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Convention on Long-range Transboundary Air Pollution

International Co-operative Programme on Assessment and  
Monitoring of Air Pollution Effects on Forests (ICP Forests)

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# **MANUAL**

on

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

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## **Part X**

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## **Sampling and Analysis of Soil**

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## 1. Introduction

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 of the data obtained in the first survey with those of future 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 depths of samplings 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 <sub>tot</sub> , N <sub>tot</sub> , (Carbonates)	Organic	Forest nutrition, atmospheric N deposition, climate change
		Mineral	Forest nutrition (0-20 cm), C- & N sinks
Nutrients	Total P, Ca, Mg, K, 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 <sub>ox</sub> , Fe <sub>ox</sub>	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 <sub>fe</sub> ) 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, 2007a, 2007b).

**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 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
<b>Pedological characterization</b>					
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
<b>Soil sampling at fixed depth</b>					
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
<b>Sampling at fixed depth for soil bulk density</b>					
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
<b>Sampling for soil water measurements</b>					
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 Guidelines for Forest Soil Profile Description (see Annex 2) which are partly based on the 4<sup>th</sup> edition of the Guidelines for Soil Profile Description and Classification (FAO, 2006). 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 (IUSS Working Group WRB 2006, 2007a, 2007b). 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 adopted description and classification guidelines (Zanella *et al.*, 2009).

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 3.

##### **4.1.1.2 Sampling time**

Has to be carried out only once to make sure that all necessary information is available for soil classification according to WRB. See Annex 2: Guidelines for forest soil description.

##### **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, Hfs and Hs) which are in use in forestry for many years (see Annex 7).

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.

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

Parameter	Unit	Decimals	Mandatory /Optional
<i>Profile characterisation</i>			
Coordinates of the profile pit	+/-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, 2007a, 2007b)	Code		M
WRB qualifiers and specifiers (see IUSS WG on WRB, 2007a, 2007b)	Code		O
Definition of diagnostic horizons, properties and materials (see IUSS WG on WRB, 2007a, 2007b)	Code		O
Upper depth limit of diagnostic horizons, properties and materials	cm from mineral soil surface	0	O
WRB reference publication	Code		M
Land use	Code		M
Parent material	Code		M
Mean highest and mean lowest groundwater table depth	Code		O <sup>(1)</sup>
Type of water table	Code		O <sup>(1)</sup>
Effective rooting depth in cm from mineral soil surface	cm from mineral soil surface	0	M
Rock depth in cm from the mineral soil surface	cm from mineral soil surface	0	M
Obstacle depth in cm from mineral soil surface	cm from mineral soil surface	0	M

Parameter	Unit	Decimals	Mandatory /Optional
			<u>Org.</u> <u>Layer</u> <u>Min.</u> <u>Layer</u>
<u>Horizon characterisation</u>			
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 <sup>(2)</sup> M <sup>(2)</sup>
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 micrometer fraction)	%	1	O
Silt (2 – 63 micrometer fraction)	%	1	O
Sand (63 – 2000 micrometer 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 <sup>-1</sup>	0	O O
Exchangeable Ca, Mg, K, Na	cmol(+)/kg	3	O O
Cation Exchange Capacity	cmol(+)/kg	2	O O
Base Saturation	%	0	O O
Code Porosity	Code		O O
Measured or Estimated Bulk Density	kg/m <sup>3</sup>	0	O O
Abundance classes of very fine, fine, medium and coarse roots	Code		O <sup>(3)</sup> O <sup>(3)</sup>

<sup>1</sup> In hydromorphic soils, this parameter is mandatory

<sup>2</sup> Master symbol is always mandatory. Subordinate symbol, indication of discontinuity, vertical subdivision only when it is defined.

<sup>3</sup> Mandatory on Level II core plots

#### 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 organic layer, sampling activities should be confined to periods with low biological activity, e.g. winter or dry season, based on expert judgement. However, the countries that participated in the first survey have to carry out the sampling activities in 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 organic layer at the soil surface is sampled separately from the underlying mineral soil. 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. Separation will be done in the field, but will be checked in the laboratory, following the internationally accepted criteria (FAO 2006, see Annex 7) to make a distinction between both layers. According to these criteria, 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, i.e. 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) or not saturated (O) with water according to the FAO-definition (FAO, 2006). The organic layer in aerated conditions may consist of one or more of the following organic subhorizons (Zanella *et al.* 2009; Zanella *et al.*, in preparation): litter (OL), fragmentation horizon (OF) and/or humus (OH). In water saturated organic layers a distinction has to be made between Hf, Hfs or Hs horizon. Detailed definition and descriptions can be consulted in Annex 7.

For the submission of data, these horizons are designated as OL, OF, OFH and OH for the aerated organic (O) layers and as Hf, Hs, Hfs for the saturated H-layers. The thickness of the different horizons has to be measured 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.

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

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

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

<sup>3</sup> Only mandatory for a first assessment, not to be repeated (optional) for a second 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 'Rock depth' or 'Obstacle 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 parameters (mandatory and optional) follow the rules for the OF, OH or OFH layer.

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 is at the top of the peat layer). The standard sampling depths should be followed as much as possible.

#### 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  $\geq 8$  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 (i.e. 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 yet 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 for the first survey, it has not to be repeated. For Level I, bulk density is a mandatory parameter too, but it can be estimated using pedo-transfer functions. 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 yet 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 4<sup>th</sup> depth layer (40-80 cm).

#### 4.1.3.4 Number of samples

Per plot, five samples with a minimal volume of 100 cm<sup>3</sup> 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 (i.e. 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 6. 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 6:** 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 6, 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 7, 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)

## 4.2 Sampling equipment

### 4.2.1 Pedological characterisation of the plot

A list of field equipment for profile description is provided in Annex 4.

### 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 cm<sup>2</sup> 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 4 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 free 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 cm<sup>3</sup>. Plastic cylinders are dissuaded. The same steel cylinders can be used for the soil water measurements (method SA14) as for determination of bulk density (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 cm<sup>3</sup>), 40.6 x 56 (100 cm<sup>3</sup>) and 50 x 79.8 (250 cm<sup>3</sup>). 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 particular 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 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, 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 organic layer.

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 ( $\text{kg}/\text{m}^2$ ), 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 ( $\text{kg}/\text{m}^2$ ) 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. 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 dm<sup>3</sup> 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 1, 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 (1994) 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, 1994). 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. For those analyses for which finely ground material is required [e.g. Carbonate content (SA07), Total Organic Carbon (SA08), Total Nitrogen (SA09) and 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 Amount of organic layer

This is the determination of the mass of the organic layer (volume-dry mass, kg/m<sup>2</sup>). For the method of soil moisture content, see Annex 1, SA02.

In the field, the total fresh mass of each layer (OL, OF and OH or Hf, Hsf, and Hs) has to be determined, preferably 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/m<sup>2</sup>).

### 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 the first 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):

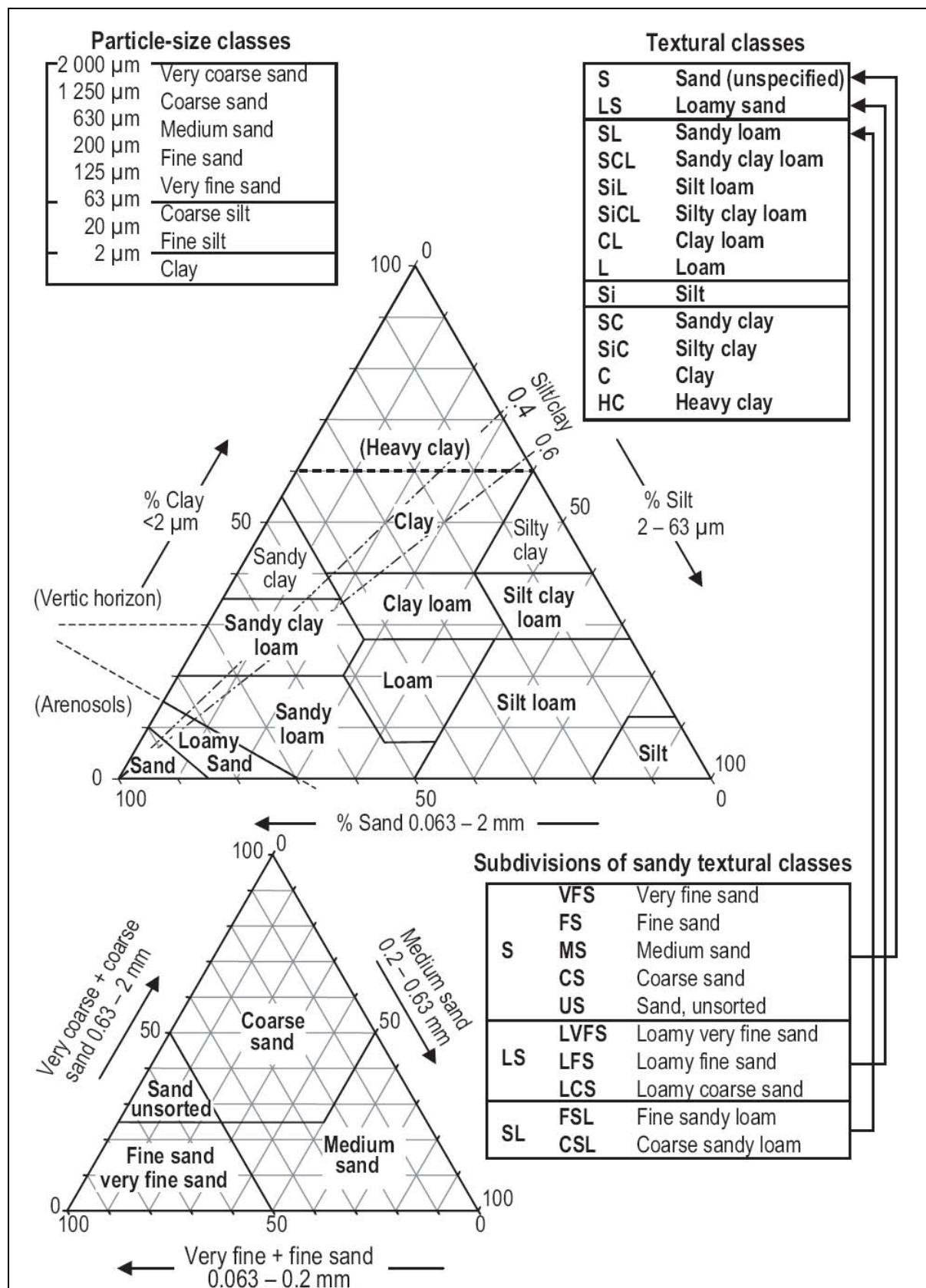
Clay	< 2	m
Silt	2 – 63	m
Sand	63 – 2000	m

For Level I, information on textural class for the mineral layers is mandatory too (though again only if not done in the first 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 6. Repetition of the determination of the granulometry is not required. For Level I, extra time and costs are minimised if estimated by finger test (described in Annex 6).

#### *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 1: 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 of the total mineral soil

#### *Definition*

Bulk density is defined as the mass of a unit volume of oven dry soil. The volume includes both solids and pores. In mineral soils without coarse fragments the bulk density of the total mineral soil is equal to the bulk density of the fine earth.

#### *Optional and mandatory parameters*

Three values of bulk density have to be reported mandatory for the mineral topsoil (0-10 cm, 10-20 cm and 20 – 40 cm) of non-stony soils. 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: five samples have to be taken with a minimal volume of 100 cm<sup>3</sup> 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). 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 normal profile description which follows the genetic layers.

### 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 7.

**Table 7:** 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 1, SA05 or (ii) the method used in Germany (see Annex 1, SA04).

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 first survey) 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 1: SA05.

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

Recent investigations (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 bulk density of both fine earth and coarse fragments has to be estimated / sampled in the field. Methods should be selected according to the prevailing conditions (i.e. 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 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. 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 fine earth referring to this volume.

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 ( $\theta$  in volume fraction,  $m^3 m^{-3}$ ) is determined at predefined matric potentials ( $\psi$ , in kPa). As indicated in Table 8, 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 8. The reference methods for all physical parameters are listed in Table 9.

**Table 8.** Overview of matric heads to assess for the determination of the SWRC

Matric potential $\psi$			Recommended instrument	Estimator	Equivalent pore size diameter	M/O
cm H <sub>2</sub> O	pF	kPa			Jurin's law, Hillel (1980)	
1	infinitely small	0	Pycnometer	$\approx \theta_{sat} = \text{water holding capacity} = \text{Total porosity}$	> 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'	7.0	-10 <sup>6</sup>	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 H<sub>2</sub>O or 1 cm H<sub>2</sub>O column = 0.097885 kPa
- 3) 100 kPa = 1 bar

**Table 9:** 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 <sup>3</sup> /m <sup>3</sup>
Bulk density	Oven drying at 105°C	kg/m <sup>3</sup>
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	kg/m <sup>2</sup> cm 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 10. 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.

Temporal changes are in the first place expected in the upper layers. Mandatory Level II parameters of the deeper layers (20-40 and 40-80 cm) that were already measured with the reference methods for the first survey, do not have to be determined again except for organic carbon that should be remeasured in the 20 to 40 cm layer. This means that if all the mandatory parameters of the 40-80 cm layer were assessed with the reference method, re-sampling of this layer is not required.

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  $\text{CaCl}_2$ -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 10:** Chemical and physical key soil parameters on the samples taken at fixed depths<sup>(1)</sup>

Parameter	Unit	Deci-mals	Level I						Level II					
			Organic Layer		Mineral Layer				Organic Layer		Mineral Layer			
			OL	OF+OH, H- <sup>(2)</sup>	0-10 cm	10-20 cm	20-40 cm	40-80 cm	OL	OF+OH, H <sup>(2)</sup>	0-10 cm	10-20 cm	20-40 cm	40-80 cm
<u>Physical soil parameter</u>														
Organic layer mass	kg/m <sup>2</sup>	2	O	M	-	-	-	-	O	M	-	-	-	-
Coarse fragments	vol %	0	-	-	M <sup>(3), (4)</sup>	M <sup>(3), (4)</sup>	M <sup>(3), (4)</sup>	O <sup>(4)</sup>	-	-	M	M <sup>(3), (4)</sup>	M <sup>(3), (4)</sup>	O <sup>(4)</sup>
Bulk density of the fine earth	kg/m <sup>3</sup>	0	-	-	M <sup>(3), (5), (6)</sup>	M <sup>(3), (5), (6)</sup>	M <sup>(3), (5), (6)</sup>	O	-	-	M <sup>(3), (5)</sup>	M <sup>(3), (5)</sup>	M <sup>(3), (5)</sup>	O
Particle size distribution (FAO, 1990)	-	-	-	-	M <sup>(3), (7)</sup>	M <sup>(3), (7)</sup>	O	O	-	-	M <sup>(3)</sup>	M <sup>(3)</sup>	M <sup>(3)</sup>	M <sup>(3)</sup>
Clay content	%	1	-	-	M <sup>(3), (7)</sup>	M <sup>(3), (7)</sup>	O	O	-	-	M <sup>(3)</sup>	M <sup>(3)</sup>	M <sup>(3)</sup>	M <sup>(3)</sup>
Silt Content	%	1	-	-	O	O	O	O	-	-	M <sup>(3)</sup>	M <sup>(3)</sup>	M <sup>(3)</sup>	M <sup>(3)</sup>
Sand Content	%	1	-	-	O	O	O	O	-	-	M <sup>(3)</sup>	M <sup>(3)</sup>	M <sup>(3)</sup>	M <sup>(3)</sup>
<u>Chemical soil parameter</u>														
pH(CaCl <sub>2</sub> )	-	2	-	M	M	M	O	O	-	M	M	M	M <sup>(3)</sup>	M <sup>(3)</sup>
pH(H <sub>2</sub> O)	-	2	-	O	O	O	O	O	-	O	O	O	O	O
Total organic carbon	g/kg	1	-	M	M	M	M	O	-	M	M	M	M	O
Total nitrogen	g/kg	1	-	M	M	M	O	O	-	M	M	M	O	O
Carbonates	g/kg	0	-	M <sup>(8)</sup>	M <sup>(8)</sup>	M <sup>(8)</sup>	O	O	-	M <sup>(8)</sup>	M <sup>(8)</sup>	M <sup>(8)</sup>	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 <sup>+</sup> , Exchangeable cations Al, Fe, Mn	cmol <sub>(+)</sub> /kg	2	-	M <sup>(9)</sup>	M <sup>(9)</sup>	M <sup>(9)</sup>	O	O	-	M <sup>(9)</sup>	M <sup>(9)</sup>	M <sup>(9)</sup>	M <sup>(3), (9)</sup>	M <sup>(3), (9)</sup>
Exchangeable cations Ca, Mg, K, Na	cmol <sub>(+)</sub> /kg	2	-	M <sup>(9)</sup>	M	M	O	O	-	M <sup>(9)</sup>	M	M	M <sup>(3)</sup>	M <sup>(3)</sup>
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 <sup>(3)</sup>	M <sup>(3)</sup>

<sup>1</sup> Abbreviations : M = mandatory parameter, O = optional parameter

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

<sup>3</sup> 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

<sup>4</sup> May be obtained by estimation or measurement

<sup>5</sup> Mandatory only in non-stony soils

<sup>6</sup> May be obtained by estimation, pedo-transfer function or measurement

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

<sup>8</sup> Only mandatory if pH(CaCl<sub>2</sub>) > 5.5 or in calcareous soils

<sup>9</sup> In calcareous soil, the measurement of this parameter is optional

This means that if all the mandatory parameters of the 40-80 cm layer were assessed with the reference method, re-sampling of this layer is not required.

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  $\text{CaCl}_2$ -extract is recommended.  $\text{pH}(\text{H}_2\text{O})$  has been made an optional parameter for reasons of comparability, as this is mostly used in literature.

### 5.2.2 Reference analytical methods

The full description of the reference methods is given in Annex 1.

Table 11 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 11:** Overview of reference methods for the chemical parameters

Parameter	Reference Analysis Method <sup>1</sup>			Unit <sup>2</sup>
	ISO	Extractant	Measurement method(s) <sup>3</sup>	
pH(CaCl <sub>2</sub> )	ISO 10390 (2005)	0.01 M CaCl <sub>2</sub>	pH-electrode	g/kg
pH(H <sub>2</sub> O)		H <sub>2</sub> O	pH-electrode	
Total nitrogen	ISO 13878 (1998)	-	Dry Combustion	g/kg
	ISO 11261 (1995)	-	Modified Kjeldahl	
Total organic carbon <sup>4</sup>	ISO 10694 (1995)	-	Dry Combustion at 900 °C	
Carbonates	ISO 10693 (1994)	HCl	Calcimeter	
P	ISO 11466 (1995)	Aqua Regia by reflux digestion	IC P	Colorimetry AAS
K, Ca, Mg, Mn				
Heavy metals: Cu, Cd, Pb, Zn			IC P	Cold vapour AAS
Other: Al, Fe, Cr, Ni, Na				
Hg			ICP	CNS - analyser
S				
Free Acidity (or sum of AC <sup>5</sup> ) and free H <sup>+</sup>	ISO 11254 (1994) modified	0.1 M BaCl <sub>2</sub>	titration to pH 7.8 or 'German' method	
Exchangeable Cations	ISO 11260 (1994) modified	0.1 M BaCl <sub>2</sub>	ICP	- FES
Al, Fe, Mn				
K, Ca, Mg, Na				
Reactive Fe and Al	ISRIC (2002)	Acid ammonium oxalate	AAS	ICP
Total Elements: Ca, Mg, Na, K, Al, Fe, Mn	ISO 14869-1 (2001)	HF or LiBO <sub>2</sub>	AAS	ICP

<sup>1</sup> Reference and full descriptions are given in Annex 1<sup>2</sup> Results have to be expressed on an oven dry basis<sup>3</sup> For the measurement of a number of parameters there are several alternatives for the equipment that can be used<sup>4</sup> Note that for total organic carbon a correction has to be made for total inorganic carbon (carbonates)<sup>5</sup> Alternative for the titration of the exchangeable acidity is the sum of the exchangeable Al, Fe, Mn and free H<sup>+</sup>

## 5.3 Data quality requirements

The quality of the soil analytical data is controlled by the regular organisation of Interlaboratory Comparisons (ring tests) by the Forest Soil Co-ordinating Centre. 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 3.3.2.1a "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, Tables 3.4.1.2.2 for the tolerable limits of the measured parameters in the FSCC Interlaboratory Comparisons.

Tolerable limits for the determination of the SWRC for laboratory performance will be derived from the reproducibility data gained by performing interlaboratory physical soil ringtests.

All reported values should have been measured according to the methods described in Annex 1.

### 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 FSCC Interlaboratory Comparisons.

The soil chemical Interlaboratory Comparisons 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 and explanatory items are found on the ICP Forests web page, at <http://www.icp-forests.org/Manual.htm>. 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 using the submission form "XX2011SO.LQA" or updated version valid for year of submission.

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, Hfs horizons or as described in Annex 7 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 QA/QC procedures in the laboratory that are given in Manual Part III on QA/QC in laboratories (§ 3.3.2.2: Cross checks between soil variables).

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

All validated data should be sent to each national focal centre and to the European 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<sup>+</sup>.

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  $\theta_r$ ,  $\theta_s$  and empirical constants  $a$ ,  $n$  and  $m = 1 - 1/n$ . Calculation of these parameters can be done using the public domain RETC programme which may be downloaded from: [http://www.pc-progress.cz/Pg\\_RetC.htm](http://www.pc-progress.cz/Pg_RetC.htm) or obtained from the FSCC. This software enables to predict Ksat from the SWRC measurements. 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. 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 3.2.3 of the Manual Part III on Quality Assurance and Quality Control in Laboratories.

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## **Annex 1**

### **Methods for Soil Analysis**



## **Soil Analysis Method 1 (SA01) Pre-treatment of Samples**

<b>Pre-treatment of Samples</b>	
Method sheet	SA01
Reference methods	ISO 11464
Method suitable for	Organic Layer; Mineral Layer

## I. Relevance in ICP Forests

All samples (organic and mineral) have to be prepared according to the standard methodology in order to maintain comparability among participating countries.

Priority	Level I	Level II
<b>Organic Layer</b>		
OL	<i>Optional</i>	<i>Optional</i>
OF+OH, H-layers	<i>Mandatory</i>	<i>Mandatory</i>
<b>Mineral layer</b>		
0- 10 cm <sup>1</sup>	<i>Mandatory</i>	<i>Mandatory</i>
10 – 20 cm	<i>Mandatory</i>	<i>Mandatory</i>
20 – 40 cm	<i>Mandatory</i>	<i>Mandatory</i>
40 – 80 cm	<i>Optional</i>	<i>Mandatory</i>

<sup>1</sup> Optionally this layer may be split in two layers: 0 – 5 cm AND 5 – 10 cm

## II. Principle

### a. Organic layer

After removal of living material (such as mosses, roots, etc.) and objects > 2 cm, collected samples (preferably not less than 500 g fresh material) should be transported to the laboratory as soon as possible and should be air dried or dried at a temperature of 40 °C. They can then be stored until analysis. The sample is subsequently crushed or milled to size < 2 mm.

When the samples are bulked in the field and only a subsample is taken to the laboratory, the fresh mass (kg/m<sup>2</sup>) of each organic sublayer should be measured in the field. Further it is strongly recommended to measure the thickness of each organic sublayer in each subsample in the field. Firstly, because the horizon thickness (in cm in terms of the upper and lower limit) is mandatory to report in the profile description file. Secondly it is useful as a cross check.

### b. Mineral layer

After removal of living material (such as mosses, roots, etc.) and objects > 2 cm, collected samples (preferably not less than 500 g fresh soil) should be transported to the laboratory as soon as possible and should be air dried or dried at a temperature of 40 °C. They can then be stored until analysis.

Living macroscopic roots and all material, mineral and organic, with a diameter larger than 2 mm, should be removed from the samples by dry or wet sieving. The particles not passing the 2-mm sieve (after crushing), may be weighed separately for the determination of the coarse fragments content (SA05). The fraction smaller than 2 mm is used for the soil analysis. The mineral soil samples should not be milled. Only sieving above a 2 mm sieve is allowed. No further grinding will be allowed except for the analysis of Carbonate content (SA07), Total Organic Carbon (SA08), total Nitrogen (SA09) and Total Elements (SA12).

