parameter is optional

5.2.2 Reference analytical methods

The full description of the reference methods is given in Annex I.

Table 12 gives an overview of the reference methods for the chemical parameters. Note that the parameters are grouped according to the analytical method. As such it is obvious which elements can be measured in the same run, without additional costs and hardly extra work involved.

Table 12: Overview of reference methods for the chemical parameters

Parameter	Reference Analysis Method ¹				Unit ²
	ISO	Extractant	Measurement method(s) ³		
pH(CaCl ₂)	ISO 10390 (2021)	0.01 M CaCl ₂	pH-electrode		
pH(H ₂ O)		H ₂ O	pH-electrode		
Total nitrogen	ISO 13878 (1998)	-	Dry Combustion		g/kg
	ISO 11261 (1995)	-	Modified Kjeldahl		
Total organic carbon ⁴	ISO 10694 (1995)	-	Dry Combustion at ≥ 900 °C		
Carbonates	ISO 10693 (1995)	HCl	Calcimeter		
P	ISO 11047 (1998)	Aqua Regia by reflux digestion or By microwave digestion	ICP	Colorimetry	mg/kg
K, Ca, Mg, Mn				AAS	
Other: Al, Fe, Cr, Ni, Na			ICP		
Hg			ICP		
S	ISO 54321 (2020)	CNS - analyser			
Free Acidity (or sum of AC ⁵) and free H ⁺	ISO 14254 (2018) modified	0.1 M BaCl ₂	titration to pH 7.8 or 'German' method		cmol(+)/kg
Exchangeable Cations	ISO 11260 (2018) modified	0.1 M BaCl ₂	ICP	AAS	
				FES	
Reactive Fe and Al Oxalate extractable P	ISRIC (2002)	Acid ammonium oxalate	AAS	ICP	mg/kg
Total Elements: Ca, Mg, Na, K, Al, Fe, Mn	ISO 14869-1 (2001)	HF or LiBO ₂	AAS	ICP	mg/kg

¹ Reference and full descriptions are given in Annex I

² Results have to be expressed on an oven dry basis

³ For the measurement of a number of parameters there are several alternatives for the equipment that can be used

⁴ Note that for total organic carbon a correction has to be made for total inorganic carbon (carbonates)

⁵ Alternative for the titration of the exchangeable acidity is the sum of the exchangeable Al, Fe, Mn and free H⁺

5.3 Data quality requirements

The quality of the soil analytical data is controlled by the regular organisation of soil ring tests by the Working Group on quality assurance and quality control in laboratories. Each soil laboratory participating in the ICP Forests programme should be qualified for the reported parameters. For qualification procedures, see Manual Part XVI on Quality Assurance and Control in Laboratories. Information on the performance of the concerning soil laboratory is reported to the data centre at each submission period.

5.3.1 Plausibility limits

See Manual Part XVI on Quality Assurance and Control in Laboratories, Table 6 “Plausible ranges for organic and mineral soil samples at the European level.” Laboratories are invited to check the data that are outside these plausibility limits before reporting.

Plausibility limits for SWRC of mineral forest soils and organic layers will be developed in the future.

5.3.2 Data completeness

Tables 9 and 11 outline for all the physical and chemical soil parameters whether and under which conditions they are mandatory or optional to report. When a country/federal state decides to report optional parameters, they should also fulfil the data quality requirements.

Soil water retention data are considered complete if volumetric water content for all six mandatory matric heads (see Table 9) is determined. For scientific reasons analysing the optional matric heads also is strongly recommended. Interpolation of volumetric water content between matric pressures is not allowed.

5.3.3 Data quality objectives or tolerable limits

See Manual Part XVI on Quality Assurance and Control in Laboratories, Table 11 for the tolerable limits of the measured parameters in the ICP Forests soil ring tests.

All reported values should have been measured according to the methods described in Annex I.