The sample materials for storage should be kept without preservative under normal room conditions with minimal temperature and humidity fluctuations, shielded from incident light.

### III. Apparatus

Drying oven.

Crusher, mill, mortar and pestle.

Plate sieve, mesh sieve

### IV. Reagents

No reagents.

### V. Procedure

#### *Drying*

Spread the material in a layer not thicker than 15 mm. If necessary, the sample is crushed while still damp and friable and again after drying. Dry the complete sample in a drying oven at a temperature of 40 °C, until the loss in mass of the sample is not greater than 5 % (m/m) per 24 h. Break down the size of larger clods (greater than 15 mm) to accelerate the drying process.

#### *Removal of fraction < 2 mm*

Remove stones and large objects by hand picking and sieving (< 2 mm). Minimise the amount of fine material adhered. Weigh separately the fraction not passing the 2 mm sieve for determination of coarse fragment content. Crush (not ground) the clods greater than 2 mm taking care that crushing of original particles is minimised. Homogenise the < 2 mm fraction.

#### *Sieving and Milling*

The organic sample is crushed or milled to size < 2 mm.

The mineral soil samples should not be milled. Only sieving above a 2 mm sieve is allowed. No further grinding will be allowed except for the analysis of Total Organic Carbon (SA08), Total Nitrogen (SA09) and Total Elements (SA12).

#### *Subsampling*

For the preparation of an analysis subsample, split up (by hand, using a sample divider or by mechanical subsampling) the sample into representative portions until the required sample number and sample size is obtained.

## **VI. Calculation**

No calculations.

## **VII. Report**

The mineral fractions (> 2 mm) obtained after sieving with a 2 mm sieve may be used for determination of coarse fragments (SA05).

## **VIII. Reference**

ISO 11464. 1994. Soil Quality – Pretreatment of samples for physico-chemical analysis.  
International Organization for Standardization. Geneva, Switzerland. 9 p. (available at  
[www.iso.ch](http://www.iso.ch))

## **Soil Analysis Method 2 (SA02): Determination of Soil Moisture Content**

<b>Soil Moisture Content</b>	
Method sheet	SA02
Reference methods	ISO 11465
Method suitable for	Organic Layer; Mineral Layer

## I. Relevance in ICP Forests

Recalculation of results obtained by lab analysis to "oven-dry mass".

Priority	Level I	Level II
<b>Organic Layer</b>		
OL	Optional	Optional
OF+OH, H-layers	Mandatory	Mandatory
<b>Mineral layer</b>		
0- 10 cm <sup>1</sup>	Mandatory	Mandatory
10 – 20 cm	Mandatory	Mandatory
20 – 40 cm	Mandatory	Mandatory
40 – 80 cm	Optional	Mandatory

<sup>1</sup> Optionally this layer may be split in two layers: 0 – 5 cm AND 5 – 10 cm

## II. Principle

Calculation and reporting of the results of soil analysis is done on basis of "oven-dry" soil. The moisture content of air-dry soil is determined prior to soil analysis. To recalculate the analysis results on dry mass basis, the moisture content of the sample has to be determined by oven-drying a sample to constant mass. The difference in mass is used to calculate water content on a mass basis.

## III. Apparatus

Moisture tins or flasks (25 – 100 ml) with closely fitting lid

Drying oven

Analytical balance (accuracy 0.001 g)

*Note: The use of an automated apparatus for measuring soil moisture content is allowed as long as it is based on the same principle.*

## IV. Reagents

No reagents.

## V. Procedure

**Mineral Layer:** Transfer 5-15 g air-dried fine earth (fraction < 2 mm) to a dried, tared moisture tin and weigh. Dry at 105±5 °C (lid removed) until constant mass is reached.

**Organic Layer :** Transfer 5 – 10 g air dried organic layer material to a dried, tared moisture tin and weigh. Dry at 105 °C (lid removed) for 24 hours.

Remove tin from oven, close with lid, cool in desiccator and weigh.

## VI. Calculation

The moisture content in mass percentage is obtained by :

$$\text{Moist \%} = \frac{A - B}{B - \text{tare tin}} * 100$$

Where:

A : Mass of tared moisture tin and air-dried soil sample

B : Mass of tared moisture tin and oven-dried soil sample

The corresponding moisture correction factor for analytical results or for amount of sample to be weighed in for analysis is:

$$\text{moisture correction factor(MCF)} = \frac{100 + \text{moist \%}}{100}$$

*Note: when reporting the results of Carbonate Content (SA07), Total Organic Carbon (SA08), Total Nitrogen (SA09), Exchangeable acidity, Free H<sup>+</sup>, Exchangeable elements (SA10), Aqua Regia Extractable elements (SA11), Total elements (SA12), Acid Oxalate Extractable Fe and Al (SA13), the results on air-dry basis should be multiplied by the moisture correction factor (MCF) to obtain the result on oven-dry basis.*

## VII. Report

Report moisture content (in %) with 1 decimal place.

## VIII. Reference

ISO 11465. 1993. Soil Quality – Determination of dry matter and water content on a mass basis – Gravimetric method. International Organization for Standardization. Geneva, Switzerland. 3 p. (available at [www.iso.ch](http://www.iso.ch))



### **Soil Analysis Method 3 (SA03): Determination of Particle Size Distribution**

<b>Particle Size Distribution</b>	
Method sheet	SA03
Reference methods	ISO 11277
Method suitable for	Mineral Layer

## I. Relevance in ICP Forests

Particle Size Distribution : USDA-FAO texture Classification and Clay Percentage

Priority	Level I	Level II
<b>Organic Layer</b>	-	-
<b>Mineral layer</b>		
0 – 10 cm	Mandatory <sup>1, 2</sup>	Mandatory <sup>1</sup>
10 – 20 cm	Mandatory <sup>1, 2</sup>	Mandatory <sup>1</sup>
20 – 40 cm	Optional	Mandatory <sup>1</sup>
40 – 80 cm	Optional	Mandatory <sup>1</sup>

<sup>1</sup> if not determined in the first soil survey

<sup>2</sup> an estimation of clay content based on finger test is allowed

Particle Size Distribution : Silt and Sand Percentage

Priority	Level I	Level II
<b>Organic Layer</b>	-	-
<b>Mineral layer</b>		
0 – 10 cm	Optional	Mandatory <sup>1</sup>
10 – 20 cm	Optional	Mandatory <sup>1</sup>
20 – 40 cm	Optional	Mandatory <sup>1</sup>
40 – 80 cm	Optional	Mandatory <sup>1</sup>

<sup>1</sup> if not determined in the first soil survey

## II. Principle

Separation of the mineral part of the soil into various size fractions and determination of the proportion of these fractions. The analysis includes all soil material, i.e. including gravel and coarser material, but the procedure below is applied to the fine earth fraction (< 2 mm) only. Of

paramount importance in this analysis is the pretreatment of the sample aimed at complete dispersion of the primary particles. Therefore, generally, cementing materials (usually of secondary origin) such as organic matter, salts, iron oxides and carbonates such as calcium carbonate are removed. After shaking with a dispersing agent, sand ( $63 \mu\text{m}$ - $2 \text{ mm}$ ) is separated from clay and silt with a  $63 \mu\text{m}$  sieve (wet sieving). The clay ( $< 2 \mu\text{m}$ ) and silt ( $2$ - $63 \mu\text{m}$ ) fractions are determined by the pipette method (sedimentation).

### III. Apparatus

Sampling pipette (10 to 50 ml) with safety bulb and water reservoir, held in frame

Constant temperature room or thermoregulated bath ( $20 - 30^\circ\text{C}$     $0.5^\circ\text{C}$ )

Glass sedimentation cylinders (approx. diam. 50 mm, approx. length 350 mm) graduated 500 ml volume with rubber bungs or stirrer

Stirrer and rod

Glass weighing vessels (with masses known to 0.0001 g)

Mechanical shaker (30 – 60 revolutions/min)

Sieves (2 mm –  $63 \mu\text{m}$ )

Balance (accuracy 0.0001 g)

Drying oven

Stopwatch (accuracy 1 s)

Glass filter funnel capable of holding the  $63 \mu\text{m}$  sieve

Wash bottle

Desiccator

650 ml glass beaker with cover glass, 100 ml measuring cylinder, 25 ml pipette

Hot plate or bunsen burner

Electrical conductivity meter (accuracy 0.1 dS/m)

Optional: Centrifuge and 300 ml centrifuge bottle

### IV. Reagents

Hydrogen peroxide ( $\text{H}_2\text{O}_2$ ), 30% volume fraction.

Dispersing agent: 3.3 % sodium hexametaphosphate and 0.7 % soda solution:

Dissolve 33 g sodium hexametaphosphate ( $\text{NaPO}_3)_6$  and 7 g soda ( $\text{Na}_2\text{CO}_3$ ) in water in a 1 l volumetric flask and make to volume. Both chemicals should be dried overnight at  $105^\circ\text{C}$  prior to use. This solution is unstable and shall be replaced after one month.

Antifoaming agent (preferably octan-2-ol, alternatives are ethanol or methanol)

Calcium chloride solution ( $\text{CaCl}_2$ ), conc. 1 mol/l

Hydrochloric acid ( $\text{HCl}$ ), conc. 1 mol/l

## V. Procedure

### *Test sample*

Depending on the soil type, weigh 10 (clay) to 30 g (sand) air-dried soil (fraction < 2 mm). Place the sample in the 650 ml glass beaker or 300 ml centrifuge bottle.

### *Destruction of organic matter*

Add 30 ml water to the test sample (add if necessary a few drops of octan-2-ol to allow thoroughly wetting). Add 30 ml of the 30 % hydrogen peroxide solution and mix using the glass or plastic rod (add if necessary a few drops of octan-2-ol to control foaming). Cover and leave overnight. The next day, place the vessel on a hot plate or bunsen burner and warm. Control foaming with octan-2-ol and stir frequently. To avoid drying out, add water if necessary. Bring the suspension to a gentle boil until all signs of bubbling due to the decomposition of hydrogen peroxide have ceased. If undecomposed organic material is still present, cool the beaker and repeat the treatment with hydrogen peroxide.

If using a centrifuge bring the volume to 150 – 200 ml by addition of water. Centrifuge the bottle until obtaining a clear supernatant (recommended 15 min at a minimum relative centrifugal force (RCF) of 400 g) and remove this supernatant by decanting or by using a suction device.

If a centrifuge is not available the mineral residues may be flocculated by adding 25 ml of 1 mol/l calcium chloride solution, stirring and bringing to about 250 ml with water. Let stand until the supernatant is clear, then siphon or decant this from the residue. Add another 250 ml of water and repeat the washing procedure until the dark residues of the decomposed organic matter have gone (if using this method, take care to check the electrical conductivity (next step) before adding the salt).

### *Removal of soluble salts and gypsum*

After destruction of organic matter add water until obtaining a soil:water ratio of 1:4 – 1:6 (v:v). Shake for 1 h using a shaking machine. Centrifuge to obtain a clear supernatant and measure electrical conductivity ( $E_c$ ) on this supernatant. If  $E_c > 0.4$  dS/m soluble salts and gypsum is present in considerable amounts and have to be removed. Remove the supernatant, add 250 ml water and shake for 1 h. Centrifuge and measure electrical conductivity again. Repeat this washing procedure until  $E_c < 0.4$  dS/m.

### *Removal of carbonates*

A distinction is made on basis of the presence or absence of calcium carbonate:

- (1)      Calcareous soils:  $\text{pH}(\text{H}_2\text{O}) > 6.8$
- (2)      Non-calcareous soils:  $\text{pH}(\text{H}_2\text{O}) < 6.8$

Where the carbonate content is greater than about 2 % mass fraction, add to the washed, centrifuged soil (above) 4 ml of 1 mol/l hydrochloric acid for each percent of carbonate, plus an excess of 25 ml of acid. Make up to about 250 ml with water, and place the suspension on the water bath at about 80 °C for 15 min, stirring the suspension from time to time. Leave the suspension to stand overnight. If the soil flocculates sufficiently to leave a perfectly clear supernatant, then this can be siphoned off or decanted, otherwise centrifugation and decantation will be necessary. Repeat the washing and decantation with water until the  $E_c$  of the supernatant is less than 0,4 dS/m.

If the carbonate content is less than about 2 % mass fraction, then only an initial 25 ml of 1 mol/l hydrochloric acid solution is required. It is recommended, therefore, that 20 ml of 1 mol/l calcium chloride solution is added at the same time as the acid. The rest of the procedure is identical as for a higher carbonate content.

**Note:** if the carbonate content is that high that the results of the particle size distribution become unreliable, this should be mentioned in the Data Accompanying Report.

#### *Dispersion*

Add sufficient water to the vessel so that the total volume is between 150 ml and 200 ml, shake the contents until all the soil is in suspension, and add 25 ml of dispersing agent from a pipette. Shake the bottle for 18 h on the end-over-end shaker.

#### *Wet sieving at 63 µm*

Place a 63 µm aperture sieve in the large glass funnel, and place the funnel in the stand so that the neck of the funnel is inside one of the 500 ml sedimentation tubes. Transfer the dispersed suspension from the centrifuge bottle quantitatively onto the sieve, and wash the soil using a jet of water from the wash-bottle until the water runs clear. The total volume of the washings should not exceed 500 ml.

Remove the sieve from the funnel and wash the residue on the sieve into an evaporating dish by means of a gentle spray from the wash-bottle. To alleviate sieve blockage, use the glass or plastic rod and rubber sleeve. Place this dish in an oven between 105 °C and 110 °C until the residue is dry. Record the mass to 0.0001 g ( $mf_s$ ).

Wash any particles adhering to the inside of the funnel into the sedimentation tube. Make up the suspension in the sedimentation tube to 500 ml with water.

#### *Calibration*

##### *Calibration sampling pipette*

Clean and dry the pipette thoroughly and immerse the tip in water. Draw water into the pipette into the safety bulb. Drain off the water in the safety bulb through the outlet tube. Drain the pipette into a weighing bottle of known mass, and determine the internal volume of the pipette. Repeat this exercise three times and take the average of the three volumes as the internal volume of the pipette to the nearest 0.05 ml ( $V_c$  ml).

##### *Calibration dispersing agent*

Pipette 25 ml of dispersing agent solution into one of the glass sedimentation tubes, and fill the tube to the 500 ml mark with water. Mix the contents of the tube thoroughly. Place the tube in the constant temperature environment, and leave the tube for at least 1 h. Between any of the times at which samples may be taken from the sampling tube (Table SA03-1), take a sample ( $V_c$  ml) of the dispersing agent solution from the sedimentation tube using the sampling pipette. Drain the pipette into a weighing vessel of known mass, and dry the contents of the vessel between 105 °C and 110 °C. Allow the vessel to cool in the desiccator and determine the mass of the residue in the vessel to 0.0001 g ( $m_r$ ).

Follow this procedure each time a new batch of dispersing agent is prepared.

#### *Sedimentation*

Place the sedimentation tube in the constant temperature environment. Agitate (at least 30 times/min for a minimum of 2 min) the contents of the sedimentation tube vigorously, either by

means of the stirrer, or by inserting a bung in the tube, followed by end-over-end shaking. Replace the tube upright in the constant-temperature environment and start the timer.

About 15 s before a sample is to be taken (Table SA03-1), lower the pipette, with the tap of the safety bulb closed, vertically into the soil suspension, and centrally in the sedimentation tube, until the tip is the appropriate depth ( $\pm 1$  mm) below the suspension surface (Table SA03-1). Take care to disturb the suspension as little as possible, and complete the operation within about 10 s. Open the tap of the safety bulb and withdraw a sample of the suspension such that the pipette and a part of the safety bulb are full. This sampling operation shall take about 10 s. Withdraw the pipette from the suspension so that the tip of the pipette is clear of the top of the sedimentation tube. Run the surplus present in the safety bulb into a small beaker by the outlet tube. Wash with water from the water reservoir until no suspension remains in this part of the system.

Place a weighing vessel of known mass (to 0.0001 g) under the tip of the pipette and open the tap so that the contents of the pipette are delivered to the vessel. Wash any suspension left on the inner walls of the pipette into the vessel by allowing water from the water reservoir to run through the system. Place the weighing vessel and contents in the oven between 105 °C and 110 °C, and evaporate to dryness. Cool the vessel in the desiccator, weigh the vessel and its contents to the nearest 0.0001 g, and determine the mass of the residue the nearest 0.0001 g ( $ms_1$ ). Clean the outside of the pipette of any adhering sediment, and take the other sample (fraction < 2 µm), in accordance with the times given in Table SA03-1, using the same pipetting procedure given above. Call the additional sample masse  $ms_2$ .

**Table SA03-1:** Pipette sampling times and fraction at different temperatures

Temperature (°C)	Time (after mixing) of starting sampling operation	
	Fraction : < 63 µm	Fraction : < 2 µm
	Sampling depth 200 mm $\pm$ 1 mm	Sampling depth 100 mm $\pm$ 1 mm
20	56 s	7 h 44 min 16 s
21	54 s	7 h 34 min 04 s
22	53 s	7 h 23 min 53 s
23	52 s	7 h 13 min 13 s
24	51 s	7 h 03 min 02 s
25	49 s	6 h 52 min 50 s
26	48 s	6 h 44 min 02 s
27	47 s	6 h 35 min 42 s
28	46 s	6 h 26 min 53 s
29	45 s	6 h 18 min 33 s
30	44 s	6 h 09 min 45 s

## VI. Calculation

### *Fractions < 63 µm*

Calculate the mass of solids in suspension in 500 ml ( $mf_1$ ,  $mf_2$ ) in grams, for each pipette sampling time from the equation:

$$\text{Mass } < 63 \mu\text{m} \text{ in 500 ml} : mf_1 = ms_1 (500/V_d)$$

$$\text{Mass } < 2 \mu\text{m} \text{ in 500 ml} : mf_2 = ms_2 (500/V_d)$$

where:

$mf_x$  is the mass (g) of solid in suspension in 500 ml;  
 $ms_x$  is the mass (g) of material from the  $x$ th pipette sampling;  
 $V_c$  is the calibrated volume of the pipette.

Each fraction however, still contains a part of dispersing agent, which has to be corrected. The mass of solid material in 500 ml of dispersant solution,  $m_d$ , in grams, is given by:

$$\text{Mass dispersing agent in 500 ml: } m_d = m_r (500/V_c)$$

where:

$m_r$  is the mass of residue, in grams;  
 $V_c$  is the calibrated volume of the pipette, in millilitres.

This gives the final fraction masses:

Clay	Mass fraction $< 2 \mu\text{m}$	=	mf <sub>2</sub> - m <sub>d</sub>
Silt	Mass fraction $2 - 63 \mu\text{m}$	=	mf <sub>1</sub> - mf <sub>2</sub>

#### *Fraction 63 μm - 2 mm*

Mass of the fraction  $63 \mu\text{m} - 2 \text{ mm} = mf_s$

#### *Proportion of fraction*

The method of calculation assumes that the sample mass is the sum of the constituent fractions, and not the mass of the test sample. The mass of sample  $< 2 \text{ mm}$  is thus the sum of the masses of the fractions obtained by wet sieving at  $63 \mu\text{m}$  and the masses of the fractions obtained by calculation. Denote this total sample mass as  $m_t$  in grams.

Calculate the proportion in each fraction  $< 2 \text{ mm}$  as follows:

Proportions = mass of fraction/ $m_t$

## **VII. Report**

It is an agreed convention that the percentage of each particle size grade is reported on the basis of oven-dry soil free of organic matter (1 decimal place).

*Note: With this calculation, the clay, silt and sand fractions are obtained in percentage of the sum of the constituent fractions (after removal of carbonates and organic matter).*

USDA-FAO texture classification is based on the USDA-FAO textural triangle (FAO, 1990) as shown in Figure 1.

## VIII. References

- ISO 11277. 1998. Soil Quality – Determination of particle size distribution in mineral soil material – Method by sieving and sedimentation. International Organization for Standardization. Geneva, Switzerland. 30 p. (available at [www.iso.ch](http://www.iso.ch))
- FAO. 1990. Guidelines for soil description, 3<sup>rd</sup> (revised) edition.

## **Soil Analysis Method 4 (SA04): Determination of Bulk Density**

<b>Bulk Density</b>	
Method sheet	SA04
Reference methods	ISO 11272
Method suitable for	Mineral Layer

## I. Relevance in ICP Forests

Priority	Level I	Level II
Organic Layer	-	-
Mineral layer		
0 – 10 cm	Mandatory <sup>1,2,3</sup>	Mandatory <sup>2,3</sup>
10 – 20 cm	Mandatory <sup>1,2,3</sup>	Mandatory <sup>2,3</sup>
20 – 40 cm	Mandatory <sup>1,2,3</sup>	Mandatory <sup>2,3</sup>
40 – 80 cm	Optional	Optional

<sup>1</sup> may also be obtained by using pedo-transfer functions

<sup>2</sup> only mandatory in non-stony soils

<sup>3</sup> in case of re-assessment (if the parameter was already measured according to the reference method in a previous survey, the measurement is optional)

## II. Principle

The dry bulk density (BD) is the ratio between the mass of oven dry soil material and the volume of the undisturbed fresh sample. The ISO defines dry bulk density as the ratio of the oven-dry mass of the solids to the volume (the bulk volume includes the volume of the solids and of the pore space) of the soil.

### *Non-gravely soils (when coarse fragments content < 5%)*

Several methods can be applied for the determination of bulk density, going from simple methods such as digging out holes of known volume to sophisticated gamma radiometry methods. The recommended method (core method) uses steel cylinders of known volume (100 cm<sup>3</sup>, 400 cm<sup>3</sup>) that are driven in the soil vertically or horizontally by percussion. Sampling large volumes results in smaller relative errors but requires heavy equipment. The method cannot be used if stones or large roots are present or when the soil is too dry or too hard.

### *Soils with high stone or root content or when the soil is too dry or too hard*

In these conditions it is advised to use measuring methods based on the following principle (excavation method): a hole on a horizontal surface is dug and then filled with a material with a known density (e.g. sand which packs to a calibrated volume or water separated from the soil material by an elastic membrane). The obtained soil from the hole, is dried to remove the water and the dry mass is weighed. Methods measuring the volume of clods or aggregates should be avoided because they tend to underestimate macroporosity.

The volumetric percentage of the coarse fragments needs to be determined in order to calculate the bulk density of the fine earth.

### *Stony soils*

Soils with a high content of gravel (0.2 – 6 cm) and/or the presence of stones (6 – 20 cm) and boulders (> 20 cm), have a low volume of fine earth. Core samplers normally used in forest monitoring are not able to representatively collect stones or large portions of coarse fragments in the field. In these cases, the above recommended excavation method will produce good results but may be considered very expensive, time-consuming and destructive. So, alternatively, a combined approach is described where the quantity of bulk density of both fine earth and coarse fragments (SA05) has to be estimated / sampled in the field.

Methods are according to the prevailing conditions (i.e. coarse fragment content and size) at each individual sampling site:

In case of coarse fragment content of more than 5 %, the fine earth fraction must be sieved and weighed. Its volume must then be determined either directly or indirectly by establishing the coarse fragment volume. Furthermore, the density of the coarse fragments (specific weight) must be known or established.

In case of content of coarse fragments > 20 mm, representative sampling is no longer possible with a core sampler. Then the coarse fragment content must be determined by additional sampling using a shovel or spade and/or estimations in the soil profile.

In case of coarse fragments content of > 60 mm, representative volume sampling is not possible and sampling with mini-core samplers is combined with an estimation in the profile pit.

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.

*Note: The determination of the bulk density of the fine earth is incorrect when the sample contains significant portions of roots in addition to the coarse fragment portions. In these cases, this must be corrected.*

### III. Apparatus

Core sample holders, thin-walled metal cylinders with a volume of 100 cm<sup>3</sup> to 400 cm<sup>3</sup>, a steel cap for driving into the soil, and a driver (or root auger, hollow stem auger, AMS core sampler with liner or alike)

Oven (heated and ventilated, temperature 105 ± 2 °C)

Desiccator

Balance (accuracy 1/1000 of measured value).

Spade, shovel

Metal sieves (2 mm, 20 mm, 60 mm)

### IV. Reagents

No reagents.

### V. Procedure

**Case 1:** Non-gravely soils (when coarse fragments content < 5%)

Press or drive a core sample holder of known volume without deflection and compaction into either a vertical or horizontal soil surface far enough to fill the sampler. Carefully remove the sample holder and its contents to preserve the natural structure, and trim the soil extending beyond each end of the sample holder with a straight-edged knife or sharp spatula. The soil sample volume is thus equal to the volume of the sample holder. Take at least five core samples from each soil layer. Place the holders containing the samples in an oven at 105 °C until constant mass is reached (minimum 48 h). Remove the samples from the oven and allow them to cool in the desiccator. Weigh the samples on the balance immediately after removal from the desiccator ( $m_i$ ). Control mass is reached when the differences in successive weighings of the cooled sample, at intervals of 4 h, do not exceed 0,01% of the original mass of the sample.

**Case 2:** Mineral soil with a coarse fragment content of more than 5% that can be sampled with a core sampler or any other representative sampler (coarse fragments < 20 mm)

The mineral soil sample is collected in the field with core samplers from the undisturbed soil. In the laboratory the sample is then dried at a temperature of 105 °C for at least 48 hours to constant mass and weighed.

The sample is then passed through a 2 mm metal sieve and the sieve residue washed in order to break down clumpy fine earth material and to rinse off earth adhering to the stones. The washed sieve residue (= coarse fragment portion) is shaken into a beaker, dried at a temperature of 105°C in a drying oven and then weighed.

**Case 3:** Mineral soil, which cannot be sampled with a core sampler or any other representative sampler (coarse fragments > 20 mm)

**Case 3.1.:** Combination of representative volume sampling with a core sampler and estimation of coarse fragments > 20 mm

The mineral soil sample is collected in the field with core samplers from the undisturbed soil. In the laboratory the sample is dried at a temperature of 105 °C for at least 48 hours to constant mass and weighed.

The sample is passed through a 2 mm metal sieve and the sieve residue washed in order to break down clumpy fine earth material and to rinse off earth adhering to the stones. The washed sieve residue (= coarse fragment portion) is shaken into a beaker, dried at a temperature of 105 °C in a drying oven and then weighed. After that, the sieve residue is passed through a 20 mm sieve and the 2 – 20 mm sieve fraction (fine and medium gravel) weighed.

For the coarse fragment portion > 20 mm an estimation from the profile description must be available.

**Case 3.2.:** Combination of representative volume sampling with a core sampler, disturbed sample and estimation of coarse fragments more than 60 mm at the profile

The mineral soil sample is collected in the field with a core sampler from the undisturbed soil. In addition, a larger sample volume, which must be representative for the coarse fragment fraction 2 – 60 mm (gravel), is collected with a shovel or a spade. In the laboratory the two samples are then dried at a temperature of 105 °C for at least 48 hours to constant mass and weighed.

The core sample is then passed through a 2 mm metal sieve and the sieve residue is washed in order to break down clumpy fine earth material and wash off earth adhering to the stones. The washed sieve residue (= coarse fragment portion) is shaken into a beaker, dried in a drying oven at a temperature of 105 °C and then weighed.

The spade sample is also dried at a temperature of 105 °C to constant mass and then weighed. The spade sample is then passed through a 2 mm sieve and the sieve residue through a 60 mm sieve. The coarse fragment fraction 2 – 60 mm obtained in this way is weighed. For the coarse fragment content > 60 mm an estimation from the profile description must be available.

**Case 4:** Representative volume sampling not possible, Sampling with mini-core samplers

With core sampler caps or mini-core samplers ( $n \geq 5$ ) several samples are taken from the undisturbed soil. In addition, a larger sample volume, which must be representative for the coarse fragment fraction 2 – 60 mm, is collected with a shovel or a spade.

In the laboratory the core sampler caps together with their contents are dried at a temperature of 105 °C for at least 48 hours to constant mass and then weighed together. The empty mass of the core sampler caps is then deducted from the total mass.

The spade sample is dried at a temperature of 105 °C for at least 48 hours to constant mass and then weighed. The sample is then passed through a 2 mm sieve and the sieve residue through a 6 mm sieve. The sieve residue is then passed through a 60 mm sieve as well. The fractions obtained < 2 mm (fine earth), 2 – 6 mm (fine gravel) and 6 – 60 mm (medium and coarse gravel) are weighed.

*Alternative to the combined approach of case 2 till case 4 in soils with high stone or root content or if the soil is too dry or too hard*

In case of gravelly or stony soils an alternative excavation method consist of excavating a quantity of soil, drying and weighing it, and determining the volume of the excavation by filling it with sand (cf. ISO 11272 – **excavation method**). Note that the excavation method measures the total dry bulk density

## VI. Calculation

**Case 1:** Non-gravely soils (when coarse fragments content < 5%)

In case of measurements, the bulk density of the fine earth ( $BD_{fe}$ ) is approximately equal to the bulk density of total soil. The bulk density ( $BD_s$ ) the for non-gravely soils is calculated as follows:

$$BD_s = BD_{fe} = \frac{M_s}{V_s} \quad (\text{equation SA04.01})$$

where:

$BD_s$	=	Bulk Density of the sample ( $\text{kg/m}^3$ )
$BD_{fe}$	=	Bulk Density of the fine earth ( $\text{kg/m}^3$ )
$M_s$	=	Dry Mass of the sample (kg)
$V_s$	=	Volume of the sample ( $\text{m}^3$ )

**Case 2:** Mineral soil with a coarse fragment content of more than 5% that can be sampled with a core sampler or any other representative sampler (coarse fragments < 20 mm)

In case of measurement with a core sampler, the bulk density of the fine earth of gravely soils ( $BD_{fe}$ ) is calculated as follows:

$$BD_{fe} = \frac{M_{fe}}{V_{fe}} = \frac{M_s - M_{cf}}{V_s - V_{cf}} = \frac{M_s - M_{cf}}{V_s - \frac{M_{cf}}{\rho_{cf}}} \quad (\text{equation SA04.02})$$

where:

$BD_{fe}$	=	Bulk density of the fine earth ( $\text{kg/m}^3$ )
$M_{fe}$	=	Dry Mass of the fine earth taken with core sampler (kg)
$V_{fe}$	=	Volume of the undisturbed fine earth ( $\text{m}^3$ )
$M_s$	=	Dry Mass of the soil sample with gravel taken with core sampler (kg)
$M_{cf}$	=	Dry Mass of the coarse fragments taken with the core sampler (kg)

$V_s$  = Volume of core sampler ( $\text{m}^3$ )

$V_{cf}$  = Volume of the coarse fragments taken with the core sampler (kg)

$\rho_{cf}$  = Density of the coarse fragments (approximated by 2650 kg/ $\text{m}^3$ )

The fine earth stock (FES) is the amount (kg) of fine earth in the soil layer under consideration expressed per ha. In stony soils, a correction for the volume of coarse fragments is required. It is calculated as follows:

$$FES = BD_{fe} \times d \times 10 \times \left(1 - \frac{V_{cf}}{V_s}\right) = BD_{fe} \times d \times 10 \times \left(1 - \frac{M_{cf}}{\rho_{cf} \times V_s}\right) \quad (\text{equation SA04.03})$$

where:

FES = Fine earth stock (t/ha)

$BD_{fe}$  = Bulk density of fine earth ( $\text{kg}/\text{m}^3$ )

d = Thickness of the sampled layer (m)

$V_{cf}$  = Volume of coarse fragment taken with core sampler (respectively core of root auger) ( $\text{m}^3$ )

$M_{cf}$  = Dry Mass of coarse fragment taken with core sampler (respectively core of root auger) (kg)

$\rho_{cf}$  = Density of the coarse fragments (approximated by 2650 kg/ $\text{m}^3$ )

$V_s$  = Volume of core sampler ( $\text{m}^3$ )

Notes:

If the core sampler sample cakes strongly as a consequence of drying, it might make sense to pulverise the sample with a crusher prior to sieving. The big stones should be removed beforehand..

In the case of non-cohesive soil (sand), there is no need to wash or dry the stones.

**Case 3:** Mineral soil, which cannot be sampled with a core sampler or any other representative sampler (coarse fragments > 20 mm)

**Case 3.1.** Combination of representative volume sampling with a core sampler and estimation of coarse fragments > 20 mm

The bulk density of the fine earth ( $BD_{fe}$ ) is calculated using equation SA04.02.

The FES is calculated as follows:

$$FES = BD_{fe} \times d \times 10 \times \left(1 - \frac{V_{cf>20}}{100} - \frac{M_{cf(2-20)}}{\rho_{cf} \times V_s}\right) \quad (\text{equation SA04.04})$$

where:

FES = Fine earth stock (t/ha)

$BD_{fe}$  = Bulk density of fine earth ( $\text{kg}/\text{m}^3$ )

d = Thickness of the sampled layer (m)

$M_{cf(2-20)}$  = Dry Mass of coarse fragment between 2 and 20 mm taken with core sampler (respectively core of root auger) (kg)

$V_{cf>20}$  = Percentage volume of coarse fragment of the fraction > 20 mm estimated at the profile (%)

$\rho_{cf}$  = Density of the coarse fragments (approximated by 2650 kg/ $\text{m}^3$ )

$V_s$  = Volume of core sampler ( $\text{m}^3$ )

Notes: see Case 2

**Case 3.2.** Combination of representative volume sampling with a core sampler, disturbed sample and estimation of coarse fragments more than 60 mm at the profile

The bulk density of the fine earth ( $BD_{fe}$ ) is calculated using equation SA04.02.

The fine earth stock (FES) is calculated as follows:

$$FES = BD_{fe} \times d \times 10 \times \left( 1 - \frac{V_{cf>60}}{100} - \frac{M_{ds(2-60)}}{BD_{cf}} \times \frac{BD_{fe}}{M_{ds} - M_{ds(2-60)} + BD_{fe} \times \frac{M_{ds(2-60)}}{\rho_{cf}}} \right)$$

(equation SA04.05)

where:

FES	=	Fine earth stock (t/ha)
$BD_{fe}$	=	Bulk density of fine earth (kg/m <sup>3</sup> )
d	=	Thickness of the sampled layer (m)
$M_{ds(2-60)}$	=	Dry Mass of coarse fragment between 2 and 60 mm of the disturbed sample (kg)
$V_{cf>60}$	=	Percentage volume of coarse fragment > 60 mm estimated at the profile (%)
$\rho_{cf}$	=	Bulk density of the coarse fragments (approximated by 2650 kg/m <sup>3</sup> )
$M_{ds}$	=	Total dry mass of the disturbed sample (kg)

Notes: see Case 2

**Case 4:** Representative volume sampling not possible, Sampling with mini-core samplers

From the mass of the sample < 6 mm and the mass of the coarse fragment fraction 2 mm – 6 mm, factor f, which is approximately the coarse fragment portion in the core sampler cap, is calculated as follows:

$$f = \frac{M_{ds(2-6)}}{M_{ds(<6)}} \quad (\text{equation SA04.06})$$

where:

$M_{ds(2-6)}$	=	Mass of coarse fragment of the fraction 2 - 6 mm of the disturbed sample (kg)
$M_{ds(<6)}$	=	Mass of the sample < 6 mm in the aliquot of the disturbed sample (kg)

For the coarse fragment content > 60 mm an estimation from the profile must be available.

The bulk density of the fine earth ( $BD_{fe}$ ) is calculated using the following formula:

$$BD_{fe} = \frac{M_{TOT} MINI \times (1 - f)}{V_{TOT} MINI - \frac{M_{TOT} MINI \times f}{\rho_{cf}}} \quad (\text{equation SA04.07})$$

where:

$M_{TOT} MINI$	=	Mass of mini-core sampler (kg)
$V_{TOT} MINI$	=	Volume of mini-core sampler (m <sup>3</sup> )
$\rho_{cf}$	=	Density of the coarse fragments (kg/m <sup>3</sup> ) (approximated by 2650 kg/m <sup>3</sup> )

The fine earth stock (FES) is calculated using equation SA04.05.

## VII. Report

The dry bulk density (BD) is recorded in kg/m<sup>3</sup> with no decimal places.

In the case of stony or gravelly soils the bulk density of the fine earth fraction (< 2 mm) should be reported together with the coarse fragment content (vol %) (See also SA05).

Furthermore, the bulk density of the coarse fragments should be known, but this may be approximated as 2650 kg.m<sup>-3</sup>. In the case that pedotransfer functions are used (Level I), the calculation procedure should be reported as well.

*Note that the "excavation method" described in ISO11272, asks for the total dry bulk density of the soil, while in this programme the bulk density of the fine earth should be reported.*

## VIII. Reference

ISO 11272. 1993. Soil Quality – Determination of dry bulk density. International Organization for Standardization. Geneva, Switzerland. 10 p. (available at [www.iso.ch](http://www.iso.ch))

DIN ISO 11272, Normenausschuß Wasserwesen (NAW) in the Dt. Inst. für Normung e.V. [Eds.] (2001): Bodenbeschaffenheit - Bestimmung der Trockenrohdichte (Soil composition, Determination of bulk density)

W. Riek, B. Wolff (2006): Evaluierung von Verfahren zur Erfassung des Grobbodenanteils von Waldböden – Erarbeitung von Empfehlungen für die Anwendung dieser Verfahren im Rahmen der Bodenzustandserhebung im Wald (BZE II). Eberswalde (Evaluation of methods to determine the coarse fragment portion of forest soils – Drawing up recommendations for the use of these methods in forest soil surveys)

## **Soil Analysis Method 5 (SA05): Determination of Coarse Fragments**

Coarse Fragments	
Method sheet	SA05
Reference methods	ISO 11464, ISO 11277
Method suitable for	Mineral horizons

## I. Relevance in ICP Forests

Priority	Level I	Level II
Organic Layer	-	-
Mineral layer		
0 – 10 cm	Mandatory <sup>1,2</sup>	Mandatory
10 – 20 cm	Mandatory <sup>1,2</sup>	Mandatory <sup>1,2</sup>
20 – 40 cm	Mandatory <sup>1,2</sup>	Mandatory <sup>1,2</sup>
40 – 80 cm	Optional <sup>1</sup>	Optional <sup>1</sup>

<sup>1</sup> may be obtained by estimation

<sup>2</sup> in case of re-assessment (if the parameter was already measured according to the reference method in a previous survey), the measurement is optional

## II. Principle

The abundance of coarse fragments can be measured in the laboratory, but is usually estimated during routine soil profile description (see Annex 2). When the estimation is based on such a visual observation, one should take into account the volume of the macropores (packing pores between the stones) which is often underestimated.

The most straightforward way to determine the volumes in the field of stones and boulders is by digging pits. This method, however, encounters practical problems such as hard manual work and destructive sampling. The 'Finnish method' or 'rod penetration method' is described here as an example of a non-destructive method. This method estimates the proportion (*volume %*) of coarse gravel (2 – 6 cm), stones (6 – 20 cm) and boulders (> 20 cm) in the 0 – 30 cm mineral layer by pushing a graduated metal rod down through the organic layer and as far as possible into the mineral soil.

Coarse fragments are separated from the fine earth fraction during the preparation of soil samples (SA01). The content of coarse fragments, *cf.* (*mass %*), is determined by weighing the residue left on a 2 mm sieve after washing and drying in the laboratory.

## III. Apparatus

**Field estimation:** The 'Finnish method' or 'rod penetration method'

graduated metal rod (diameter 10 mm, length 80 – 100 cm)

**Laboratory measurement**

No apparatus, using data obtained in preparation of soil sample (SA01).

## IV. Reagents

**Field estimation: The 'Finnish method' or 'rod penetration method'**

No reagents.

**Laboratory measurement**

No reagents, using data obtained in preparation of soil sample (SA01).

## V. Procedure

**Field estimation: The 'Finnish method' or 'rod penetration method'**

The volume of stones is estimated in the 0-30 cm mineral soil layer. A steel rod ( $d = 10$  mm, length = 80...100 cm, with a tip of hard metal, gradation lines at 10 cm intervals, see Fig. 1) is pushed down (through the organic layer) into the mineral soil with sufficient force that the rod will stop if it comes into contact with a stone of 2 cm or larger (moderate push). The measuring rod is pushed down into the mineral soil at e.g. 20 or 30 systematically located (using a tape measure or even paces) points. The depth of penetration is measured with respect to the surface of the ground. If there is an organic layer present, then its thickness has to be measured using the rod or by taking a sample of the organic layer and measuring its thickness, and then subtracted from the penetration depth. In Finland, penetration is measured and organic layer samples are taken at the same time. The average penetration value and stoniness of the 0-30 cm mineral soil layer is calculated as follows (only 5 points in this example):

Penetration depth (cm)	Organic layer thickness (cm)	Penetration depth – organic layer thickness (cm)	Penetration in the ≤30 layer (cm)
12	2	10	10
40	4	36	30
4	4	0	0
35	3	32	30
22	5	17	17
			Average = 17.4

The great advantage of the rod method is that a large number of measurements can be made easily and quickly over the whole plot. The inaccuracy and other drawbacks of the method outweigh the lack of representability involved in measuring (estimating) stoniness in a very restricted number of soil pits.



FIGURE 2: TIP OF THE PENETRATION ROD

FIGURE 1: PENETRATION ROD

### **Laboratory measurement**

No procedure, using data obtained in preparation of soil sample (SA01).

## **VI. Calculation**

### **Field estimation**

0 – 30 cm layer

$$\text{Volume of stones (\%)} = 83 - 2.75 * \text{average penetration (cm)} \quad [\text{Equation SA05.01}]$$

The volume of stones in the example =  $83 - 2.75 * 17.4 = 35\%$  in the 0-30 cm layer. According to equation SA05.01, the volume of stones is 0.5 % when the average penetration into the mineral soil is 30 cm, and volume of stones is 83 % when the average penetration is 0 cm.

It is possible to estimate the stoniness of thinner layers if the empirical relationship between penetration depth and volumetric stone percentage remains the same. The relevant equations are as follows:

0-10 cm layer

$$\text{Volume of stones (\%)} = 83 - 8.25 * \text{average penetration (cm) for the layer}$$

0-20 cm layer

$$\text{Volume of stones (\%)} = 83 - 4.125 * \text{average penetration (cm) for the layer.}$$

The constant maximum depth of each penetration should be set so that it reaches the target mineral soil depth, i.e. 30, 20 or 10 cm, through the thickest possible organic layer. On upland soils an extra 10 cm is commonly added to the target depth, i.e. there is a target depth of 40 cm if the studied layer is 0-30 cm, or to 30 cm if the layer is 0-20 cm.

**Note:** Equation SA05.01 is based on a very specific material [Finnish till (morainic) soils] but has not been tested on other soils, and in some respects it is somewhat illogical (see Eriksson and Holmgren, 1996). It is therefore of utmost importance that the equation is calibrated locally before it can be applied on other soil types..

### Laboratory measurement

The content of coarse fragments,  $cf$  (mass%), is determined by weighing the residue left on a 2 mm sieve after washing and drying according to:

$$cf(\text{mass}\%) = \frac{\text{mass\_of\_soil\_fraction} > 2\text{mm}}{\text{mass\_of\_the\_total\_oven\_dry\_soil}} \times 100$$

In order to convert the content by mass to an expression by volume, the bulk density of both the coarse fragments and the fine earth should be determined.

$$cf(\text{vol}\%) = \frac{BD_s}{BD_{cf}} * cf(\text{mass}\%)$$

where:

$BD_s$	=	Bulk density of the total soil ( $\text{kg/m}^3$ )
$BD_{cf}$	=	Bulk density of the coarse fragments (approximated by $2650 \text{ kg/m}^3$ )
$cf(\text{vol}\%)$	=	Volumetric percentage of coarse fragments in the soil (%)
$cf(\text{mass}\%)$	=	Mass percentage of coarse fragments in the soil (%)

## VII. Report

The amount of coarse fragments (stones and gravel with a diameter  $> 2 \text{ mm}$ ) has to be reported for the individual mineral layers in volume % without decimals.

**Note:** The Rod penetration method only allows reporting for the 0 – 10 cm, 0 – 20 cm or 0- 30 cm layer and for the coarse fragments  $> 2 \text{ cm}$

## VIII. References

- Eriksson, C.P., Holmgren, P. 1996. Estimating stone and boulder content in forest soils – evaluating the potential of surface penetration methods. *Catena* 28: 121 – 134.
- ISO 11464. 1994. Soil Quality – Pretreatment of samples for physico-chemical analysis. International Organization for Standardization. Geneva, Switzerland. 9 p. (available at [www.iso.ch](http://www.iso.ch))
- ISO 11277. 1998. Soil Quality – Determination of particle size distribution in mineral soil material – Method by sieving and sedimentation. International Organization for Standardization. Geneva, Switzerland. 30 p. (available at [www.iso.ch](http://www.iso.ch))

Mikkelsen, J. Cools, N., Langohr, R. 2006 Guidelines for Forest Soil Profile Description, adapted for optimal field observations within the framework of the EU Forest Focus Demonstration Project. BIOSOIL. Partly based on the 4<sup>th</sup> edition of the Guidelines for Soil Profile Description and Classification (FAO, 2006).

Tamminen, P. 1991. Kangasmaan ravinntunnusten ilmaiseminen ja viljavuuden alueellinen vaihtelu Etelä-Suomessa. Summary: Expression of soil nutrient status and regional variation in soil fertility of forested sites in Southern Finland. *Folia Forestalia* 777: 1-40.

Viro, P., 1947. Metsämaan raekoostumus ja viljavuus varsinkin maan kivisyyttä silmällä pitäen. Summary: The mechanical composition and fertility of forest soil taking into consideration especially the stoniness of the soil. *Communicationes Instituti Forestalis Fenniae* 35, 115.

- 1952. Kivisyyden määrittämisestä. Summary: On the determination of stoniness. *Communicationes Instituti Forestalis Fenniae* 40, 23.
- 1958. Suomen metsämaiden kivisyydestä. Summary: Stoniness of forest soil in Finland. *Communicationes Instituti Forestalis Fenniae* 49, 45

## **Soil Analysis Method 6 (SA06): Determination of Soil pH**

<b>pH</b>	
Method sheet	SA06
Reference methods	ISO 10390
Method suitable for	Organic Layer; Mineral Layer

## I. Relevance in ICP Forests

pH(CaCl<sub>2</sub>)

<b>Priority</b>	<b>Level I</b>	<b>Level II</b>
<b>Organic Layer</b>		
OL	-	-
OF+OH, H-layers	Mandatory	Mandatory
<b>Mineral layer</b>		
0 – 10 cm	Mandatory	Mandatory
10 – 20 cm	Mandatory	Mandatory
20 – 40 cm	Optional	Mandatory <sup>2</sup>
40 – 80 cm	Optional	Mandatory <sup>2</sup>

<sup>2</sup> in case of re-assessment (if the parameter was already measured according to the reference method in a previous survey), the measurement is optional

pH(H<sub>2</sub>O)

<b>Priority</b>	<b>Level I</b>	<b>Level II</b>
<b>Organic Layer</b>	Optional	Optional
OL	-	-
OF+OH, H-layers	Optional	Optional
<b>Mineral layer</b>		
0 – 10 cm	Optional	Optional
10 – 20 cm	Optional	Optional
20 – 40 cm	Optional	Optional
40 – 80 cm	Optional	Optional

## II. Principle

The pH of the soil is potentiometrically measured in the supernatant suspension of 1:5 (volume fraction). This liquid is made up of a 0.01 mol/l solution of calcium chloride in water for pH(CaCl<sub>2</sub>) or deionised water for pH(H<sub>2</sub>O).

## III. Apparatus

End-over-end shaking machine  
pH meter with appropriate electrode  
Thermometer (accuracy 1 °C)  
Sample bottle (capacity at least 50 ml) with cap  
Accurate measuring spoon

## IV. Reagents

Water (grade 2)  
Calcium chloride (CaCl<sub>2</sub>), conc. 0.01 mol/l  
make a solution of 1.47 g CaCl<sub>2</sub>.2H<sub>2</sub>O/liter water  
pH buffer solutions

## V. Procedure

### *Preparation of the suspension*

Take a representative sample (at least a volume of 5 ml) of the air-dried soil (fraction < 2 mm) using the accurate measuring spoon. Place the test sample in the sample bottle and add five times its volume of calcium chloride solution (pH-CaCl<sub>2</sub>) or deionised water (pH-H<sub>2</sub>O). Shake or mix the suspension for 60 min +/- 10 min, using the mechanical shaker or mixer, and wait for at least for 1 hour before measuring but not longer than 3 hours. Ingres of air during standing after shaking should be avoided.