5.3.4 Data quality limits

The laboratory results are considered of sufficient quality when the laboratory received a qualification for the concerning parameter(s) after participation in the soil ring test which is organised every third year.

The soil chemical ring test should include at least 5 soil samples (mineral and organic). When 50% of the samples in the ring test are within the tolerable limits, the laboratory is qualified to analyse the concerning parameter and the survey results can be reported to the central database.

6 Data handling

6.1 Data submission procedures and forms

Forms for data submission, dictionaries and explanatory items are found on the ICP Forests web page, at <https://icp-forests.org/documentation/>. The quality information on the labs has to be sent together with the PLS, PRF, PFH, SOM, SWC and SWA forms to the data centre.

The following rules apply:

- Data will be reported for the H- and O-horizons and for the mineral soil.
- For the organic layers reporting is done according to the OL-, OF-, OH-, OFH-, HF, HS, HM horizons or as described in Annex II of this Part of the Manual.
- For the mineral soil, reporting is done according to the defined mandatory depth layers.
- For the peat layers, reporting is done according to the defined depth layers (Mandatory: H01, H12 and Optional: H24 or H48) and following the parameter list for the OF, OH and H-layers of the organic horizons.

6.2 Data validation

Data checks should be done as soon as results from the analyses are available. Data validation and quality assurance should be applied in accordance with the guidelines for quality assurance procedures within the ICP Forests Monitoring Programme (Manual Part III) and in the laboratory within Manual Part XVI on Quality Assurance and Quality Control in Laboratories (§ 3.4.2.2: Cross-checks between soil variables).

6.3 Transmission to co-ordinating centres, with timetable and rules

All validated data should be sent by each National Focal Centre (NFC) and to the central data storage facility at the ICP Forests Programme Coordinating Centre. Detailed time scheduled is provided by the relevant bodies.

6.4 Data processing guidelines

6.4.1 Derived soil parameters

Chemical derived soil parameters such as cation exchange capacity (CEC), Base Saturation (BS), C:N ratio, C:P ratio are not reported, but are directly calculated from organic carbon, total nitrogen and phosphorus, exchangeable cations, acidity and Free H⁺.

A typical example of derived soil physical parameters is the available water capacity (AWC), field capacity (FC), wilting point (WP) and total porosity which may be derived from the SWRC. Soil water retention curve models are fitted to the raw data. For forest soils, one of the best performing functions is the Van Genuchten equation defined by its empirical parameters θ_r , θ_s and empirical constants α , n and $m = 1 - 1/n$. The Van Genuchten model parameters should also be stored.

6.4.2 Data Classification

When presenting the forest soil condition data of Level I on a map, a selection of classes is required. The number of classes is best limited. The limits are then selected in function of the frequency distribution of the parameter results.

In case the results approximate a normal distribution, class limits are chosen more or less symmetrically around a central class. The difference between upper and lower class limits are kept constant, consequently more results are assigned to the middle class.

However, most parameters results are not normally distributed. Often the distributions are positively skewed, showing a tail towards larger values. In order to obtain a distribution of results among the classes similar to normally distributed parameters, the differences between upper and lower class limits are gradually increased.

For the classification of elevated heavy metal concentrations, use is made of available 'toxic' values found in literature and critical levels.

6.4.3 Clustering Soil Observation Plots

Soil chemical properties usually vary within a wide range. They are influenced by many external factors such as climate, soil parent material, age of the soil material and vegetation type. Evaluation of the soil condition based at a large number of observation sites involves the study of relationships among individual soil properties and among soil properties and external influencing factors. In order to investigate these relationships statistically, the need arises to compare groups of individual soils, having similar properties. Considering the site factors that determine forest soil conditions and limitations associated with data availability, the following criteria for clustering each soil observation plot can be used: climatic region, atmospheric

deposition load, soil type, parent material class, texture class, humus type, biogeographical region.