### *Calibration of pH meter*

Calibrate the pH-meter as prescribed in the manufacturer' s manual, using the buffer solutions.

### *pH measurement*

Measure the pH in the suspension at 20°C ± 2°C immediately after or whilst being stirred. The stirring should be at such a rate to achieve a reasonable homogeneous suspension of the soil particles, but entrainment of air should be avoided. Read the pH after stabilisation of the value is reached.

## **VI. Calculations**

No calculations.

## **VII. Report**

Note the recorded values to two decimal places.

## **VIII. Reference**

ISO 10390. 2005. Soil Quality – Determination of pH. International Organization for Standardization. Geneva, Switzerland. 5 p. (available at [www.iso.ch](http://www.iso.ch))

**Soil Analysis Method 7 (SA07): Determination of Carbonate Content**

<b>Carbonates</b>	
Method sheet	SA07
Reference methods	ISO 10693
Method suitable for	Organic Layer, Mineral Layer

## I. Relevance in ICP Forests

<b>Priority</b>	<b>Level I</b>	<b>Level II</b>
<b>Organic Layer</b>		
OL	-	-
OF+OH, H-layers	Mandatory <sup>1</sup>	Mandatory <sup>1</sup>
<b>Mineral layer</b>		
0 – 10 cm	Mandatory <sup>1</sup>	Mandatory <sup>1</sup>
10 – 20 cm	Mandatory <sup>1</sup>	Mandatory <sup>1</sup>
20 – 40 cm	Optional	Optional
40 – 80 cm	Optional	Optional

<sup>1</sup> Only mandatory if pH(CaCl<sub>2</sub>) > 5.5 or in calcareous soils

## II. Principle

The soil sample is treated with a strong acid. The volume of the carbon dioxide produced is measured by using a calcimeter (Scheibler unit), and is compared with the volume of carbon dioxide produced by pure calcium carbonate.

## III. Apparatus

Calcimeter (Scheibler unit)

Analytical balance (accuracy 0.0001 g)

Reaction vessels (capacity 150 ml)

Plastic cups (which can pass through the neck of the reaction vessel)

Tong

Watch glass

## IV. Reagents

Distilled water

Hydrochloric acid (HCl), conc. 4 mol/l

Dilute 340 ml of concentrated hydrochloric acid ( $\rho = 1.19 \text{ g/ml}$ ) to 1000 ml with water.

Calcium carbonate ( $\text{CaCO}_3$ ), pure.

## V. Procedure

### *Preparation*

The mass of the test portion is determined based on the carbonate content. For a preliminary test on carbonate content, add some hydrochloric acid to a portion of the soil on a watch glass. The carbonate content of the sample can be estimated on the basis of the intensity and duration of effervescence (Table SA07-1). Determine from table SA07-1 the mass of test portion (air-dried soil fraction < 2 mm).

**Table SA07-1:** Mass of test portion for determination of carbonate content based on intensity of effervescence

Intensity of effervescence	Carbonate content (g/kg)	Mass of test sample (g)
None or only limited	< 20	10
Clear, but for a short time	20 – 80	5
Strong, for a long time	80 – 160	2.5
Very strong, for a long time	> 160	1 <sup>1</sup>

<sup>1</sup> use sample that is crushed to a particle size of less than 250 µm

### *Measurement*

Transfer the sample into the reaction vessels and add 20 ml of water. Fill the plastic cup with 7 ml of hydrochloric acid and place this, using tongs in the reaction vessel containing the test portion. Take care that there is no contact between the hydrochloric acid and the soil before the reaction vessel is connected to the calcimeter (Scheibler unit). Warm the reaction vessel by hand.

Connect the reaction vessel to the calcimeter. Carefully add the hydrochloric acid from the cup to the soil by tilting the reaction vessel at an angle. The gas produced will lower the water level in the tube on the right and at the same time will raise the water level in the tube on the left. Shake for 5 min and note the volume when it no longer varies. If it still varies, continue shaking until the volume is stable, but not longer than 1 h. At the end of the shaking period, bring the water level in both tubes to the same height and measure the volume of gas in the calibrated tube with an accuracy of 0.1 ml.

### *Calibration*

Determinations of samples, blanks and the calcium carbonate used as standard material, shall be performed simultaneously in a room where temperature and pressure do not vary too much during the measurement.

Weigh the standards of 0.200 g and 0.400 g of calcium carbonate, transfer these amounts into the reaction vessels and add 20 ml of water. For the blank determinations, use reaction vessels containing 20 ml of water.

## VI. Calculations

$$w(\text{CaCO}_3) = 1000 \times \frac{m_2(V_1 - V_3)}{m_1(V_2 - V_3)}$$

$w(\text{CaCO}_3)$  = carbonate content of sample (g/kg) on basis of air dried soil

$m_1$  = mass (g) of test sample

$m_2$  = mean mass (g) of standards

$V_1$  = volume (ml) of CO<sub>2</sub> produced by test sample

$V_2$  = mean volume (ml) of CO<sub>2</sub> produced by standards

$V_3$  = volume change (ml) in blank determinations (can be negative)

## VII. Report

The results of the carbonate (g/kg) must be reported without decimals on the basis of oven-dried soil.

## VIII. Reference

ISO 10693. 1994. Soil Quality – Determination of carbonate content - Volumetric method.

International Organization for Standardization. Geneva, Switzerland. 7 p. (available at [www.iso.ch](http://www.iso.ch))

**Soil Analysis Method 8 (SA08): Determination of Organic Carbon Content**

<b>Organic Carbon</b>	
Method sheet	SA08
Reference methods	ISO 10694
Method suitable for	Organic Layer, Mineral Layer

## I. Relevance in ICP Forests

<b>Priority</b>	<b>Level I</b>	<b>Level II</b>
<b>Organic Layer</b>		
OL	-	-
OF+OH, H-layers	Mandatory	Mandatory
<b>Mineral layer</b>		
0 – 10 cm	Mandatory	Mandatory
10 – 20 cm	Mandatory	Mandatory
20 – 40 cm	Mandatory	Mandatory
40 – 80 cm	Optional	Optional

## II. Principle

The carbon present in the soil is oxidised to carbon dioxide ( $\text{CO}_2$ ) by heating the soil to at least 900 °C in a flow of oxygen-containing gas that is free from carbon dioxide. The amount of carbon dioxide released is then measured by titrimetry, gravimetry, conductometry, gas chromatography or using an infrared detection method, depending on the apparatus used.

When the soil is heated to a temperature of at least 900 °C, any carbonates present are completely decomposed.

Total organic carbon can be determined directly or indirectly. Direct determination consists of previous removal of any carbonates present by treating the soil with hydrochloric acid. Indirect determination consists of a correction of the total carbon content for the carbonates present.

## III. Apparatus

Glassware

Analytical balance (accuracy 0.0001 or 0.00001 g)

Apparatus for determination of total carbon content (temperature at least 900 °C)

Crucibles proper for the apparatus

## IV. Reagents

Combustion gas - chemicals and catalysts proper to the apparatus

Calibration substances

Hydrochloric acid (HCl), conc. 4 mol/l

## V. Procedure

### *Laboratory sample*

Use sample of air-dried soil (fraction < 2 mm) of known moisture and carbonate content.

### *Calibration of the apparatus*

Calibrate the apparatus as described in the relevant manual using the calibration substances.

### *Direct determination of organic carbon content*

Add an excess of hydrochloric acid (4 mol/l) to the crucible containing a weighed quantity of air-dried soil and mix. Wait 4 h and dry the crucible for 16 h at a temperature of 60 °C to 70 °C. The amount of test portion taken for analysis depends on the expected carbon content and on the apparatus used. Weigh out  $m_1$  g of the air-dried sample in a crucible. Carry out the analyses in accordance with the manufacturer's manual for the apparatus.

### *Indirect determination of organic carbon content*

The procedure is identical to the direct determination of organic carbon content, without adding hydrochloric acid. The measured total carbon content is calculated according to the amount of test portion taken for analysis which depends on the expected total carbon content and on the apparatus used. Weigh out  $m_1$  g of the air-dried sample in a crucible. Carry out the analyses in accordance with the manufacturer's manual for the apparatus.

## VI. Calculation

### *Direct determination of organic carbon content*

The organic carbon content (on basis of air-dried soil) is obtained by :

$$w_{C,o} = 1000 \times \frac{m_2}{m_1} \times 0.2727$$

where

$w_{C,o}$	=	Organic carbon content (g/kg) on basis of air-dried soil
$m_1$	=	Mass (g) of test portion
$m_2$	=	Mass (g) of released CO <sub>2</sub>
0.2727	=	Conversion factor for CO <sub>2</sub> to C

### *Indirect determination of organic carbon content*

The total carbon content (on basis of air-dried soil) is obtained by :

$$w_{C,t} = 1000 \times \frac{m_2}{m_1} \times 0.2727$$

where

$w_{C,t}$	=	Total carbon content (g/kg) on basis of air-dried soil
$m_1$	=	Mass (g) of test portion
$m_2$	=	Mass (g) of released $\text{CO}_2$
0.2727	=	Conversion factor for $\text{CO}_2$ to C

Calculate the organic carbon content of the sample using a correction for carbonates. The organic carbon content (on basis of air dried soil) is calculated by:

$$w_{C,o} = w_{C,t} - (0.12 \times w_{\text{CaCO}_3})$$

where

$w_{C,o}$	=	Organic carbon content (g/kg) on basis of air-dried soil
$w_{C,t}$	=	Total carbon content (g/kg) on basis of air-dried soil
0.12	=	Conversion factor
$w_{\text{CaCO}_3}$	=	Carbonate content (g/kg) on basis of air-dried soil

## VII. Report

Report organic carbon content (in g/kg) with 1 decimal place on the basis of oven-dried soil.

## VII. Reference

ISO 10694. 1995. Soil Quality – Determination of organic and total carbon after dry combustion (elementary analysis). International Organization for Standardization. Geneva, Switzerland.  
7 p. (available at [www.iso.ch](http://www.iso.ch))

**Soil Analysis Method 9 (SA09): Determination of Total Nitrogen Content**

<b>Total Nitrogen</b>	
Method sheet	SA09A
Reference methods	ISO 13878
Method suitable for	Organic Layer, Mineral Layer

## I. Relevance in ICP Forests

Priority	Level I	Level II
<b>Organic Layer</b>		
OL	-	-
OF+OH, H-layers	Mandatory	Mandatory
<b>Mineral layer</b>		
0 – 10 cm	Mandatory	Mandatory
10 – 20 cm	Mandatory	Mandatory
20 – 40 cm	Optional	Optional
40 – 80 cm	Optional	Optional

## II. Principle

The nitrogen content of a soil is determined by heating to a temperature of at least 900 °C in the presence of oxygen gas. Mineral and organic nitrogen compounds are oxidised and/or volatilised. The combustion products are oxides of nitrogen ( $\text{NO}_x$ ) and molecular nitrogen ( $\text{N}_2$ ). After transforming all nitrogen forms into  $\text{N}_2$ , the content of total nitrogen is measured using thermal conductivity.

## III. Apparatus

Laboratory glassware

Analytical balance (accuracy 0.0001 or 0.00001 mg)

Apparatus for determination of total nitrogen content (temperature at least 900 °C)

Crucibles proper for the apparatus

## IV. Reagents

Combustion gas - chemicals and catalysts proper to the apparatus

Calibration substances

## V. Procedure

### *Laboratory sample*

Use fraction of air-dried soil (fraction < 2 mm) of known moisture content. If a soil mass of less than 2 g is required for nitrogen analysis, mill a representative subsample further, to pass a sieve of an aperture specified in the manufacturer's manual to ensure sufficient test reproducibility.

### *Calibration of the apparatus*

Calibrate the apparatus as described in the relevant manual using the calibration substances.

### *Determination of total nitrogen content*

The amount of test sample for analysis depends on the expected total nitrogen content and on the apparatus used. Weigh out  $m_1$  g of the air-dried sample or subsample into a crucible. Carry out the analyses in accordance with the manufacturer's manual for the apparatus.

Normally the primary results are given as milligrams nitrogen ( $X_1$ ) or a mass fraction of nitrogen ( $X_2$ ), expressed as a percentage, referred to the mass of air-dry soil used ( $m_1$ ).

## VI. Calculation

Calculate the total content of nitrogen ( $w_N$ ), in milligrams per gram, on the basis of the air-dried soil according to the following equations:

- For primary results given in milligrams of nitrogen:

$$w_N = \frac{X_1}{m_1}$$

- For primary results, given as percent mass fraction of nitrogen:

$$w_N = 10 \cdot X_2$$

where

$w_N$ : total nitrogen content (g/kg) on basis of air-dried soil

$m_1$ : mass (g) of test portion

$X_1$ : primary result as milligrams N

$X_2$ : primary result as percentage N

## VII. Report

Report total nitrogen (in g/kg) with 1 decimal place on the basis of oven-dried soil.

## VIII. Reference

ISO 13878. 1998. Soil Quality – Determination of total nitrogen content by dry combustion ("elemental analysis"). International Organization for Standardization. Geneva, Switzerland.  
5 p. (available at [www.iso.ch](http://www.iso.ch))

Total Nitrogen Modified Kjeldahl method	
Method sheet	SA09B
Reference methods	ISO 11261
Method suitable for	Organic Layer, Mineral Layer

## I. Relevance in ICP Forests

Priority	Level I	Level II
<b>Organic Layer</b>		
OL	-	-
OF+OH, H-layers	Mandatory	Mandatory
<b>Mineral layer</b>		
0 – 10 cm	Mandatory	Mandatory
10 – 20 cm	Mandatory	Mandatory
20 – 40 cm	Optional	Optional
40 – 80 cm	Optional	Optional

## II. Principle

The modified Kjeldahl method determines the total nitrogen content (including ammonium-N, nitrate-N, nitrite-N and organic N) of a soil. The method is based on a Kjeldahl digestion, but instead of selenium (Kjeldahl method) titanium dioxide is used as the catalyst.

## III. Apparatus

Digestion flasks or tubes (50 ml)

Digestion stand

Distillation apparatus

Burette (intervals of 0.01ml or smaller)

## IV. Reagents

Salicylic acid / Sulfuric acid: Dissolve 25g of salicylic acid in 1 litre of concentrated sulfuric acid ( $\rho = 1.84 \text{ g/cm}^3$ )

Potassium sulfate catalyst mixture: Grind and thoroughly mix the following substances;

$$\left\{ \begin{array}{l} 200 \text{ g of potassium sulfate} \\ 6 \text{ g of copper (II) sulfate pentahydrate} \\ 6 \text{ g of titanium dioxide with the crystal structure of anatase} \end{array} \right.$$

Sodium thiosulfate pentahydrate: Crush the crystals of Sodium thiosulfate pentahydrate until they form a powder that passes through a sieve with an aperture of 0.25mm

Sodium hydroxide:  $c(\text{NaOH}) = 10 \text{ mol/l}$

Boric acid solution:  $\rho(\text{H}_3\text{BO}_3) = 20 \text{ g/l}$

Mixed indicator: Dissolve 0.1 g of bromocresol green and 0.02 g of methyl red in 100 ml of ethanol

Sulfuric acid:  $c(\text{H}^+) = 0.01 \text{ mol/l}$

## V. Procedure

Place a test portion from 0.2g (expected N-content 0.5%) to 1g (expected N-content of 0.1%) of the air-dried soil sample in the digestion flask.

Add 4 ml of salicylic/sulfuric acid and swirl the flask until the acid is thoroughly mixed with the soil. Let the mixture stand for at least several hours (or overnight).

Add 0.5 g of sodium thiosulfate trough a dry funnel with a long stem that reaches down into the bulb of the digestion flask. Heat the mixture cautiously on the digestion stand until frothing has ceased.

Cool the flask and add 1.1g of the catalyst mixture, heat until the digestion mixture becomes clear.

Boil the mixture gently for up to 5 h. (in most cases a boiling period of 2h. is sufficient) so that the sulfuric acid condenses about 1/3 of the way up to the neck of the flask. Make sure that the temperature of the solution does not exceed 400°C.

Allow the flask to cool down after the digestion and add about 20ml of water slowly while shaking. Then swirl the flask to bring any insoluble material into suspension and transfer then the contents to the distillation apparatus. Rinse tree time with water to complete the transfer.

Add 5 ml of boric acid to a 100 ml conical flask. Place the flask under the condenser of the distillation apparatus, make sure that the end of the condenser dips into the solution.

Add 20 ml of sodium hydroxide to the funnel of the apparatus and run the alkali slowly into the distillation chamber.

Distil about 40 ml of the condensate and rinse the end of the condenser.

Add a few drops of indicator to the distillate and titrate with sulfuric acid to a violet endpoint or use a potentiometric titration with endpoint pH=5.

**Notes:**

Carry out a blank test in which the same procedure is performed without soil.  
 A potentiometric titration is also possible (endpoint of titration should be pH = 5).  
 If steam distillation is used, a distillation rate up to about 25ml/min is applicable. Stop the distillation when about 100ml of distillate have been collected.

## **VI. Calculation**

The total nitrogen content is calculated by use of the following formula:

$$w_N = \frac{(V_1 - V_0) \times c(H^+) \times M_N}{m} \times \frac{100 + w_{H_2O}}{100}$$

Where

$w_N$	=	The total nitrogen content (mg/g = g/kg)
$V_1$	=	Volume of the sulfuric acid used in the titration of the sample (ml)
$V_0$	=	Volume of the sulfuric acid used in the titration of the blank sample (ml)
$c(H^+)$	=	Concentration of H <sup>+</sup> in the sulfuric acid (moles/litre)
$M_N$	=	The molar mass of nitrogen (= 14 g/mol)
$m$	=	Mass of the air-dried soil sample (g)
$w_{H_2O}$	=	Water content of the soil sample, based on oven-dried soil (% by mass)

## **VII. Report**

Report total nitrogen in g/kg with 1 decimal place on the basis of oven-dried oil.

## **VIII. Reference**

ISO 11261. 1995. Soil Quality – Determination of total nitrogen – Modified Kjeldahl method. International Organization for Standardization. Geneva, Switzerland. 4p. (available at [www.iso.ch](http://www.iso.ch))

**Soil Analysis Method 10 (SA10): Determination of Exchangeable Cations  
(Al, Ca, Fe, K, Mg, Mn, Na), Free H<sup>+</sup> and Exchangeable Acidity**

Exchangeable acidity and exchangeable cations	
Method sheet	SA10
Reference Methods	ISO 11260 & ISO 14254
Method suitable for	Organic Layer, Mineral Layer

## I- Relevance in ICP Forests

*Basic exchangeable cations (Ca, Mg, K, Na)*

Priority	Level I	Level II
<b>Organic Layer</b>		
OL	-	-
OF+OH, H-layers	Mandatory <sup>1</sup>	Mandatory <sup>1</sup>
<b>Mineral layer</b>		
0 - 10 cm	Mandatory	Mandatory
10 - 20 cm	Mandatory	Mandatory
20 - 40 cm	Optional	Mandatory <sup>2</sup>
40 - 80 cm	Optional	Mandatory <sup>2</sup>

<sup>1</sup> in calcareous soil, this parameter is optional

<sup>2</sup> in case of re-assessment (if the parameter was already measured according to the reference method in a previous survey), the measurement is optional

Acid exchangeable cations (Al, Fe, Mn), free H<sup>+</sup> acidity and Exchangeable acidity

Priority	Level I	Level II
<b>Organic Layer</b>		
OL	-	-
OF+OH, H-layers	Mandatory <sup>1</sup>	Mandatory <sup>1</sup>
<b>Mineral layer</b>		
0 - 10 cm	Mandatory <sup>1</sup>	Mandatory <sup>1</sup>
10 - 20 cm	Mandatory <sup>1</sup>	Mandatory <sup>1</sup>
20 - 40 cm	Optional	Mandatory <sup>1,2</sup>
40 - 60 cm	Optional	Mandatory <sup>1,2</sup>

<sup>1</sup> in calcareous soil, this parameter is optional

<sup>2</sup> in case of re-assessment (if the parameter was already measured according to the reference method in a previous survey), the measurement is optional

## II. Principle

The soil is first saturated with respect to barium by treating the soil one single time with a 0,1 mol/l barium chloride solution.

Concentrations of the exchangeable basic cations sodium, potassium, calcium and magnesium and the exchangeable acid cations iron, manganese, aluminium are determined in the 0.1 mol/l barium chloride extract of the soil using spectrometry.

To determine exchangeable acidity, the 0.1 mol/l extract is titrated with a 0.05 mol/l NaOH solution up to pH = 7.8. Determination of the free H<sup>+</sup> acidity is realised using a method in which sodium fluoride is added to the soil extract before the titration (Aluminium ions are complexed and only the free H<sup>+</sup> acidity is detected during the titration process).

**Note:** the reference method deviates from ISO 11260 & ISO 14254 in the sense that one single barium chloride extraction must be used instead of three extractions

Alternatively the free H<sup>+</sup> acidity can be determined by the "German calculation method" based on the pH of the barium chloride solution before and after extraction (König *et al.* 2005). The exchangeable acidity is subsequently calculated based on the sum of the acid cations and the free H<sup>+</sup>.

### III. Apparatus

Centrifuge + centrifuge tubes

Mechanical shaker

Laboratory glassware

Magnetic stirrer

Funnel (diam. approx. 110 mm)

Filter paper (diam. 150 mm)

PE-bottles

pH-meter

Burette

Atomic Absorption Spectrometer (AAS) / Flame Emission Spectrometer (FES) / Inductively Coupled Plasma Spectrometer (ICP)

### IV. Reagents

Barium chloride (BaCl<sub>2</sub>) solution, conc. 0.1 mol/l

Sodium hydroxide (NaOH) solution, conc. 0.05 mol/l

Sodium fluoride (NaF) solution, conc. 1 mol/l

pH buffer solutions

Calibration substances

### V. Procedure

#### *Laboratory sample*

Use 2.5 g air-dried soil (particle size < 2 mm) of known moisture content.

*Leaching procedure*

Place the laboratory sample in a 50 ml centrifuge tube. Add 30 ml barium chloride solution and shake for 2 hours. Centrifuge at 3000 g for 10 min. Transfer the supernatant liquid through a filter into a PE-bottle. Retain the extract for analysis (Volume V).

If the filtered extract solution is not enough for the measurement of all cations and pH the extract solution can be diluted (for example 1:5) with barium chloride solution. This has to be considered when calculating the concentrations in the extract. Alternatively it is allowed to use higher volumes of barium chloride solution, but the ratio soil to solution must always be the same (e.g. 5.0 g soil and 60 ml barium chloride solution)!

*Note: According to ISO 11260 & ISO 14254 three BaCl<sub>2</sub> extractions should be done and each time shaken for 1 hour in contrast to this analytical method (SA10).*

*Determination of exchangeable cations (Ca, Mg, K, Na, Al, Fe, Mn)*

Measure the exchangeable cations in the extract using one of the spectrometric determination methods.

*Determination of free H<sup>+</sup>*

Pipette 25 ml of the extract (Volume V<sub>s</sub>). Add 1.25 ml of the sodium fluoride (1 mol/l) solution. Titrate with the sodium hydroxide (0.05 mol/l) solution to a pH value of 7.8. Titrate a blank in the same way.

*Note: If 25 ml is not sufficient for the titration, new BaCl<sub>2</sub> extract, in accordance to ISO 11260, should be obtained and used.*

*Determination of exchangeable acidity*

Pipette 25 ml of the extract into a container of sufficient capacity to also receive the electrodes of the pH-meter. Insert the electrodes and titrate with the sodium hydroxide (0.05 mol/l) solution until a pH value of 7.8 is reached and remains stable for 30 s. Repeat the procedure for a blank 0.1 mol/l BaCl<sub>2</sub> solution extract.

*Note: If 25 ml is not sufficient for the titration, new BaCl<sub>2</sub> extract, in accordance to ISO 11260, should be obtained and used.*

## VI. Calculation

*Determination of exchangeable cations (Ca, Mg, K, Na, Al, Fe, Mn)*

Calculation according to apparatus taking into account following equivalent mass in g/mol:

$$\begin{array}{llll} \text{Na}^+ = 22,99 & \text{Ca}^{2+} = 20,04 & \text{Fe}^{3+} = 18,62 & \text{Al}^{3+} = 8,99 \\ \text{K}^+ = 39,10 & \text{Mg}^{2+} = 12,16 & \text{Mn}^{2+} = 27,47 & \text{H}^+ = 1,01 \end{array}$$

Calculation of the ion equivalents per g soil:

$$\text{IE} = \frac{\text{c} * \text{V}}{\text{m} * \text{EQ} * 10}$$

where

IE ion equivalent in cmol/kg

c element concentration in the extract in mg/l

V volume of the added BaCl<sub>2</sub>-solution in ml (30 ml)

m mass of the soil sample in g (2,5 g)  
 EQ equivalent mass of the element in g/mol

#### *Determination of exchangeable acidity*

The total exchangeable acidity on basis of air-dried soil is given by:

$$E_A = \frac{(V_a - V_b) \cdot C_{NaOH} \cdot 100 \cdot V}{V_s \cdot m}$$

where

$E_A$  total exchangeable acidity (cmol/kg) of the soil on basis of air-dried soil  
 $V_a$  volume NaOH (ml) used for the test sample  
 $V_b$  volume NaOH (ml) used for the blank  
 $C_{NaOH}$  concentration of NaOH (mol/l)  
 $V_s$  volume (ml) pipetted for analysis  
 $m$  mass (g) of the laboratory sample  
 $V$  final volume (ml) of the extract

#### *Determination of free H<sup>+</sup>*

For free H<sup>+</sup> acidity use the same equation as for exchangeable acidity but use the volumes  $V_a$  and  $V_b$  for the volume NaOH used in the titration for free acidity.

#### *Alternative method for the determination of free H<sup>+</sup> ("German" calculation method)*

Calculation of the Proton equivalent per gram soil:

$$H^+ (\text{cmol/kg}) = 10^{-1} * \frac{(10^{-pH_p} - 10^{-pH_0}) * V * 1000}{m * 0,88} - \frac{c(Al) * V}{m * M(Al) * \left(1 + \frac{10^{-pH_p}}{10^{-5,85}}\right)}$$

Or

$$H^+ (\text{cmol/kg}) = 10^{-1} * \frac{(10^{-pH_p} - 10^{-pH_0}) * V * 1000}{m * 0,88} - \frac{c(Al) * V}{m * M(Al) * F}$$

Where

F = the Ulrich/Prenzel factor. Values of the F factor for different pH values can be read from Table SA10-1.

$H^+$  = Free H<sup>+</sup> in cmol/kg  
 $10^{-1}$  = Conversion factor between units ( $\mu\text{mol/g}$  to cmol/kg)  
 $pH_p$  = pH-value of the BaCl<sub>2</sub> extract after the leaching procedure  
 $pH_0$  = pH-value of the pure BaCl<sub>2</sub>-extract  
 $V$  = Final Volume of the extract in ml (30 ml)  
 $m$  = Mass of the laboratory sample in g (2.5 g)  
 $c(Al)$  = Concentration of the Aluminium in the BaCl<sub>2</sub> extract in mg/l  
 $M(Al)$  = Molar mass of Aluminium in g/mol (26,98 g/mol)  
 $F$  = Ulrich/Prenzel factor (cf. Table SA10-1)

*Note: As alternative method, the exchangeable acidity can be calculated as the sum of the exchangeable acid cations (Al, Fe, Mn, free H<sup>+</sup>).*

**Table SA10-1:** The Ulrich/Prenzel factor (F) for a range of pH<sub>p</sub> values (König and Fortman, 1996)

pH	F	pH	F	pH	F	pH	F	pH	F	pH	F
		4,6	18,8	4,1	57,2	3,6	179	3,1	563	2,6	1774
		4,59	19,2	4,09	58,5	3,59	183	3,09	576	2,59	1816
		4,58	19,6	4,08	59,9	3,58	187	3,08	590	2,58	1858
		4,57	20,1	4,07	61,3	3,57	192	3,07	604	2,57	1900
		4,56	20,5	4,06	62,7	3,56	196	3,06	618	2,56	1943
		4,55	21	4,05	64,1	3,55	201	3,05	632	2,55	1993
		4,54	21,4	4,04	65,6	3,54	205	3,04	647	2,54	2035
		4,53	21,9	4,03	67,1	3,53	210	3,03	662	2,53	2084
		4,52	22,4	4,02	68,6	3,52	215	3,02	677	2,52	2134
		4,51	22,9	4,01	70,2	3,51	220	3,01	693	2,51	2183
		4,50	23,4	4	71,8	3,5	225	3	709	2,5	2233
		4,49	23,9	3,99	73,5	3,49	230	2,99	721	2,49	2289
		4,48	24,4	3,98	75,1	3,48	235	2,98	743	2,48	2341
		4,47	25	3,97	76,9	3,47	241	2,97	757	2,47	2401
		4,46	25,5	3,96	78,6	3,46	246	2,96	778	2,46	2451
		4,45	26,1	3,95	80,4	3,45	252	2,95	792	2,45	2511
		4,44	26,7	3,94	82,3	3,44	258	2,94	813	2,44	2571
		4,43	27,3	3,93	84,2	3,43	264	2,93	827	2,43	2631
		4,42	27,9	3,92	86,2	3,42	270	2,92	848	2,42	2691
		4,41	28,5	3,91	88,1	3,41	276	2,91	870	2,41	2751
		4,4	29,2	3,9	90,1	3,4	283	2,9	891	2,4	2821
		4,39	29,8	3,89	92,2	3,39	289	2,89	912	2,39	2881
		4,38	30,5	3,88	94,3	3,38	296	2,88	933	2,38	2961
		4,37	31,2	3,87	96,5	3,37	303	2,87	954	2,37	3021
		4,36	31,9	3,86	98,7	3,36	310	2,86	976	2,36	3091
		4,35	32,6	3,85	101	3,35	317	2,85	997	2,35	3161
		4,34	33,4	3,84	103	3,34	325	2,84	1024	2,34	3241
		4,33	34,1	3,83	106	3,33	332	2,83	1046	2,33	3311
		4,32	34,9	3,82	108	3,32	340	2,82	1067	2,32	3391
		4,31	35,7	3,81	111	3,31	348	2,81	1095	2,31	3471
		4,3	36,5	3,8	113	3,3	356	2,8	1117	2,30	3551
4,8	12,2	4,29	37,3	3,79	116	3,29	364	2,79	1145	2,29	3631
4,79		4,28	38,2	3,78	118	3,28	373	2,78	1173	2,28	3721
4,78		4,27	39	3,77	121	3,27	381	2,77	1202	2,27	3801
4,77	13	4,26	39,9	3,76	124	3,26	390	2,76	1230	2,26	3891
4,76	13,3	4,25	40,8	3,75	127	3,25	399	2,75	1258	2,25	3981
4,75	13,6	4,24	41,7	3,74	130	3,24	408	2,74	1286	2,24	4071
4,74	13,9	4,23	42,7	3,73	133	3,23	418	2,73	1315	2,23	4171
4,73	14,2	4,22	43,9	3,72	136	3,22	430	2,72	1350	2,22	4271
4,72	14,5	4,21	44,7	3,71	139	3,21	438	2,71	1378	2,21	4371

4,71	14,8		4,20	45,1		3,70	142		3,20	448		2,70	1413		2,20	4471
4,7	15,1		4,19	46,7		3,69	146		3,19	458		2,69	1442		2,19	4571
4,69	15,5		4,18	47,3		3,68	149		3,18	469		2,68	1477		2,18	4681
4,68	15,8		4,17	48,9		3,67	152		3,17	480		2,67	1512		2,17	4791
4,67	16,1		4,16	50		3,66	156		3,16	491		2,66	1548		2,16	4901
4,66	16,5		4,15	51,1		3,65	159		3,15	502		2,65	1583		2,15	5001
4,65	16,8		4,14	52,3		3,64	163		3,14	514		2,64	1618		2,14	5131
4,64	17,2		4,13	53,5		3,63	167		3,13	526		2,63	1654		2,13	5251
4,63	17,6		4,12	54,7		3,62	170		3,12	538		2,62	1695		2,12	5371
4,62	18		4,11	56		3,61	175		3,11	551		2,61	1731		2,11	5501
4,61	18,4		4,10	57,2		3,60	179		3,10	563		2,60	1774		2,10	5621

## VII. Report

Report (in cmol<sub>(+)</sub>/kg) total exchangeable acidity, exchangeable cations and free H<sup>+</sup> with 2 decimal places on the basis of oven-dried soil.

## VIII. References

- ISO 11260. 1994. Soil Quality – Determination of effective cation exchange capacity and base saturation level using barium chloride solution. International Organization for Standardization. Geneva, Switzerland. 10 p. (available at [www.iso.ch](http://www.iso.ch))
- ISO 14254. 1994. Soil Quality – Determination of exchangeable acidity in barium chloride extracts. International Organization for Standardization. Geneva, Switzerland. 5 p. (available at [www.iso.ch](http://www.iso.ch))
- König and Fortmann 1996. Probenvorbereitungs-, Untersuchungs- und Element-bestimmungsmethoden des Umweltlabors der Niedersächsischen Forstlichen Versuchsanstalt und des Zentrallabors II des Forschungszentrums Waldökosysteme, Teil 4: Probenvorbereitungs- und Untersuchungsmethoden, Qualitätskontrolle und Datenverarbeitung; Berichte des Forschungszentrums Waldökosyst. B, Bd. 49, Untersuchungsmethode Boden AKEG1.1
- Gutachterausschuss Forstliche Analytik: Handbuch Forstliche Analytik. König, N. and Bartners, H. Eds. 2005. Loseblatt-Sammlung der Analysemethoden im Forestbereich, 433 pg. (Method A3.2.1.3)



**Soil Analysis Method 11 (SA11): Aqua Regia Extractant Determinations  
P, Ca, K, Mg, Mn, Cu, Pb, Cd, Zn, Al, Fe, Cr, Ni, S, Hg, Na**

<b>Aqua Regia extractant determinations P, Ca, K, Mg, Mn, Cu, Pb, Cd, Zn, Al, Fe, Cr, Ni, S, Hg, Na</b>	
Method sheet	SA11
Reference methods	ISO 11466
Method suitable for	Organic Layer, Mineral Layer

## I. Relevance in ICP Forests

Aqua Regia extractant determinations (P, Ca, K, Mg, Mn)

Priority	Level I	Level II
<b>Organic Layer</b>		
OL	Optional	Optional
OF+OH, H-layers	Mandatory	Mandatory
<b>Mineral layer</b>		
0 - 10 cm	Optional	Optional
10 – 20 cm	Optional	Optional
20 – 40 cm	Optional	Optional
40 – 80 cm	Optional	Optional

Aqua Regia extractant determinations (Cu, Pb, Cd, Zn)

Priority	Level I	Level II
<b>Organic Layer</b>		
OL	Optional	Optional
OF+OH, H-layers	Mandatory	Mandatory
<b>Mineral layer</b>		
0 - 10 cm	Mandatory	Mandatory
10 – 20 cm	-	-
20 – 40 cm	-	-
40 – 80 cm	-	-

Aqua Regia extractant determinations (Al, Fe, Cr, Ni, S, Hg, Na)

Priority	Level I	Level II
<b>Organic Layer</b>		
OL	Optional	Optional
OF+OH, H-layers	Optional	Optional
<b>Mineral layer</b>		
0 - 10 cm	Optional	Optional
10 – 20 cm	-	-
20 – 40 cm	-	-
40 – 80 cm	-	-

## II. Principle

The dried sample is extracted with a hydrochloric/nitric acid mixture by standing for 16 h at room temperature, followed by boiling under reflux for 2 h. The digestion by the use of a microwave apparatus is not allowed as results are not comparable for matrices which give residues after digestion. The extract is then clarified and made up to volume with nitric acid. Elements are determined by spectrometry.

## III. Apparatus

Analytical balance (accuracy 0.001 g)

Desiccator (2 l)

Reaction vessel (250 ml)

Reflux condenser

Absorption vessel, non return type, containing 15 ml of nitric acid (0.5 mol/l) (only necessary for determination of mercury)

Roughened glass beads or antibumping granules

Temperature-controlled heating apparatus

Funnel (diam. approx. 110 mm)

Volumetric flask (110 ml)

Filter paper (diam. 150 mm, pore size approx. 8 µm)

Atomic Absorption Spectrometer (AAS) / Flame Emission Spectrometer (FES) / Inductively Coupled Plasma Spectrometer (ICP) / Colorimeter

## IV. Reagents

Watesr (grade 2)

Hydrochloric acid (HCl) concentration 12 mol/l, 1.19 g/ml

Nitric acid (HNO<sub>3</sub>) concentration 15.8 mol/l, 1.42 g/ml

Nitric acid (HNO<sub>3</sub>) concentration 0.5 mol/l

## V. Procedure

### *Laboratory sample*

Weigh 3.000 g air-dried soil (particle size < 2 mm) of known moisture content in the 250 ml reaction vessel.

*Note: Because we are interested in the easily available elements, it is not allowed to mill the < 2mm sample. This deviates from the ISO standard.*

### *Aqua regia extraction*

Moisten with about 0.5 ml to 1.0 ml of water and add, while mixing, 21 ml of hydrochloric acid followed by 7 ml of nitric acid (15.8 mol/l), drop by drop if necessary, to reduce foaming. Connect the condenser (and the absorption vessel) to the reaction vessel, and allow to stand for 16 h at room temperature to allow for slow oxidation of the organic matter in the soil.

The amount of aqua regia is sufficient only for oxidation of about 0.5 g of organic carbon. If there is more than 0.5 g of organic carbon in the 3 g subsample, proceed as follows. Allow the first reaction with the aqua regia to subside. Then add an extra 1 ml of nitric acid (15.8 mol/l) only to every 0.1 g of organic carbon above 0.5 g. Do not add more than 10 ml of nitric acid at any time, and allow any reaction to subside before proceeding further.

Raise the temperature of the reaction mixture slowly until reflux conditions are reached and maintain for 2 h, ensuring that the condensation zone is lower than 1/3 of the height of the condenser, then allow to cool.

Allow the reaction vessel to stand so that most of any insoluble residue settles out of suspension. (Add the contents of the absorption vessel to the reaction vessel, via the condenser, rinsing both the absorption vessel and condenser with a further 10 ml of nitric acid (0.5 mol/l)). Decant the relatively sediment-free supernatant carefully onto a filter paper, collecting the filtrate in a 100 ml volumetric flask. Allow all the initial filtrate to pass through the filter paper, then wash the insoluble residue onto the filter paper with a minimum of nitric acid (0.5 mol/l). Collect this filtrate with the first. before proceeding further. The extract thus prepared is ready for the determination of trace elements, by an appropriate method.

### *Determination of elements (P, Ca, K, Mg, Mn, Cu, Pb, Cd, Zn, Al, Fe, Cr, Ni, S, Hg, Na)*

Measure the elements cations in the extract using one of the spectrometric determination methods.

*Note: ISO 11047 can be used as a guideline for the determination of Cd, Cr, Cu, Pb, Mn, Ni and Zn.*

## VI. Calculation

Determination of elements (P, Ca, K, Mg, Mn, Cu, Pb, Cd, Zn, Al, Fe, Cr, Ni, S, Hg, Na)

Calculation according to apparatus.

## VII. Report

Report aqua regia extract determinations (mg/kg) with 1 decimal place on the basis of oven-dried soil.

*Note: Laboratories which have the possibility to determine the Cu content up to 2 decimal places and the Hg content up to 3 decimal places, are given the opportunity to report accordingly.*

## VIII. Reference

ISO 11466. 1995. Soil Quality – Extraction of trace elements soluble in *aqua regia*. International Organization for Standardization. Geneva, Switzerland. 6 p. (available at [www.iso.ch](http://www.iso.ch))

ISO 11047. 1998. Soil Quality – Determination of cadmium, chromium, cobalt, copper, lead, manganese nickel and zinc. Flame and electrothermal atomic absorption spectrometric methods. International Organization for Standardization. Geneva, Switzerland. 6 p. (available at [www.iso.ch](http://www.iso.ch))



**Soil Analysis Method 12 (SA12): Determination of Total Elements Ca,  
Mg, Na, K, Al, Fe, Mn**

<b>Total Elements: Ca, Mg, Na, K, Al, Fe, Mn</b>	
<b>Method 1 : Dissolution with hydrofluoric and perchloric acids</b>	
Method sheet	SA12A
Reference methods	ISO 14869
Method suitable for	Organic Layer, Mineral Layer

## I. Relevance in ICP Forests

Priority	Level I	Level II
<b>Organic Layer</b>		
OL	-	-
OF+OH, H-layers	-	-
<b>Mineral layer</b>		
0 – 10 cm	-	Optional
10 – 20 cm	-	Optional
20 – 40 cm	-	Optional
40 – 80 cm	-	Optional

## II. Principle

This method specifies the complete dissolution, using hydrofluoric and perchloric acids, of the following elements in soils:

**Al, Ba, Cd, Ca, Cs, Cr, Co, Cu, Fe, K, Li, Mg, Mn, Na, Ni, P, Pb, Sr, V, Zn.**

This procedure may be appropriate for the subsequent determination of other elements provided their concentrations are high enough relative to the sensitivity of the measurement methods. The low acid concentration of the final solution allows the use of a large range of analytical devices and the volatilisation of silicon simplifies analytical procedures.

The dried and ground sample is pre-treated to destroy organic matter, and then digested with a mixture of hydrofluoric and perchloric acids. After evaporation to near dryness, the residue is dissolved in dilute hydrochloric or nitric acid. Hydrofluoric acid decomposes silicates by the reaction of F with Si to form volatile SiF<sub>4</sub>. As it evaporates last, perchloric acid forms readily-soluble perchlorate salts.

To minimise the danger of acid ejection due to violent oxidation of organic matter by perchloric acid, two alternative procedures have been adopted to destroy organic matter prior to digestion:

- dry ashing at 450 °C;
- pretreatment with nitric acid.

### III. Apparatus

Mill

Drying oven and desiccator

Analytical balance (accuracy 0.0001 g)

Crucible of fused silica or platinum (10 - 30 ml)

Furnace (temperature 450 °C)

Evaporating dishes made of polytetrafluoroethylene (PTFE)

Hot plate (150 °C)

Fume hood

Volumetric flask of polypropylene (50 ml)

Atomic Absorption Spectrometer (AAS) / Flame Emission Spectrometer (FES) / Inductively Coupled Plasma Spectrometer (ICP)

### IV. Reagents

Water (grade 2)

Hydrofluoric acid (HF), conc. 27.8 mol/l, = 1.16 g/ml

Perchloric acid (HClO<sub>4</sub>), conc. 11.6 mol/l, = 1.67 g/ml

Hydrochloric acid (HCl), conc. 12.1 mol/l, = 1.19 g/ml

Nitric acid (HNO<sub>3</sub>), conc. 15.2 mol/l, = 1.41 g/ml

### V. Procedure

#### Attention !

**Always use latex gloves while working with HF and keep the ointment against HF acid bites ready for eventual accidents !**

#### Laboratory sample

Use air-dried soil milled as fine as possible. Weigh precisely 0.250 g of the milled sample.

#### Destruction organic matter

##### Dry ashing

Transfer soil sample to a crucible. Place the crucible in the furnace and allow the temperature to reach 450 °C, progressively over 1 h. Maintain this temperature for 3 h. Allow the furnace to cool to room temperature and transfer the ash quantitatively to a PTFE evaporating dish with a minimum amount of water. Using a platinum crucible of about 30 ml avoids ash being transferred to a PTFE dish and allows digestion to be performed in the same container as dry ashing.

##### Nitric acid pre-treatment

Transfer soil sample to an evaporating dish and add 5 ml of nitric acid. Place the dish on the hot plate at 150 °C and evaporate until approximately 1 ml of nitric acid remains. Note that several

successive additions of nitric acid may be necessary until the emission of nitrous vapours ceases to remove all the organic matter. In such cases, remove the dish from the hot plate and cool to room temperature before adding the next portion of nitric acid. After the last addition of nitric acid, remove the dish from the hot plate and cool to room temperature.

#### *Total digestion*

Add 5 ml of hydrofluoric acid and 1.5 ml of perchloric acid to the pretreated test portion in the PTFE dish or the 30 ml platinum crucible. Heat this mixture on the hot plate until the dense fumes of perchloric acid and silicon tetrafluoride cease. Do not allow the mixture to evaporate to complete dryness. Remove the dish from the hot plate, allow to cool, add 1 ml of hydrochloric acid or 1 ml of nitric acid and approximately 5 ml of water to dissolve the residue. Warm the dish briefly on the hot plate to assist dissolution. Transfer this solution quantitatively to the 50 ml volumetric flask, fill to the mark and mix well.

A solid phase remaining in the resultant solution indicates incomplete dissolution. It may be of no importance with respect to the elements of interest, especially when pure silica constitutes the solid phase, but in that case, the procedure shall be completed by one of the following stages.

The procedure is stopped at this point and failure of total dissolution with a possible effect on the determination of total contents is noted in the test report.

The procedure is adjusted to improve the dissolution. One or a combination of the three following treatments is carried out.

The procedure is started again with a new test portion but a further dose of 5 ml of hydrofluoric acid and 1.5 ml of perchloric acid is added after evaporation of the first one to near dryness. The second dose is also evaporated as above and the procedure is carried on as described above.

The procedure is started again with a new test portion but after the addition of hydrofluoric and perchloric acids the mixture is left overnight at room temperature before being heated as described above.

The whole procedure is not changed but the mass of the test portion is reduced.

If a solid phase remains in spite of these further treatments, then failure of total dissolution is mentioned in the test report.

#### *Blank test*

Use the same procedure, without the sample, to perform at least one blank test within each batch of digestions.

#### *Determination of total elements (Ca, Mg, K, Na, Al, Fe, Mn)*

Measure the total elements in the extract using one of the spectrometric determination methods.

## **VI. Calculation**

Determination of total elements (Ca, Mg, K, Na, Al, Fe, Mn)

Calculation according to apparatus.

## **VII. Report**

Report total elements (mg/kg) with one decimal place on the basis of oven-dried soil.

## **VIII. References**

ISO 14869-1. 2001. Soil Quality – Dissolution for the determination of total element content - Part 1: Dissolution with hydrofluoric and perchloric acids. International Organization for Standardization. Geneva, Switzerland. 5 p. (available at [www.iso.ch](http://www.iso.ch))



**Total Elements: Ca, Mg, Na, K, Al, Fe, Mn**

**Method 2 : Total element analysis by fusion with lithium metaborate**

Method sheet	SA12B
Reference methods	ISO 14869
Method suitable for	Organic Layer, Mineral Layer

**I. Relevance in ICP Forests**

Priority	Level I	Level II
<b>Organic Layer</b>		
OL	-	-
OF+OH, H-layers	-	-
<b>Mineral layer</b>		
0 – 10 cm	-	Optional
10 – 20 cm	-	Optional
20 – 40 cm	-	Optional
40 – 80 cm	-	Optional

**II. Principle**

This method specifies the fusion using lithium metaborate.

**III. Apparatus**

Platinum crucibles

Muffle furnace

Magnetic stirring devices

Analytical balance (accuracy 0.0001 g)

Filter paper prewashed (with a 10% HNO<sub>3</sub> or HCl solution)

**IV. Reagents**

Lithium metaborate (LiBO<sub>2</sub>) on powder

Nitric acid (HNO<sub>3</sub>), 4 %

## V. Procedure

### *Laboratory sample*

Use air-dried soil (milled < 0.4 mm). Weigh 0.4 g of the milled sample.

### *Destruction organic matter*

The sample is put to each platinum crucible and pre-ignited at 850 C for 30 min as to avoid damaging the platinum crucible when it would be mixed with lithium metaborate. The reason for this is that the soil organic matter can cause reduction of the platinum during the fusion.

### *Fusion*

After the crucibles are cooled (usually it takes one night) the pre-ignited soil is mixed thoroughly (by means of a pipette tip) with 2 g of lithium metaborate powder in a platinum crucible and fused for 15 min at 950°C in a preheated muffle furnace. The crucible and flux that is formed are allowed to cool for one night. The reason for this is that if we try to remove them from the furnace immediately after heating by means of something metallic, there will be a reaction between the platinum and the metal.