6.4.4 Statistical methods

For each parameter, three statistical approaches can be applied:

1. Descriptive statistics (boxplots, histograms, frequency distributions, means, percentiles, etc)
2. Classical statistical data analysis and testing (parametric and non-parametric methods)
3. Geostatistical approach (including the spatial component)

The statically obtained information offers opportunities for further modelling.

6.5 Data reporting

Data should be accompanied by a “Data accompanying report” (DAR) and any other information requested by the European central data storage facility at the PCC data centre. The DAR should include all details on sampling and analytical procedures. In addition, irregularities in sampling and analytical procedure, missing data, estimated values and encountered errors in the validation, should be documented.

All details on how data are treated and how the calculations are made shall be documented and shall accompany the result to the data storage facility. If values are below the quantification limit (not the detection limit), a value of -1 should be reported. Definitions of the quantification and detection limits can be found in Section 3.3 of the Manual Part XVI on Quality Assurance and Quality Control in Laboratories.

7 References

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Annex I – Methods for Soil Analysis

Please see additional document at <https://www.icp-forests.net/manual>.

Annex II – Soil profile description and classification

Please see additional document at <https://www.icp-forests.net/manual>.

Annex III – Main changes compared to the previous Manual version

Page	Item	Version 2020	Change	Motivation for change
7	Pedological characterisation	reference to IUSS WG on WRB, 2015	reference to IUSS WG on WRB, 2022	At previous FSEPMs it was decided to always work according to the most recent version of WRB. At least when a new pedological characterisation should be carried out. At the FSEPM in 2023 in Vienna, the WRB 2022 system was adopted and agreed to update the soil manual accordingly.
7	Pedological characterisation	reference to IUSS WG on WRB, 2015	reference to IUSS WG on WRB, 2022	The WRB classifies the soil profile from below the OL layer (so from the top of the OF layer - if present) till a depth of 2 m. WRB considers the top of the OF layer as 0 cm line. However, this is in conflict with the sampling regime that ICP Forests maintains for the soil monitoring programme. This means that ONLY FOR CLASSIFICATION purposes (and harmonisation with international soil classification systems) we 'move' the 0 cm line to the boundary below the OL and the OF layer (if present). For defining the sampling depths (and reporting soil depths to the ICP Forests central database), the 0 cm line remains at the boundary between the bottom of forest floor (O or H horizon) and the mineral soil (except when the organic layer is thicker than 40 cm).
pg. 26 and several places in Annex I 'Methods for soil analysis'	References to ISO standards	References to ISO standards 1) ISO 11464 (1994) pre-treatment of samples 2) ISO 11466 (1995), ISO 12914 (2012), ISO 16174 (2012) aqua regia extractable elements 3) ISO 11272 (1994) dry bulk density 4) ISO 11260 (1994) CEC, BS in BaCl ₂ solution 5) ISO 11277 (1998) particle size distribution sieving and sedimentation 6) ISO 11465 (1993) dry weight / water content 7) ISO 10390 (2005) determination of soil pH	Update to revised ISO standard: 1) ISO 11464 (2006) pretreatment of samples 2) ISO 11466 (1995), ISO 12914 (2012) and ISO 16174 (2012) on aqua regia extractable elements replaced by ISO 54321 (2020) 3) ISO 11272 (2017) dry bulk density and volume coarse fragments by excavation method 4) ISO 11260 (2018) CEC, BS in BaCl ₂ solution (reviewed and confirmed in 2024) 5) ISO 11277(2020) particle size distribution sieving and sedimentation + amendment (2024) 6) ISO/DIS 11465 (2025) dry weight/water content (<i>in February '25 still under construction</i>) 7) ISO 10390 (2021) determination of soil pH	When referring to ISO standards, this should always be the valid version. ISO standards are reviewed every 5 years. Either an old version is confirmed or replaced by a new edition. In the latter case the old edition is not longer valid. So in our manual we need to refer to the valid version.
Annex II till VII	Guidelines for Forest Soil Description	Detailed guidelines for forest soil profile description based on FAO (1990) and draft of FAO (2006) update	Replace Annex II till Annex VII by concise guidelines on the variables asked in PRF and PFH files. Refer to maximal extend to existing guidelines provided in IUSS WG on WRB, 2022 (Annex I Field Guide).	Annex II till VII in the ICP Forests Manual were developed prior to the survey of 2006 when the FAO guidelines (2006) were still under revision (and the FAO (1990) were outdated). In 2022 the IUSS updated the FAO (2006) guidelines and included updated profile description guidelines in Annex I in the IUSS Working Group on WRB (2022)