The crucibles are immersed in a 100 ml beaker and covered with 70-80 ml of 4% nitric acid. A magnetic stirring bar is then placed inside the crucible and stirring can begin immediately. The flux is dissolved in 3 to 4 hr (occasionally it might take a little more) and the solution is made up to 100 ml, filtered through a prewashed (with a 10% HNO<sub>3</sub> or HCl solution) paper filter of 0.45 mm and stored for analysis.

### *Blank test*

Use the same procedure, without the sample, to perform at least one blank test within each batch of digestions.

### *Determination of total elements (Ca, Mg, K, Na, Al, Fe, Mn)*

Measure the total elements in the extract using one of the spectrometric determination methods.

## VI. Calculation

Determination of total elements (Ca, Mg, K, Na, Al, Fe, Mn)

Calculation according to apparatus.

## VII. Report

Report total elements (mg/kg) with one decimal place on the basis of oven-dried soil.

## VIII. References

Michopoulos, P. 1995. Studies on manganese cycling in forest soils. PhD Thesis. University of Aberdeen.

Potts, P.J. 1987. A handbook of Silicate Rock Analysis. Blackie, New York.

**Soil Analysis Method 13 (SA13). Determination of Acid Oxalate Extractable Fe and Al**

<b>Acid Oxalate Extractable Fe and Al</b>	
Method sheet	SA13
Reference methods	ISRIC, 2002
Method suitable for	Organic Layer, Mineral Layer

## I. Relevance in ICP Forests

Priority	Level I	Level II
<b>Organic Layer</b>		
OL	-	-
OF+OH, H-layers	Optional	Optional
<b>Mineral layer</b>		
0 – 10 cm	Optional	Mandatory
10 – 20 cm	Optional	Mandatory
20 – 40 cm	Optional	Mandatory <sup>1</sup>
40 – 80 cm	Optional	Mandatory <sup>1</sup>

<sup>1</sup> In case of re-assessment (if the parameter was already measured according to the reference method in a previous survey), the measurement is optional

## II. Principle

The sample is shaken with a complexing acid ammonium oxalate solution dissolving the "active" or "short range order" ( "amorphous") compounds of Fe, Al (and Si) and a (variable) amount of organically complexed Fe and Al which are determined in the extract by AAS or ICP-AES. The ammonium oxalate buffer extraction is sensitive to light, especially UV light. The exclusion of light during the extraction reduces the dissolution effect of crystalline oxides.

Superfloc is added as a flocculent to the solution to remove the fine, suspended, solid particles, often made up of iron minerals (ferrihydrite). While conducting this analysis for classification purposes, no Superfloc should be added.

## III. Apparatus

Reciprocating shaking machine

Centrifuge

Atomic absorption spectrophotometer (with nitrous oxide/acetylene flame) or Inductive Coupled Plasma Atomic Emission Spectrofotometer (ICP-AES)

Polythene shaking bottles, wide mouth, 100 and/or 250 ml

## IV. Reagents

In this procedure *distilled* water is used since deionised water may contain Si.

Acid ammonium oxalate solution, 0.2 M in oxalate, pH 3:

Dissolve 81 g  $(COONH_4)_2 \cdot H_2O$  and 54 g  $(COOH)_2 \cdot 2H_2O$  in 4.5 l water and make to 5 l. Prepare 1 l of two separate 0.2 M solutions of  $NH_4$ -oxalate (28 g/l) and oxalic acid (25 g/l) and add some of either solution to the mixture until the pH is 3.

Potassium suppressant solution, 10,000 mg/l K:

Dissolve 19 g KCl in 800 ml water and make up to 1 l.

"Superfloc" solution, 0.2%:

Dissolve 0.1 g superfloc flocculent in 50 ml water. Stir overnight in the dark. (Note: store in the dark. This solution can be kept for about a week).

Superfloc is a flocculent used in waste water treatment. E.g. Cyanamid Superfloc N-100 and Floerger Kemflock FA 20H

Diluent solution (5x):

Make 2.38 g KCl and 25 ml conc. HCl to 1 l with water.

Diluent solution (20x):

Make 2.01 g KCl, 158 ml acid ammonium oxalate solution and 21 ml conc. HCl to 1l with water.

Standard solutions Fe and Al, 250 mg/l:

Dilute standard analytical concentrate ampoules (1g/l) according to instruction to make 1000 mg/l solutions. Dilute each to 250 mg/l by pipetting 50 ml into a 200 ml volumetric flask and making up to volume with water.

Mixed standard series of Fe and Al:

1. To each of five 250 ml volumetric flasks add 50 ml of the acid oxalate reagent, 25 ml of the KCl suppressant solution and 5 ml conc. HCl (or 10 ml 6 M HCl)
2. Of each 250 mg/l standard solution pipette 0-5-10-25-50 ml into the 250 ml volumetric flask (same volumes into same flasks respectively) and make to volume with water.

The standard series are then: Fe, Al, 0-5-10-25-50 mg/l.

## V. Procedure

1. Weigh 1 g of fine earth (accuracy 0.01 g) into a 100 ml shaking bottle. Include two blanks and a control sample.
2. Add 50.0 ml oxalate reagent and close the bottle. (Note: for soils with relatively high contents of oxalate-extractable material (Al, Fe>2%) use 100.0 ml oxalate reagent and a 250 ml shaking bottle).
3. Shake for four hours **in the dark**.
4. Transfer about 35 ml to a 50 ml centrifuge tube
5. Add 3-4 drops of superfloc solution and swirl well (preferably on a Vortex mixer) and centrifuge.

6. Prepare 5x and 20x dilutions:

5x dilution

Pipette 1 ml of the clear supernatant and 4 ml of the diluent solution (5x) into a test tube and homogenise.

20x dilution

Pipette 1 ml of the clear supernatant solution and 19 ml ( by varispencer or burette) of the diluent solution (20x) into a wide test tube and homogenise

7. Measure Fe by AAS at 248.3 nm using an air/acetylene flame and measure Al by AAS at 309.3 nm using a nitrous oxide/acetylene flame. Or measure by ICP-AES. Refer to the manufacturer's manual for operation.

*Note: In case of over ranged (diluted) extracts, dilute these once more 1+1 with the zero standard solution. Therefore, of the latter an extra 250 or 500 ml should be prepared. Change the calculation accordingly.*

## VI. Calculation

Calculate the oxalate extractable Fe and Al, on the basis of the air-dried soil according to the following equation:

$$Fe, Al (mg / kg) = \frac{(a - b) \times df}{s} \times m_{ox.} \times 1000$$

where

- a = mg/l Fe, Al in diluted sample extract
- b = diato in diluted blank
- df = dilution factor
- ml ox. = ml of oxalate reagent used (50 or 100)
- s = air dry sample weight in milligram
- 1000 = conversion factor to mg/kg basis

## VII. Report

Report oxalate extractable Fe and Al (mg/kg) with one decimal place on the basis of oven-dried soil.

## VIII. Reference

ISRIC, FAO. 2002. Procedures for soil analysis. Sixth ed. ISRIC Technical Paper 9. L.P. Van Reeuwijk (ed). Wageningen, The Netherlands.

USDA National Resources Conservation Service, 2004. Survey Laboratory Methods Manual. Soil Investigations report N°.42, Version 4.0, pages 312-317.

**Soil Analysis Method 14 (SA14): Determination of the Soil Water  
Retention Characteristic**

Soil water retention characteristic (pF analysis) (SWRC)	
Method sheet	SA14
Reference method	ISO 11274
Method suitable for	Mineral and organic soil horizons, undisturbed samples

## I. Relevance in ICP Forests

PRIORITY	LEVEL I AND LEVEL II	LEVEL II CORE
<b>Organic layer</b>		
OL	-	-
OF-OH, H - layers	Optional	Mandatory if > 5 cm
<b>Mineral layer</b>		
0 – 20 cm	Optional	Mandatory
20 – 40 cm	Optional	Mandatory
40 – 80 cm	Optional	Mandatory
> 80 cm	Optional	Optional
Extra (specific) layer	Optional	Optional

The volumetric water content at matric heads 0, -1, -5, -33 and -1500 kPa plus the dry soil bulk density are mandatory to determine on Level II core plots. Extra observations of the SWRC at pressures -10, -100 and -250 kPa are optional but they greatly improve fitting the soil water retention characteristic (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 (BD) (lowest pressure at about  $10^{-6}$  kPa) derived in the oven at 105°C.

## II. Principle

This method sheet describes the determination of the soil water retention in the laboratory, extending from saturated soil (no pressure or suction; 0 kPa) to oven-dry soil (about  $10^{-6}$  kPa) based on measurements of the drying or desorption curve. All methods described by ISO 11274 are allowed, except method B, using a porous plate and burette apparatus for matric pressures from 0 to -20 kPa.

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

The volumetric soil water content at matric pressure 0 kPa is approximated by the total porosity of the soil.

The ISO 11274:1998 allows 4 methods to determine matric pressures within specific ranges:

method using sand, kaolin or ceramic suction tables for determination of matric pressures from 0 kPa to - 50 kPa;

method using a porous plate and burette apparatus for determination of matric pressures from 0 kPa to - 20 kPa; (single sample)

method using a pressurized gas and a pressure plate extractor for determination of matric pressures from - 5 kPa to - 1500 kPa;

method using a pressurized gas and pressure membrane cells for determination of matric pressures from - 33 kPa to - 1500 kPa.

Since method B allows only processing a single sample at the time, use of this method is not recommended. Laboratories are free to apply methods A, C and D according to the ISO 11274 standard. Guidelines for choosing the most appropriate method for specific soil types are given in ISO 11274, chapter 3.

Before applying methods A, C or D, general recommendations for sample preparation are:

For measurements at pressures from 0 to -50 kPa, use a nylon mesh to retain the soil sample in the sleeve and secure it with an elastic band or tape;

Ensure maximum contact between the soil core, mesh and the porous contact medium of the suction tables, plates or membranes; remove any small projecting stones if necessary;

Avoid smearing the surface of (clayey) soils, especially when water saturated;

Inspect the sample for bioturbation (worms, isopods) or germination of seeds during analysis; the use of a biocide is discouraged;

Report the temperature at which the water-retention measurements are made;

Ideally, measurements use field-moist samples [i.e. do not dry the undisturbed samples first (hysteresis effect)]. Prior to analysis, samples are saturated with water.

Respect wetting times before starting measurements to obtain a saturated sample. General guidelines for wetting times according to ISO 11274 are:

- sand 1 to 5 days
- loam 5 to 10 days
- clay 5 to 14 days or longer
- peat 5 to 20 days.

**Table I.14.1:** Overview of matric heads to assess for the determination of the SWRC.

Matric potential $\psi$			Recommended instrument/Method	Estimator	M/O
cm H <sub>2</sub> O	pF	kPa			
1	0.0	0	Pycnometer	$\approx \theta_{sat} = WHC =$ Total porosity	M
10	1.0	-1	Sand suction table (method A)		M
51	1.7	-5			M
102	2.0	-10		FC sand	O
337	2.5	-33	Kaolin suction table (method A)	FC siltloam	M
1022	3.0	-100	Pressure plate extractor (method C) or Pressure membrane cells (method D)	FC clay	O
2555	3.4	-250			O
15330	4.2	-1500		PWP	M
10 <sup>7</sup>	7.0	-10 <sup>6</sup>	Oven	Dry BD	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 H<sub>2</sub>O or 1 cm H<sub>2</sub>O column = 0.097885 kPa
- 3) 100 kPa = 1 bar

### III. Apparatus

**Method A:** Determination of the soil water characteristic using sand, kaolin and ceramic suction tables

Suction table (watertight, rigid container with outlet in base and close fitting cover)

Drainage system for suction table, enabling to maintain suction at specific matric pressures

Sand, silt or kaolin packing material, appropriate for use in suction tables (homogenous, sieved, graded and washed, free of organic material or salts). Material should achieve the required air entry values (see ISO 11274 for details)

Drying oven capable of maintaining temperature of 105 ± 2 °C

Balance (accuracy 0.1% of measured value)

**Method C:** Determination of soil water characteristic by pressure plate extractor

Pressure plate extractor with porous ceramic plate

Sample retaining rings/soil cores with discs and/or lids

Air compressor (1700-2000 kPa), nitrogen cylinder or other pressurized gas)

Pressure regulator and test gauge

Drying oven capable of maintaining temperature of 105 ± 2 °C

Balance (accuracy 0.1% of measured value)

Follow the manufacturer's instruction to assemble and operate the apparatus.

**Method D:** Determination of soil water characteristic using pressure membrane cells

Pressure cells with porous baseplates

Cellulose acetate membrane

Pressure regulator

Air compressor (1700-2000 kPa, nitrogen cylinder or other pressurized gas)

Drying oven capable of maintaining temperature of  $105 \pm 2^\circ\text{C}$

Balance (accuracy 0.1% of measured value)

Follow the manufacturer's instruction to assemble and operate the apparatus.

#### IV. Procedure

**Method A:** Determination of the soil water characteristic using sand, kaolin and ceramic suction tables

Weigh the cores and then place them on a suction table at the desired matric pressure with table cover closed. The reference 0 cm height for setting the suction level is the middle of the core;

Leave the cores for 7 days (minimum equilibration time). Equilibrium is reached if daily change in mass of the core is less than 0,02 %;

If equilibrium is reached, weigh the cores, if not, replace cores firmly onto the suction table and wait until equilibrium is reached.

**Method C:** Determination of soil water characteristic by pressure plate extractor

Take small subsamples from the undisturbed sample: soil cores of approximately 5 cm diameter and between 5 mm and 10 mm in height; smaller samples for lower pressures are used in order to avoid long equilibration times;

It is acceptable to use disturbed samples at pressures lower than - 100 kPa, providing that the disturbance consists only in breaking off small pieces of soil and not in compressing or remoulding the soil.

Use at least three replicate samples of each sample and place them on a presaturated plate;

Wet the samples by immersing the plate and the samples until a thin film of water can be seen on the surface of the samples;

Create a saturated atmosphere in the extractor;

Apply the desired gas pressure and keep to a constant level, check for leaks;

Record on a daily basis the evacuated water from the samples, when no change are observed (volume in a burette remains static) the samples have come to an equilibrium;

At equilibrium status, soil samples are weighed, oven-dried and reweighed to determine the water content at the predetermined pressures

**Method D:** Determination of soil water characteristic using pressure membrane cells

Soil subsamples are placed on a porous cellulose acetate membrane

Equilibrium status is attained when water outflow from the pressure cell ceases and soil water content is determined by weighing, oven-drying and reweighing the sample.

Gas pressure methods are only suited to determine matric pressures below - 33 kPa

## V. Calculation

### V.1. Volumetric water content

ISO 11274 describes two procedures:

Procedure for soils containing less than 20 % coarse material (diameter greater than 2 mm)

Procedure for stony soils; conversion of results to a fine earth basis

#### 1. For soils with less than 20% coarse material:

Calculate the water content mass ratio at matric pressure  $\psi_i$  using the formula:

$$WC\psi_i = (M\psi_i - M_{dry}) / M_{dry}$$

where

$WC\psi_i$  is the water content mass ratio at a matric pressure  $\psi_i$ , in grams;

$M\psi_i$  is the mass of the soil sample at matric pressure  $\psi_i$ , in grams;

$M_{dry}$  is the mass of the oven-dried soil sample, in grams.

Calculate the volumetric water content at matric pressure  $\psi_i$  using the formula:

$$\theta\psi_i = [(M\psi_i - M_{dry}) / (V \times \rho_w)] \times 10^{-3}$$

alternatively

$$\theta\psi_i = WC\psi_i \times (\rho_b / \rho_w)$$

where

$\theta\psi_i$  is the water content volume fraction at matric pressure  $\psi_i$ , expressed in  $m^3 m^{-3}$  (volume of water per volume of soil);

$M\psi_i$  is the mass of the soil sample at matric pressure  $\psi_i$ , in grams;

$M_{dry}$  is the mass of oven dried soil sample, in grams;

$V$  is the volume of the soil sample in  $m^3$

$\rho_w$  is the density of water, in  $kg m^{-3}$

$\rho_b$  is the bulk density of oven dried soil at 105°C, in  $kg m^{-3}$ .

#### 2. For soils with more than 20% coarse material, data needs conversion to a fine earth basis as follows:

The volumetric water content of the fine earth ( $\theta_f$ ) equals:

$$\theta_f = \theta_t / (1 - \theta_s)$$

where:

$\theta_f$  water content of the fine earth, expressed as a volume fraction ( $m^3 m^{-3}$ );

$\theta_s$  volume of non-porous stones, expressed as a fraction of total core volume ( $m^3 m^{-3}$ );

$\theta_t$  is the water content of the total earth, expressed as a fraction of total core volume

( $\text{m}^3 \text{ m}^{-3}$ );

For porous stones, a different correction should be applied as described in ISO 11274.

If volumetric water content is reported on fine earth basis, this should be clearly reported along with the volume of non-porous stones in the sample.

### **V.2. Calculation of the total porosity**

A value for porosity can be calculated from the bulk density  $\rho_{\text{bulk}}$  and particle density  $\rho_{\text{particle}}$ :

$$\phi = 1 - \frac{\rho_{\text{bulk}}}{\rho_{\text{particle}}}$$

Often the particle density or true density of soil is approximated by  $2650 \text{ kg.m}^{-3}$  (mineral density of quartz). But the direct measurement of the particle density is strongly recommended to be done by the means of a pycnometer.

### **V.3. Determination of dry bulk density**

Determination of dry bulk density is done according to method SA04. The dry bulk density (BD) is recorded in  $\text{kg m}^{-3}$  with no decimal places.

In the case of stony or gravelly soils the bulk density of the fine earth fraction ( $< 2 \text{ mm}$ ) should be reported. Furthermore, the bulk density of the coarse fragments should be known, but this may be approximated as  $2650 \text{ kg.m}^{-3}$ .

## **VI. Report**

Report for each undisturbed soil sample, the raw volumetric soil water content ( $\theta = \text{VWC in } \text{m}^3 \text{ m}^{-3}$ ) with four decimal places using the xx20xx.SWA data form. Report the dry bulk density (BD) in  $\text{kg m}^{-3}$  without decimal places using the xx20xx.SWC file.

Together with the laboratory results, following field data should be reported: plot ID, sampling data, pit ID, code depth layer, horizon number, sample ring depth (upper and lower side of the ring) in cm below the top of the mineral soil.

## **VII. References**

ISO 11274:1998(E). Soil Quality – Determination of the water-retention characteristic – Laboratory methods. International Organization for Standardization. Geneva, Switzerland. 20 p.  
(available at [www.iso.ch](http://www.iso.ch))



## **Annex 2: Guidelines for Forest Soil Description**



	<b>Parameter</b>	<b>Report in file</b>	<b>M/O</b>
<b>1</b>	<b><u>GENERAL SITE INFORMATION</u></b>		
1.1.	<b>Observation plot number</b>	*.PLS, *.PRF, *.PFH, *.SOM, *.LQA	M
1.2.	<b>Profile ID</b>	*.PRF, *.PFH	M
1.3.	<b>Date of profile description</b>	*.PRF	M
1.4.	<b>Profile latitude-/ longitude coordinates</b>	*.PRF	M
1.5.	<b>Elevation</b>	*.PRF	O
<b>2</b>	<b><u>SOIL FORMING FACTORS</u></b>		
2.1.	<b>Climate and weather conditions</b>		
2.1.1.	<i>Present weather conditions</i>		
2.1.2.	<i>Former weather conditions</i>		
2.2.	<b>Soil climate</b>		
2.2.1.	<i>Cryic horizon</i>		
2.2.2.	<i>Soil climate classification</i>		
2.3.	<b>Topography</b>		
2.3.1.	<i>Slope position</i>		
2.3.2.	<i>Slope form</i>		
2.3.3.	<i>Slope gradient</i>		
2.3.4.	<i>Slope length</i>		
2.3.5.	<i>Slope orientation</i>		
2.4.	<b>Land use</b>	*.PRF	M
2.5.	<b>Human influence</b>		
2.6.	<b>Vegetation</b>		
2.6.1.	<i>Forest type</i>		
2.6.2.	<i>Tree species composition</i>		
2.7.	<b>Parent material</b>	*.PRF	M
2.8.	<b>Natural Drainage Classes</b>		
2.9.	<b>Water availability</b>	*.PLS	M
2.10.	<b>Period of water saturation</b>		
2.11.	<b>External drainage</b>		
2.12.	<b>Flooding</b>		
2.12.1.	<i>Frequency</i>		
2.12.2.	<i>Duration (days/year)</i>		
2.12.3.	<i>Depth (of standing water)</i>		
2.13.	<b>Groundwater table</b>		
2.13.1.	<i>Mean highest and mean lowest groundwater depth</i>	*.PRF	O
2.13.2.	<i>Type of water table</i>	*.PRF	O

<b>2.14.</b>	<b>Rock outcrops</b>		
2.14.1.	Surface cover		
2.14.2.	Distance between rock outcrops		
<b>2.15.</b>	<b>Coarse surface fragments</b>		
2.15.1.	Surface cover		
2.15.2.	Size classes		
<b>2.16.</b>	<b>Erosion &amp; sedimentation</b>		
2.16.1.	Type of erosion/sedimentation		
2.16.2.	Area affected		
2.16.3.	Degree		
2.16.4.	Activity		
<b>2.17.</b>	<b>Surface sealing (FAO, 2006)</b>		
2.17.1.	Thickness		
2.17.2.	Consistence when dry		
<b>2.18.</b>	<b>Surface cracks (FAO, 2006)</b>		
2.18.1.	Size (Width)		
2.18.2.	Distance between cracks		
<b>2.19.</b>	<b>Salt</b>		
2.19.1.	Cover		
2.19.2.	Thickness		
<b>2.20.</b>	<b>Profile depth</b>	*.PRF	M

<b>3</b>	<b>SOIL HORIZON DESCRIPTION</b>		
<b>3.1.</b>	<b>Horizon boundary</b>		
3.1.1.	Horizon number	*.PFH	M
3.1.2.	Depth of horizon limits	*.PFH	M
3.1.3.	Distinctness	*.PFH	O
3.1.4.	Topography	*.PFH	O
<b>3.2.</b>	<b>Photographic recordings</b>		
<b>3.3.</b>	<b>Soil colour</b>	*.PFH	M
<b>3.4.</b>	<b>Mottling</b>		
3.4.1.	Colour		
3.4.2.	Abundance		
3.4.3.	Size		
3.4.4.	Contrast		
3.4.5.	Boundary		
<b>3.5.</b>	<b>Redoximorphic properties</b>		
3.5.1.	Reducing conditions		
3.5.2.	Reductimorphic and oximorphic colours		

<b>3.6.</b>	<b>Texture of the fine-earth fraction</b>	*.PFH	M
<b>3.7.</b>	<b>Rock fragments</b>		
3.7.1.	<i>Abundance</i>	*.PFH	M
3.7.2.	<i>Size of rock fragments and artefacts</i>		
3.7.3.	<i>Dominant shape of rock fragments</i>		
3.7.4.	<i>State of weathering of rock fragments</i>		
3.7.5.	<i>Nature (type) of rock fragments</i>		
<b>3.8.</b>	<b>Andic material</b>		
<b>3.9.</b>	<b>Soil structure</b>		
3.9.1.	<i>Type</i>	*.PFH	M
3.9.2.	<i>Size</i>		
3.9.3.	<i>Grade</i>		
<b>3.10.</b>	<b>Consistence</b>		
3.10.1.	<i>Consistence when dry</i>		
3.10.2.	<i>Consistence when moist</i>		
3.10.3.	<i>Consistence when wet</i>		
<b>3.11.</b>	<b>Cutanic features</b>		
3.11.1.	<i>Type</i>		
3.11.2.	<i>Abundance</i>		
3.11.3.	<i>Contrast</i>		
3.11.4.	<i>Location</i>		
<b>3.12.</b>	<b>Porosity</b>	*.PFH	O
<b>3.13.</b>	<b>Cementation and compaction</b>		
3.13.1.	<i>Nature (type)</i>		
3.13.2.	<i>Continuity</i>		
3.13.3.	<i>Structure</i>		
3.13.4.	<i>Degree</i>		
<b>3.14.</b>	<b>Nodules and other mineral concentrations</b>		
3.14.1.	<i>Kind</i>		
3.14.2.	<i>Type</i>		
3.14.3.	<i>Abundance (by volume)</i>		
3.14.4.	<i>Size</i>		
3.14.5.	<i>Shape</i>		
3.14.6.	<i>Hardness</i>		
3.14.7.	<i>Colour</i>		
<b>3.15.</b>	<b>Roots</b>		
3.15.1.	<i>Abundance</i>	*.PFH	M*
3.15.2.	<i>Distribution</i>		

<b>3.16.</b>	<b>Other biological features</b>		
3.16.1.	<i>Kind</i>		
3.16.2.	<i>Abundance</i>		
3.16.3.	<i>Continuity</i>		
<b>3.17.</b>	<b>Carbonates</b>		
3.17.1.	<i>Presence of carbonates</i>		
3.17.2.	<i>Type of secondary carbonates</i>		
<b>3.18.</b>	<b>Gypsum</b>		
3.18.1.	<i>Abundance of gypsum</i>		
3.18.2.	<i>Abundance of secondary gypsum</i>		
<b>3.19.</b>	<b>Readily soluble salts</b>		
<b>3.20.</b>	<b>Man-made materials</b>		
<b>3.21.</b>	<b>Human-transported material</b>		
<b>3.22.</b>	<b>Soil horizon designation</b>	*.PFH	M

M: Mandatory to report

O: Optional to report

M\*:Mandatory on plots with continuous soil moisture measurements

## 0. Introduction

These guidelines are intended to help you to make the necessary field recordings and to collect other additional information enabling proper description of a forest soil profile and subsequent classification according to the World Reference Base for Soil Resources (IUSS Working Group WRB, 2007a, 2007b) as asked by the Manual Part X on Sampling and Analysis of Soil. These guidelines elaborate on variables which are not described elsewhere in this ICP Forests Manual. On the other hand, information collected in the other surveys can be very relevant to understand and interpret the soil profile correctly. In that case, reference is made to the concerning Parts of this Manual.

These guidelines are largely based on the FAO (2006) Guidelines for soil description (4<sup>th</sup> edition) but do deviate for a number of parameters:

1. Concerning the horizon designation of the organic layer (master symbol 'O'), the ICP Forests Manual continues to use the symbols OL, OF and OH, which is a tradition in the description of European forest soils whereas the FAO (2006) adopted recently the subordinate symbols i, e and a from the American soil classification system.
2. By consequence, there is not need to change the subordinate symbol 'a' standing for 'evidence of cryoturbation' as in FAO (1989) into the @ symbol as in FAO (2006).

## 1. General site information

### 1.1 Observation plot number

(to be reported in \*.PLS, \*.PRF, \*.PFH, \*.SOM and \*.LQA file)

The observation plot number is the unique number given to each plot. It is reported with 4 digits.

Example: Observation plot number [1012]

### 1.2 Profile ID

(to be reported in \*.PRF and \*.PFH file)

The soil profile identification number/code. It is reported with 4 characters.

Example: Profile number [0149]

### 1.3 Date of profile description

(to be reported in \*.PRF file)

The date of description in forms how old the data are and in what season they are recorded. The date of description is given as DDMMYY (6 digits).

Example: 160504 (16 May 2004)

## 1.4 Profile latitude-/ longitude coordinates

(to be reported in \*.PRF file)

The coordinates of the centre of the observation plot is mandatory information. In addition it is also recommended to record the latitude and longitude coordinates of the soil profile pit as accurately as possible (in degrees and sexagesimal minutes and seconds). The point observation of the soil profile is the central point of the described soil profile wall.

If possible, by preference a Global Positioning System (GPS) is used. If recording of the soil profile coordinates is impossible, then the distance (in metres and centimetres) and direction (in degrees) from the experimental plot centre should be measured and recalculated in coordinates.

The location of the profile pit is reported as:

Latitude geographic coordinates (+/- degrees, minutes, seconds) [WGS84]

Longitude geographic coordinates (+/- degrees, minutes, seconds) [WGS84]

Example:      Latitude:      51° 23' 31'' N; is reported as +512331  
                   Longitude:    11° 52' 40'' E; is reported as +115240

## 1.5 Elevation

(to be reported in \*.PRF file)

The elevation or altitude (m above sea level) of the site should be obtained as accurately as possible.

# 2. Soil forming factors

Although the description of the soil forming factors are important for the correct interpretation of the soil profile, the soil module of the database does not ask to report most of these parameters, except for land use, parent material, water availability, mean highest and mean lowest groundwater level, profile depth (root, rock, obstacle depths). On the other hand, FSCC strongly recommends to record and store all soil forming variables at national level. The information may be derived from a combination of field measurements/observations, climatic records, topographical, geological and geomorphological maps and documents, and partly from the other surveys already conducted on the observation plots.

## 2.1 Climate and weather conditions

For the collection of meteorological data, see Manual IX on Meteorological Measurements.

### 2.1.1 Present weather conditions

The weather condition at the time of the profile description is recorded, using following classes (after BBC weather)

Code	Description
1	sunny
2	partly cloudy
3	overcast
4	light rain

5	heavy rain
6	thunder storm
7	sleet <sup>1)</sup>
8	hail <sup>2)</sup>
9	snow

<sup>1)</sup> Sleet refers to snow that has partially melted when it touches the ground, due to surrounding air that is sufficiently warm to partially melt it while falling, but not warm enough to fully melt droplets into rain. Thus it refers to partially melted droplets, a mixture of snow and rain. It does not tend to form a layer on the ground, unless the ground has a temperature that is below freezing, when it can form a dangerous layer of invisible ice on surfaces known as 'black ice'. This similarly occurs when rain freezes upon contact with the ground (freezing rain) (<http://en.wikipedia.org/>).

<sup>2)</sup> Hail is a type of graupel, a form of precipitation, composed of spears or irregular lumps of ice. It occurs when supercooled water droplets (remaining in a liquid state despite being below the freezing point, 0 °C) in a storm cloud aggregates around some solid object, such as a dust particle or an already-forming hailstone. The water then freezes around the object. Depending on the wind patterns within the cloud, the hailstone may continue to circulate for some time, increasing in size. Eventually, the hailstone falls to the ground, when the updraft is no longer strong enough to support its weight (<http://en.wikipedia.org/>).

### 2.1.2 Former weather conditions (AG-Boden, 2004)

This refers to the weather conditions prior to the time of the profile description.

Code	Description
1	no rain during the last month
2	no rain during the last week
3	no rain during the last 24 hours
4	light rain during the last 24 hours
5	heavy rain or thunder during the last 24 hours
6	extremely rainy or snow melting

## 2.2 Soil climate

### 2.2.1 Cryic horizon

Providing information on the soil climate is optional, except for those soils that may have a cryic horizon<sup>1</sup>. A cryic horizon has following definition with respect to soil temperature:

*A cryic horizon must have a soil  
temperature at or below 0°C for two or  
more years in succession.*

### 2.2.2 Soil climate classification (FAO, 2006)

The soil climate classification can be indicated, if applicable. The soil moisture and temperature regimes according to the USDA Keys to Soil Taxonomy (Soil Survey Staff, 2003) may be used. Where such information is not available or cannot be derived from representative climatic data with confidence, it is better to leave the space blank. Other agro-climatic variables worth mentioning would be a local climate class, the agro-climatic zone and length of growing period.

---

<sup>1</sup> Note that the temperature requirement for a cryic diagnostic horizon is different than for a cryic soil temperature regime. Whereas the temperature for the cryic soil temperature regime is measured at 50 cm depth, the cryic horizon may be present in all depths within the upper 100 cm to qualify for Cryosols, or within the upper 200 cm for other reference soil groups.

Soil Temperature Regime				Soil Moisture Regime			
Code	Description	Code	Description	Code	Description	Code	Description
PG	Pergelic			AQ	Aquic	PQ	Peraquic
CR	Cryic			AR	Aridic		
FR	Frigid	IF	Isofrigid	TO	Torric		
ME	Mesic	IM	Isomesic	UD	Udic	PU	Perudic
TH	Thermic	IT	Isothermic	US	Ustic		
HT	Hyperthermic	IH	Isohyperthermic	XE	Xeric		

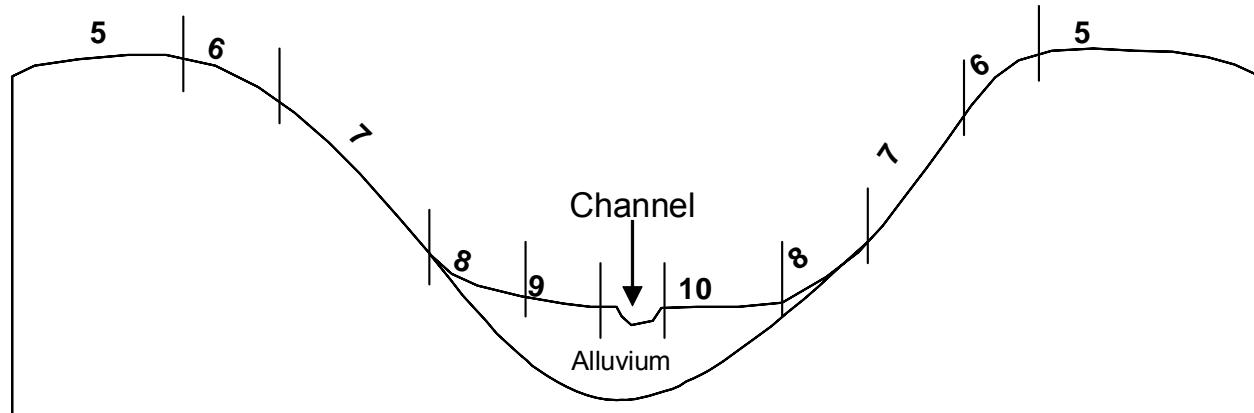
## 2.3 Topography

### 2.3.1 Slope position

(after FAO, 2006)

The position of the soil profile with respect to the slope is very important. Not only will the slope position have an influence on the external and internal drainage, but also the runoff and the subsurface flow are affected. A separate terminology is used for flat or almost flat (slopes of <10%) and undulating to mountainous terrains (slopes >10%) (Figure 1):

Position in flat or almost flat terrain		Position in undulating to mountainous terrain	
Code	Description	Code	Description
1	Higher part (rise)	5	Crest (summit)
2	Intermediate part	6	Upper slope (shoulder)
3	Lower part (and dip)	7	Middle slope
4	Bottom (drainage line)	8	Lower slope (foot slope)
		9	Toe slope
		10	Bottom (flat)



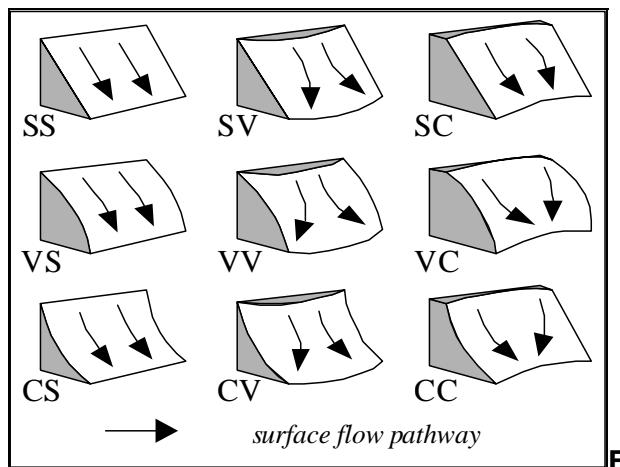
**Figure 1:** Slope positions in undulating and mountainous terrain (After Ruhe, 1975; Schoeneberger et al., 2002; FAO, 2006)

### 2.3.2 Slope form

(after Schoeneberger et al., 2002; FAO, 2006)

The slope form is described in two directions: up and down slope, which is perpendicular to the contour, and across slope, which is along the horizontal contour. The slope form classes defined are provided in Figure 2:

<b>Code</b>		<b>Description</b>
1	SS	Straight, straight
2	SV	Straight, convex
3	SC	Straight, concave
4	VS	Convex, straight
5	VV	Convex, convex
6	VC	Convex, concave
7	CS	Concave, straight
8	CV	Concave, convex
9	CC	Concave, concave
10		Terraced
11		Complex (irregular)



**Figure 2:** Slope forms and surface drainage pathways (after Schoeneberger et al., 2002)

### 2.3.3 Slope gradient

(Modified from FAO, 2006)

The slope gradient in the immediate surrounding of the soil profile should be measured using a clinometer, an abney level or a similar instrument. If measurements in the field are not possible, then the gradient can be calculated from the contour lines on detailed topographical maps.

In practice, measuring slopes in forest can be problematic especially when the slope gradient is very gentle because then a longer distance is necessary to make an accurate measurement. If so, it is advised to base very gentle gradients (<2%) on topographical map readings.

If the slope gradient is measured in degrees, then the gradient is calculated into percent, knowing that 45° equals 100%. Values above 100% are possible (often such steep slopes are indeed left for forests).

<b>Code</b>	<b>Description</b>	<b>Class limits</b>
1	Flat	0 – 0.2 %
2	Level	0.2 – 0.5 %
3	Nearly level	0.5 – 1.0 %
4	Very gently sloping	1 - 2 %
5	Gently sloping	2 - 5 %
6	Sloping	5 - 10 %
7	Strongly sloping	10 - 15 %
8	Moderately steep	15 - 30 %
9	Steep	30 - 60 %
10	Very steep	60 - 100 %
11	Extremely steep	>100 %

### 2.3.4 Slope length (FAO, 2006)

In addition to the above attributes of slope, the slope length along the slope (particularly above the site) is recorded in meters.

### 2.3.5 Slope orientation

The slope orientation (azimuth) of the plot is mandatory to report in the general plot file at the time of the installation of the monitoring plot (XXGENER.PLT file) according to classes. See Part II of this Manual.

## 2.4 Land use

(to be reported in \*.PRF file)

Land use applies to the current use of the land. Land use greatly influences the direction and rate of soil formation; its recording greatly enhances the interpretative value of the soil data. The land-use should be described according to following list:

<b>Code</b>	<b>Description</b>
50	Natural forest and woodland (mostly natural regeneration)
51	Natural forest and woodland without felling
52	Natural forest and woodland with selective felling
53	Natural forest and woodland with clear felling
60	Plantation forestry (mostly planted)
61	Plantation forestry without felling
62	Plantation forestry with selective felling
63	Plantation forestry with clear felling
70	Agro-forestry
80	Nature protection
90	Other (explain)

Further information:

Is hunting allowed? (Y/N/X; where X stands for no information)

Is the wild life protected including density control? (Y/N/X; where X stands for no information)

Is grazing by domesticated animals (e.g. cattle, pigs...) practised, or not? (Y/N/X; where X stands for no information)

## 2.5 Human influence

(modified from FAO, 2006)

Any evidence of human activity, which is likely to have affected the landscape or the physical and chemical properties of the soil should be recorded (erosion is dealt with separately, see paragraph 2.16). Below are the most common examples of human influence listed. Observations of human impact on the soil profile is reported in chapter 3.20 (Man Made Materials) and 3.21 (Human Transported materials). Here only observations observed in the landscape on meso and micro scale are recorded.

<b>Code</b>	<b>Description</b>	<b>Code</b>	<b>Description</b>
1	No influence	15	Raised beds
2	Vegetation disturbed (not specified)	16	Terracing
3	Vegetation slightly disturbed	17	Land fill
4	Vegetation moderately disturbed	18	Levelling
5	Vegetation strongly disturbed	19	Artificial drainage
6	Mineral additions (not specified)	20	Irrigation (not specified)
7	Sand additions	21	Clearing
8	Organic additions (not specified)	22	Burning
9	Ploughing (not specified)	23	Surface compaction
10	Shallow ploughing (<20 cm)	24	Traffic traces
11	Ploughing (20-40 cm)	25	Application of fertilizers
12	Deep ploughing (>40 cm)	26	Pollution
13	Spitting (traces of spade marks)	30	Others (explain)
14	Plaggen		

## 2.6 Vegetation

### 2.6.1 Forest type classification

The forest type is described, according the European forest type classification, which was validated in the BioSoil biodiversity project of the majority of the Level I plots (EEA, 2007). On Level I, Level II and the Level II core plots, the forest type should be described in the general plot file (see Manual Part II).

### 2.6.2 Tree species composition

The main tree species together with the type of tree species mixture (single tree wise mixture, group wise mixture, mixture by layers, etc.) is to be reported in the general plot file at the installation of the monitoring plot. See Manual Part II. The social class is mandatory to report on Level II for crown condition assessment.

## 2.7 Parent material

(originating from SG-DBEM, 2003) (**to be reported in \*.PRF file**)

The parent material is the material from which the soil has been derived. There are two groups of parent material: either un lithified materials (mostly sediments), or weathering materials overlying the hard rock from which they originate. There are also restored natural soil materials or sediments as well as man-made (technogenic) materials.

The detailed table on parent material applied by the Soil Geographical Data Base (SG-DBEM, 2003) is presented in the explanatory items. The parent material should be described at least on the major class level. The 9 major classes summarised below are not listed hierarchically:

<b>Code</b>	<b>Description of Major Class level</b>
0000	No information
1000	Consolidated-clastic-sedimentary rocks
2000	Other sedimentary rocks (chemically precipitated, evaporated, or organogenic or biogenic in origin)
3000	Igneous rocks
4000	Metamorphic rocks
5000	Unconsolidated deposits (alluvium, weathering residuum and slope deposits)
6000	Unconsolidated glacial deposits / glacial drift
7000	Aeolian deposits
8000	Organic materials
9000	Anthropogenic deposits

## 2.8 Natural Drainage Classes

(Soil Survey Staff, 1993)

Soil drainage is usually reflected by soil colour, but relict features may persist after natural or artificial changes in drainage. The depth of occurrence and intensity of gley features usually indicate the drainage status of the soil but not always: some soil materials will not develop strong features of gleaming because of their specific chemical composition, texture, structure or porosity, other mottles may be the results of weathering minerals, rather than an impact of drainage conditions.

<b>Code</b>	<b>Class name</b>	<b>Description</b>
1	Excessively drained	Water is removed from the soil very rapidly. Internal free ("gravitational") water commonly is very rare or very deep. The soils are commonly coarse-textured, and have very high saturated hydraulic conductivity, or are very shallow.
2	Somewhat excessively drained	Water is removed from soil rapidly. Internal free water occurrence commonly is very rare or very deep. The soils are commonly coarse-textured, and have high saturated hydraulic conductivity or are very shallow.
3	Well drained	Water is removed from the soil readily, but not rapidly. Internal free water occurrence commonly is deep or very deep; annual duration is not specified. Wetness does not inhibit growth of roots for significant periods during most growing seasons. The soils are mainly free of the deep to redoximorphic features that are related to wetness.
4	Moderately well drained	Water is removed from the soil somewhat slowly during some periods of the year. Internal free water commonly is moderately deep and may be transitory or permanent. The soil is wet for only a short time within the rooting depth during the growing season, but long enough that most mesophytic crops are affected. The soil commonly has a moderately low or lower saturated hydraulic conductivity within 1 m of the surface, or periodically receives high rainfall, or both.
5	Somewhat poorly drained	Water is removed slowly so that the soil is wet at a shallow depth for significant periods during the growing season. Internal free water is commonly shallow to moderately deep and transitory to permanent. Unless the soil is artificially drained, the growth of most mesophytic plants is markedly restricted. The soil commonly has a low or very low saturated hydraulic conductivity or a high water table, or receives water from lateral flow, or persistent rainfall, or some combination of these factors.

6	Poorly drained	Water is removed so slowly that the soil is wet at shallow depths periodically during the growing season or remains wet for long periods. Internal free water is shallow to very shallow and common or persistent. Unless the soil is artificially drained, most mesophytic crops cannot be grown, unless the soil is artificially drained. The soil, however, is not continuously wet directly below ploughing depth ( $\pm 25$ cm). Free water at shallow depth is usually present. The water table is commonly the result of low or very low saturated hydraulic conductivity or persistent rainfall, or a combination of both factors.
7	Very poorly drained	Water is removed from the soil so slowly that free water remains at or near the soil surface during much of the growing season. Internal free water is very shallow and persistent or permanent. Unless the soil is artificially drained, most mesophytic crops cannot be grown. The soils are commonly level or depressed and frequently ponded. If rainfall is persistent or high, the soil can be very poorly drained even on gentle slopes.
X	Not known	

## 2.9 Water availability

(to be reported in \*.PLS file)

An estimate of the water availability to principal tree species during the growing season is made:

Code	Description
1	Insufficient
2	Sufficient
3	Excessive

## 2.10 Period of water saturation

(FAO, 1990)

In the description of drainage classes, the period when the soil is saturated or very wet is not satisfactory, especially where the rainfall is strongly seasonal or irregular. Very permeable sands may be permanently or seasonally waterlogged and impermeable clays may never be saturated or only for a few days a year.

The period during which the soil near the surface is saturated should be indicated, based on local information or judgment supplemented by gleying features in the profile:

Code	Description
1	Never saturated
2	Rarely saturated (a few days in some years)
3	Saturated for short periods in most years (up to 30 days)
4	Saturated for long periods every year
5	Always saturated

## 2.11 External drainage

(FAO, 1990)

The external drainage of a site refers to its relative position in the landscape. Is the site in a landscape position where it will overall receive water from upslope or rather shed water downslope? And if shedding, by which speed is the water lost? The following classes are defined:

Code	Description
1	Ponded (run-on site)
2	Neither receiving nor shedding water
3	Slow run-off
4	Moderately rapid run-off
5	Rapid run-off

## 2.12 Flooding

(FAO, 1990)

Flooding is described according to its frequency, duration and depth. At most sites it is difficult to assess flooding accurately. Information may be obtained from records of past flooding or from local enquiry. The frequency and duration classes give an indication of the average occurrence of flooding. It is very important to evaluate if the flooding is a relict or if it is still active at present.

### 2.12.1 Frequency

Code	Description
1	Daily
2	Weekly
3	Monthly
4	Annually
5	Biennially
6	Once every 2-4 years
7	Once every 5-10 years
8	Rare (less than once in 10 years)
9	Inactive today, but has been active in historical time
10	Inactive today, but has once been active (ancient time)
11	None

### 2.12.2 Duration (days/year)

Code	Description
1	Less than 1 day
2	1- 15 days
3	15- 30 days
4	30- 90 days
5	90 -180 days
6	180- 360 days

### 2.12.3 Depth (of standing water)

Code	Description	Class limits
1	Very shallow	0 - 25 cm
2	Shallow	25 - 50 cm
3	Moderately deep	50 - 100 cm
4	Deep	100 - 150 cm
5	Very deep	> 150 cm

## 2.13 Groundwater table

(Modified from SG-DBEM, 2003)

The groundwater table level within or below a soil profile often varies in time. Therefore, the mean highest and mean lowest permanent or perched groundwater table level should be the average for at least the past 10 years. This information can in some cases be derived from maps. Seasonal variations are not recorded. The different groundwater table classes to be used are as follows:

### 2.13.1 Mean highest and mean lowest groundwater table

(to be reported in \*.PRF file)

The mean highest and mean lowest permanent or perched groundwater table level is the average level for at least the past 10 years. Generally this information is lacking and so these values need generally to be estimated by an expert.