				publication. These guidelines are much more in line with the most recent developments of the WRB (2022) classification system. To avoid replication or contradictions, FSCC recommends to follow WRB (2022) to maximal extend. The Annex II in the manual of ICP Forests will only included guidelines to be able to properly complete the PFH and PRF files.
Annex II	Distinction of the layer's lower boundary	Annex II, Horizon Boundary modified from FAO, 2006)	Change compared previous version of the ICP Forests manual, would be the removal of a 'complex' boundary.	The 'complex' boundary was never clearly defined. So recommendation is no longer using it in the future.
Annex II	Structure	Reference was made to the structure type of FAO (2006, table 48)	IUSS Working Group on WRB (2022) revised the structure type and described 5 additional structure type (lenticular, polyhedral, flat-edged, pseudosalt/pseudosilt, stratified)	Of these additional structure, possible only 'stratified' will be relevant for European forests soils. Other type are added for the sake of completeness.
Annex III	Recommendations for locating and describing a soil profile	Annex III: Recommendations for locating and describing a soil profile	Annex III become part of Annex II (with small editorial corrections)	These recommendations are still valid and are now incorporated in the new Annex II. Edits concern the change from 'experimental plot' to 'plot' as ICP Forests plots are observational plots, not experimental plots.
Annex IV	Field equipment	Annex IV: Field equipment	Annex IV become part of Annex II.	These recommendations are still valid and are now incorporated in the new Annex II.
Annex V	Additional information on colour measurements	This annex provided additional information on the colours, particular for optimal measurements for classification purposes (IUSS Working Group on WRB 2006)	reference to IUSS WG on WRB, 2022	In contrast to WRB 2006 and 2014/15, WRB 2022 simplified the colour determinations. No reference is made any longer to 'crushed' or 'broken' colours.
Annex VI	Guidelines for field estimates of textural classes	Annex VI provided a key based on Schlichting et al. 1995 to estimate texture by hand.	Reference to the flow chart provided by IUSS Working Group on WRB (2022), Annex 1 to estimate texture by hand.	Key of IUSS Working Group on WRB (2022) is more feasible and realistic compared to key of Schlichting et al. (1995)
Annex VII	Detailed definitions, rules and conventions on master and subordinate horizon symbols	Based on FAO (2006) guidelines and a preliminary draft of the forest floor horizons provided by the European Humus Reserach Group (Zanella et al. 2006)	IUSS Working Group on WRB (2022) revised the suffix symbols, and added an additional coding list with Greek letter symbols. For the symbols of the humus layers, reference is made to Zanella et al. (2018, 2022).	For sake of harmonisation at the international level, ICP Forests will need to incorporate the WRB symbols in the database. For the humus form, the FSEP adopted the system of Zanella et al. (2018) coming with its own prefix symbols, necessary to classify the humus form.
Annex II	humus classification	Classification of the humus form up to the highest hierarchical level of Zanella et al. 2006	Mandatory: Classification up to the highest hierachical level of Zanella et al. (2018, 2022) on both level I and level II. On Level II, it is optional to report up to the second level of detail.	The Zanella et al. (2006) system was developed for sake of the BioSoil survey but not yet mature enough for official publications in Zanella et al. (2018) and Zanella et al. (2022).

Annex IV – Minor changes after 2025

Date	Minor change to latest published version in 2025	Affected sections of this document