The depth classes for groundwater for mean lowest and mean highest depth levels are:

Code	Description	Class limits
9	No water table observed or unknown	
1	Very shallow to shallow	0 - 50 cm
2	Moderately deep	50 - 100 cm
3	Deep	100 - 150 cm
4	Very deep	150 - 200 cm
5	Extremely deep	>200 cm

### 2.13.2 Type of water table

(to be reported in \*.PRF file)

Code	Description
9	No water table
1	Perched water table (= stagnating water table)
2	Permanent water table (= groundwater table)

## 2.14 Rock outcrops

(after FAO, 2006)

Rock outcrops should be described in terms of percentage surface cover, size and spacing of the individual outcrops. It is necessary to walk around in increasing larger circles around the profile

plot in order to make a correct estimate. The classes of percentage of surface cover and of average distance between rock outcrops (single or clusters) are as follows:

#### 2.14.1 Surface cover

Code	Description	Class limits
1	None	0 %
2	Very few	0 - 2 %
3	Few	2 - 5 %
4	Common	5 - 15 %
5	Many	15 - 40 %
6	Abundant	40 - 80 %
7	Dominant	>80 %
8	Rock outcrops are present but mostly hidden below forest litter	

#### 2.14.2 Distance between rock outcrops

Code	Description
1	>50 m
2	20 - 50 m
3	5 - 20 m
4	2 - 5 m
5	<2 m

### 2.15 Coarse surface fragments

(FAO, 2006)

Coarse surface fragments, boulders and stones, including those that are partly buried, should be described in terms of percentage of surface cover and size of the fragments. It is often not easy to observe boulders and stones under forest due to the litter layer, so a careful survey may be necessary. Remember, a stone or boulder partly buried is only included in the coverage and class estimate based on the visible part, it is not the purpose to uncover partly or completely buried coarse fragments. The classes of coverage and size handled are:

#### 2.15.1 Surface cover

Code	Description	Class limits
9	None	0 %
1	Very few	0 - 2 %
2	Few	2 - 5 %
3	Common	5 - 15 %
4	Many	15 - 40 %
5	Abundant	40 - 80 %
6	Dominant	>80 %

### 2.15.2 Size classes

Size classes according to the greatest dimension of the individual gravels/stones:

Code	Description	Class limits
1	Fine gravel	0.2 - 0.6 cm
2	Medium gravel	0.6 - 2.0 cm
3	Coarse gravel	2 - 6 cm
4	Stones	6 - 20 cm
5	Boulders	20 - 60 cm
6	Large boulders	60 - 200 cm

## 2.16 Erosion & sedimentation

(modified from FAO, 2006)

Although under most forest covers erosion and sedimentation will be of minor importance, these variables have been included in these guidelines. Since, when it is present, it is of great importance for the soil formation and dynamics.

### 2.16.1 Type of erosion/sedimentation

Erosion and sedimentation can be described according to the agency - water, wind, mass movements (landslides and related phenomena). In forested sites the major or only erosion may occur along patches, roads, timber tracks etc. Description should also include deposition of transported material:

Code	Description
9	No evidence of erosion
1	Water erosion and sedimentation
2	Sheet erosion by water
3	Rill erosion by water
4	Gully erosion by water
5	Tunnel erosion by water
6	Mass movement (landslides and similar phenomena)
7	Sedimentation by water
8	Wind erosion and sedimentation
9	Sedimentation by wind
10	Shifting sands
11	Salt deposition
12	Other erosion/sedimentation, related to human

### 2.16.2 Area affected

The proportion of total area affected by erosion/sedimentation is estimated:

Code	Description
9	0 %
1	0 - 5 %
2	5 - 10 %
3	10 - 25 %
4	25 - 50 %
5	> 50 %

### 2.16.3 Degree

It is difficult to define classes of the degree of erosion which are equally appropriate for all soils, environments, and the various types of erosion. Classes may have to be defined further for each type or combination of erosion and sedimentation and each specific environment. For example, in the case of gully and rill erosion, the depth and spacing may be recorded; for sheet erosion the loss of topsoil; for dunes the height; for sedimentation the thickness of the layer. The following classes are recommended to describe the degree of erosion:

Code	Class name	Description
9	None	No erosion nor sedimentation
1	Heavy sedimentation	Soils buried below >50 cm of accumulated sediment
2	Considerable sedimentation	Soils buried below 5 - 50 cm of accumulated sediment
3	Noticeable sedimentation	Soils buried below <5 cm of accumulated sediment, continuously distributed
4	Traces sedimentation	Soils buried below <5 cm of accumulated sediment, discontinuously distributed
5	Slight erosion	Some evidence of damage to the topsoil; original biotic functions largely intact
6	Moderate erosion	Removal of topsoil; original biotic functions partly destroyed
7	Severe erosion	Surface layers completely removed and subsurface layers exposed
8	Extreme erosion	Substantial removal of deeper subsurface horizons (badlands)

### 2.16.4 Activity

The period of activity of accelerated erosion, or sedimentation, may be described as follows:

Code	Description
9	Accelerated and natural erosion not observed
1	Period of activity not known
2	Active in historical times
3	Active in recent past (up till past 50 - 100 years)
4	Active at present

## 2.17 Surface sealing

(FAO, 2006)

Where the mineral soil is exposed, a surface crust may develop. Only in extreme cases a surface sealing will develop on forest soils, as the soil should be exposed to wetting and drying and not be protected by a litter layer. If surface crusts develop it will have a negative effect on seed germination, reduce water infiltration and increase run-off. The thickness and the consistence of the crust is described (for consistence definitions see paragraph 3.11):

### 2.17.1 Thickness

Code	Description	Class limits
9	None	
1	Thin	<2 mm
2	Medium	2 - 5 mm
3	Thick	5 - 20 mm
4	Very thick	>20 mm

### 2.17.2 Consistence when dry

Code	Description
1	Slightly hard
2	Hard
3	Very hard
4	Extremely hard

### 2.18 Surface cracks

(FAO, 2006)

Mineral surface cracks develop in many clay-rich soils during drying. The width (average, or average width and maximum width) of the cracks, and the average spacing between cracks are measured.

### 2.18.1 Size (Width)

Code	Description	Class limits
1	Fine	<1 cm
2	Medium	1 - 2 cm
3	Wide	2 - 5 cm
4	Very wide	5 - 10 cm
5	Extremely wide	>10 cm

### 2.18.2 Distance between cracks

Code	Description	Class limits
1	Very closely spaced	<0.2 m
2	Closely spaced	0.2 - 0.5 m
3	Moderately widely spaced	0.5 - 2 m
4	Widely spaced	2 - 5 m
5	Very widely spaced	>5 m

## 2.19 Salt

(FAO, 2006)

The occurrence of surface salt may be described in terms of cover and appearance. Classes for the percentage of surface cover and thickness are:

### 2.19.1 Cover

Code	Description	Class limits
9	None	0 - 2 %
1	Low	2 - 15 %
2	Moderate	15 - 40 %
3	High	40 - 80 %
4	Dominant	>80 %

### 2.19.2 Thickness

Code	Description	Class limits
1	Thin	<2 mm
2	Medium	2 - 5 mm
3	Thick	5 - 20 mm
4	Very thick	>20 mm

## 2.20 Profile depth

(to be reported in \*.PRF file)

The depth of the soil is defined with 3 attributes: effective rooting depth, rock depth and obstacle depth. The depth must be given in cm from the top of the mineral soil surface.

The 'Effective rooting depth' is defined as the depth of the soil at which root growth is strongly inhibited. As rooting depth is plant specific, it is recommended that representative species are used to indicate the effective rooting depth of the soil. The effective rooting depth is governed by such factors as the presence of cemented, toxic or compacted layers, hard rock, or indurated gravel layers. A high permanent water table may also control the rooting depth, but may change after drainage. The effective hydrological depth may be much greater. Apart from obvious situations such as the presence of hard rock, it is realized that the estimation of effective soil depth is subject to individual interpretation.

The depth to the underlying bedrock should be recorded under 'Rock depth of the soil profile'.

The field 'Obstacle depth of the soil profile' can be used to record the depth to any other limiting horizon, such as a petrocalcic horizon, fragipan, duripan, waterlogging, accumulation of toxic elements.

### **3. Soil horizon description**

In the following chapter, the horizon variables are presented. The sequence is different from those presented in the FAO guidelines (FAO 1990; FAO 2006).

Usually a forest soil is composed of mineral and organic horizons, stones, bedrock etc., which together constitute the soil profile. In the following chapter a series of variables are listed, but not all of them are equally relevant to particular organic horizons. For the definitions of organic material and organic horizons, see Annex 7.

In principle knowledge of the content of organic matter is required to differentiate between organic and mineral materials. In the field, organic horizons are usually easy to recognise, only border cases will need analytical data to check for the content of organic carbon. In the table below the subchapters relevant for organic and mineral horizons are summarised:

<b>Relevant for:</b>	<b>organic horizons</b>	<b>mineral horizons</b>
3.1 Horizon boundary	Yes	Yes
3.2 Photographic recordings	Yes	Yes
3.3 Soil Colour	Yes	Yes
3.4 Mottling	No	Yes
3.5 Redoximorphic properties	Yes	Yes
3.6 Texture	No	Yes
3.7 Rock fragments	Yes	Yes
3.8 Andic material	No	Yes
3.9 Soil structure	No	Yes
3.10 Consistence	No	Yes
3.11 Cutanic features	No	Yes
3.12 Porosity	Yes	Yes
3.13 Cementation and compaction	No	Yes
3.14 Nodules	No	Yes
3.15 Roots	Yes	Yes
3.16 Other biological features	Yes	Yes
3.17 Carbonates	Yes	Yes
3.18 Gypsum	No	Yes
3.19 Readily soluble salts	Yes	Yes
3.20 Man made materials	Yes	Yes
3.21 Human transported material	No	Yes
3.22 Soil horizon designation	Yes	Yes

After having selected the most representative location for the soil profile (see also Annex 3), the profile is opened and cleaned. A list of suggested field equipment is presented in Annex 4. The recommended sequence of description is as follows:

delineation and description of the horizon boundaries,  
photographic recordings of the soil in general and of special features in detail,  
colour measurements (see also Annex 5),  
from this stage on, the profile wall is gently broken apart to record texture, rock fragments, structure, consistence, porosity, cutans, cementations and nodules, this is followed by  
the description of roots and other biological activity, and by  
the description of carbonates, gypsum and salts,  
each horizon is designated one or more horizon master and subordinate symbols, and  
the necessary samples are collected.

### **3.1 Horizon boundary (modified from FAO, 2006)**

The nature of the boundaries between soil layers, or horizons, may indicate the processes that have formed the soil. In some cases, they reflect anthropogenic impacts. Horizon boundaries are described in terms of depth, distinctness and topography.

#### **3.1.1 Horizon number**

(to be reported in \*.PFH file)

After delineation of the horizon boundaries, each horizon is numbered: 1, 2, 3 etc. While the horizon symbols may change according to new information, the horizon number is not to be changed at any point of the further profile description and sampling. The numbering starts from the interface between air and soil no matter whether the surface horizon is an organic or a mineral horizon (see Figure 3). If at a later stage, a new horizon is discovered or an existing horizon is subdivided, a new number should be created. Avoid renumbering of the existing horizons to keep the link with the initial description in the field.

#### **3.1.2 Depth of horizon limits**

(to be reported in \*.PFH file)

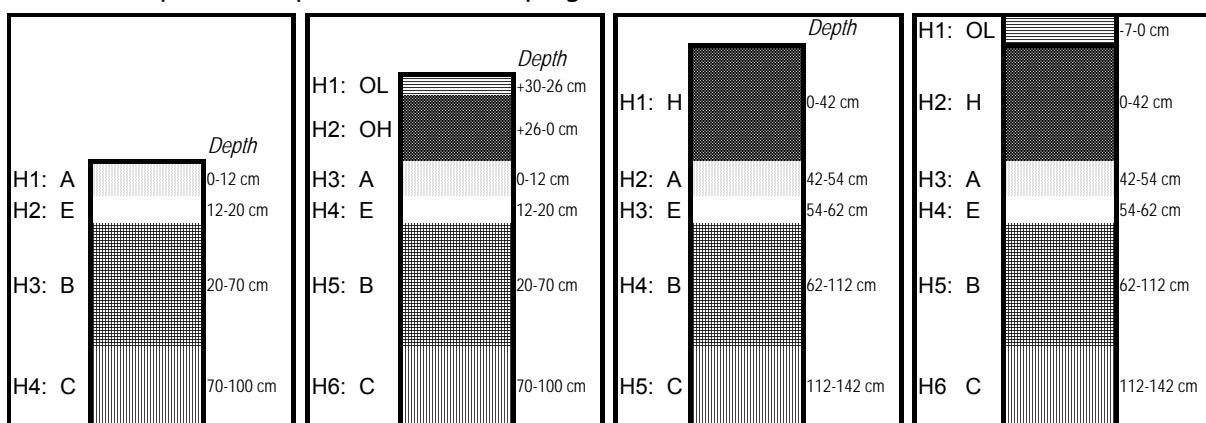
The depth of the upper and lower boundary of each horizon is measured in centimetres from the surface of the mineral soil.

If the soil is covered by (an) organic layer(s), either:

- a. 10 cm or more thick from the soil surface to a lithic or paralithic contact, or
- b. 40 cm or more thick,

then the depth is measured from the surface of the organic cover. The depth requirements correspond to the limit for Histosols (organic soils).

**Figure 3:** Examples on how the horizon depth should be recorded in the field. These depths are important for the profile description and for the sampling.



If the organic layer(s) is (are) too shallow to fulfil the above depth requirement(s), then its depth is recorded from the zero-point and upwards (see Figure 3), using negative depths.

The depth is measured perpendicular to the slope.

Most soil boundaries are zones of transition rather than sharp breaks. The distinctness together with the topography describe the transition between the different horizons and substitute for the need to describe the depth ranges as for instance from 28 (25-31) cm to 45 (39-51) cm.

### 3.1.3 Distinctness

(to be reported in \*.PFH file)

The distinctness of the lower horizon boundary refers to the thickness of the boundary zone in between adjacent horizons.

Code	Description	Class limits
3	Abrupt	0 - 2 cm
4	Clear	2 - 5 cm
5	Gradual	5 - 15 cm
6	Diffuse	>15 cm

Concerning the boundary between the organic (as in forest floors) and the organo-mineral horizons, following classes of distinctness shall be used:

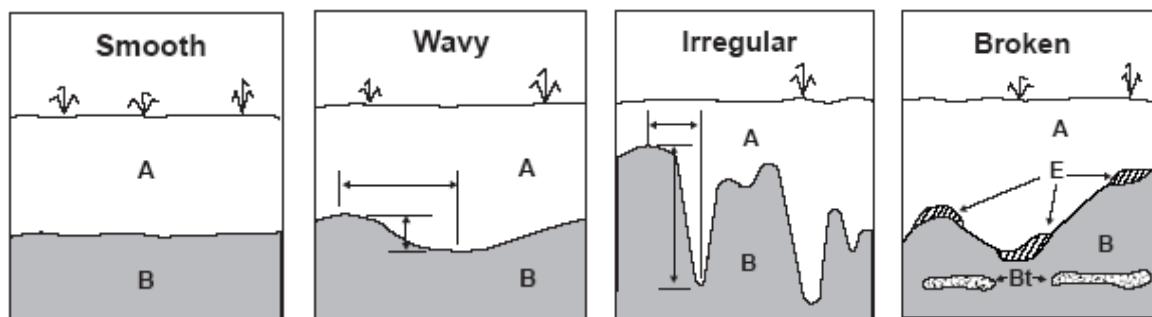
Code	Distinctness	Class limits
7	very sharp	< 0.3 cm
8	sharp	≥ 0.3 and < 0.5 cm
9	not sharp	≥ 0.5 cm

### 3.1.4 Topography

(to be reported in \*.PFH file)

The topography of the boundary indicates its shape (see Figure 4).

Code	Class name	Description
1	Smooth	Nearly plane surface
2	Wavy	Pockets shallower than they are wide
3	Irregular	Pockets deeper than they are wide
4	Broken	Discontinuous
5	Complex	



**Figure 4:** Illustration of the most common horizon topographies, which is the lateral undulation and continuity of the boundary between horizons (after Schoeneberger et al., 2002)

### 3.2 Photographic recordings

Quality photographs are essential for the soil database. A scale is needed on all photos, preferentially a bicoloured centimetre-scale. The use of tools for scaling should be avoided. If a tool e.g. a spade is used anyway the length of the spade should be clearly stated in the photo legend.

Partly shade partly sunshine on the profile wall should be avoided. You may use a dark and uniform coloured umbrella to shade the profile. If possible avoid the use of a camera flash by using a tripod or a monopod. If an analogue camera is used, try always to use the same brand of film rolls. If using a digital camera, use a high resolution (5 Mega Pixels or more) and a camera with a good quality lens. The photographic database can/should include following images:

General photo illustrating the geomorphology and vegetation of the area surrounding the profile

Photo of the immediate vicinity of the profile

The profile after cleaning and before indication of the soil horizons on the profile wall.

The profile after the soil horizons are outlined gently on the profile wall with e.g. a knife

The profile with partly visible structure and partly with a cleaned surface

The profile with indications where to sample

Close-up of the organic topsoil horizon(s)

Horizontal sections, e.g. in depths where the bulk density (BD) is sampled

Special features

### 3.3 Soil colour

(to be reported in \*.PFH file)

The colour of the soil matrix in each horizon should be recorded in moist and dry condition using the Munsell notation (e.g. Munsell, 2000). The colour notation is composed of hue, value and chroma. Hue is the dominant spectral colour (red, yellow, green, blue, violet), value is the lightness or darkness of colour ranging from 1 (dark) to 8 (light), and chroma is the purity or strength of colour ranging from 1 (pale) to 8 (bright). If there is no dominant colour, the horizon is described as mottled and two or more colours are given, making use of the observation field. In addition to the colour notations, the standard Munsell colour names should be recorded as well.

*Example:* Greyish brown 10YR 5/2 (moist) and light brownish grey 10YR 6/2 (dry); where 10YR (yellowish red) is the hue, 5 (or 6) is the value and 2 the chroma.

*Example:* Dark greyish brown to greyish brown 2.5Y 4.5/2 (moist) and light brownish grey 2.5Y 6/2 (dry); Notice that interpolation between colours is possible for hue, value and/or chroma

*Example:* Dark greenish grey 5GY 4/1 (moist) and greenish grey 10GY 5/1 (dry); where 5GY or 10GY (greenish yellow) is the hue, 4 (or 5) is the value and 1 is the chroma.

More detailed information about optimal colour measurements and special colours required for soil classification is to be found in Annex 5.

### 3.4 Mottling

(FAO, 2006)

Mottles are spots of different colours interspersed with the dominant colour of the soil. They commonly indicate that the soil has been subject to alternate wet (reducing) and dry (oxidizing) conditions. Other mottles can be the result of rock weathering, clay (+iron) migration and accumulation, selective decay by fungi of organic matter etc.

Mottling is described in terms of abundance, size, contrast, boundary and colour. In addition, the shape, position or any other feature may be recorded.

#### 3.4.1 Colour

Measure the colours using the Munsell Soil Colour Charts.

If the colour changes after exposure to the air, measure both the colours before and after oxidation.

#### 3.4.2 Abundance

Abundance is described as an exact figure or in classes indicating the percentage of the exposed surface occupied by the mottles. When mottles are so abundant that distinction of matrix and mottle colour is not possible, the predominant colours should be described as soil matrix colours.

<b>Code</b>	<b>Description</b>	<b>Class limits</b>
1	None	0 %
2	Very few	0 - 2 %
3	Few	2 - 5 %
4	Common	5 - 15 %
5	Many	15 - 40 %
6	Abundant	>40 %

### 3.4.3 Size

The following classes are used to indicate the approximate diameters of individual mottles.

<b>Code</b>	<b>Description</b>	<b>Class limits</b>
1	Very fine	< 2 mm
2	Fine	2 - 6 mm
3	Medium	6 - 20 mm
4	Coarse	20 - 40 mm
5	Very coarse	40 - 80 mm
6	Extremely coarse	> 80 mm

### 3.4.4 Contrast

The colour contrast between mottles and soil matrix can be described as:

<b>Code</b>	<b>Description</b>
1	Faint: mottles are evident only on close examination. Soil colours in both the matrix and mottles are similar.
2	Distinct: although not striking, the mottles are readily seen. The hue, chroma or value of the matrix is easily distinguished from the mottles. They may vary by as much as 2.5 units of hue or several units in chroma or value.
3	Prominent: the mottles are conspicuous. Hue, chroma and value, alone or in combination, are several units apart.

### 3.4.5 Boundary

The boundary between mottle and matrix is described according to the width of the transition zone.

<b>Code</b>	<b>Description</b>	<b>Class limits</b>
1	Sharp	<0.5 mm
2	Clear	0.5 - 2 mm
3	Diffuse	2 - 5 mm
4	Very diffuse	>5 mm

### 3.5 Redoximorphic properties (IUSS Working Group WRB, 2007)

Redoximorphic features concerns a colour pattern observed in the soil, which is the result of depletion or concentration compared to the matrix colour, formed by oxidation/reduction of iron and/or manganese.

#### 3.5.1 Reducing conditions

If reducing conditions prevail in a soil horizon, it can be tested in following ways:

1. Is the negative logarithm of the hydrogen partial pressure (rH) less than 20?
2. Are  $\text{Fe}^{2+}$  ions present, as tested by spraying the freshly exposed soil surface with a 0.2% (m/v)  $\alpha,\alpha$  dipyridyl solution in 10% (v/v) acetic acid solution. The test yields a striking reddish colour in the presence of  $\text{Fe}^{2+}$  ions (be careful, the chemical is slightly toxic). Did a reddish colour (almost like red wine) appear on the tested soil surface after a few minutes?
3. Is iron sulphide present?
4. Is methane present?

If the answer to any of above 4 questions is yes, report: **Y**

If none of the test above are positive, report: **N**

If data for some reason are missing or impossible to collect, indicate: **X**

#### 3.5.2 Reductimorphic and oximorphic colours

If oximorphic and/or reductimorphic mottles as present they are first of all described according to the chapter on mottles (see chapter 3.4). Note that gleyic mottles should be recorded as fast as possible after the profile has been prepared, sometimes even while digging the profile, due to the fast oxidation of certain minerals.

Are reductimorphic colours, reflecting permanently wet conditions, present on more than 90% of the soil surface? Reductimorphic colours are neutral white to black (Munsell N1/ to N8/) or bluish to greenish (Munsell 2.5Y, 5Y, 5G, 5B).

#### Y/N/X (Yes/No/Not known)

Oximorphic colours reflect alternating reducing and oxidizing conditions, as is the case in the capillary fringe and in the surface horizons with fluctuating groundwater levels. They comprise any colour, excluding reductimorphic colours (see above). Are 5% or more of the soil surface cover by oximorphic coloured mottles?

#### Y/N/X (Yes/No/Not known)

The above described field tests may to some degree illustrate the actual redoximorphic conditions at the moment of fieldwork, rather than the general condition of the soil. For the same reason it is strongly recommended in case of gley soils to give special attention to:

roots (presence/absence), and

the soil water (indications of a fluctuating or permanent water tables etc.)

#### 3.5.3 Stagnic and gleyic colour pattern

Dending on the origin of the water, which is either the groundwater table, either surface water that is (at least temporarily) saturating the soil layer, two different colour patterns will develop. It is important to distinguish between both type of colour patterns during the profile description.

Note: When a stagnic colour pattern is identified in a genetic horizon, it is designated by the horizon subordinate symbol 'g'. When a gleyic colour pattern is seen, the horizon received the subordinate symbol 'l'. See Annex 7. The latter symbol did not yet exist during the BioSoil demonstration project (Mikkelsen et al., 2006) and so is only applied in the ICP Forests programme from 2010 onwards.

## Stagnic colour pattern

### *General description*

Soil materials develop a stagnic colour pattern (from Latin stagnare, to stagnate) if they are, at least temporarily, saturated with surface water (or were saturated in the past, if now drained) for a period long enough that allows reducing conditions to occur (this may range from a few days in the tropics to a few weeks in other areas).

### *Diagnostic criteria*

A stagnic colour pattern shows mottling in such a way that the surfaces of the peds (or parts of the soil matrix) are lighter (at least one Munsell value unit more) and paler (at least one chroma unit less), and the interiors of the peds (or parts of the soil matrix) are more reddish (at least one hue unit) and brighter (at least one chroma unit more) than the non-redoximorphic parts of the layer, or than the mixed average of the interior and surface parts.

### *Additional characteristics*

If a layer has a stagnic colour pattern in 50 percent of its volume the other 50 percent of the layer are non-redoximorphic (neither lighter and paler nor more reddish and brighter).

## Gleyic colour pattern

### *General description*

Soil materials develop a gleyic colour pattern (from Russian gley, mucky soil mass) if they are saturated with groundwater (or were saturated in the past, if now drained) for a period that allows reducing conditions to occur (this may range from a few days in the tropics to a few weeks in other areas).

### *Diagnostic criteria*

A gleyic colour pattern shows one or both of the following:

1. 90 percent or more (exposed area) reductimorphic colours, which comprise neutral white to black (Munsell hue N1/ to N8/) or bluish to greenish (Munsell hue 2.5 Y, 5 Y, 5 G, 5 B); or
2. 5 percent or more (exposed area) mottles of oximorphic colours, which comprise any colour, excluding reductimorphic colours.

### *Field identification*

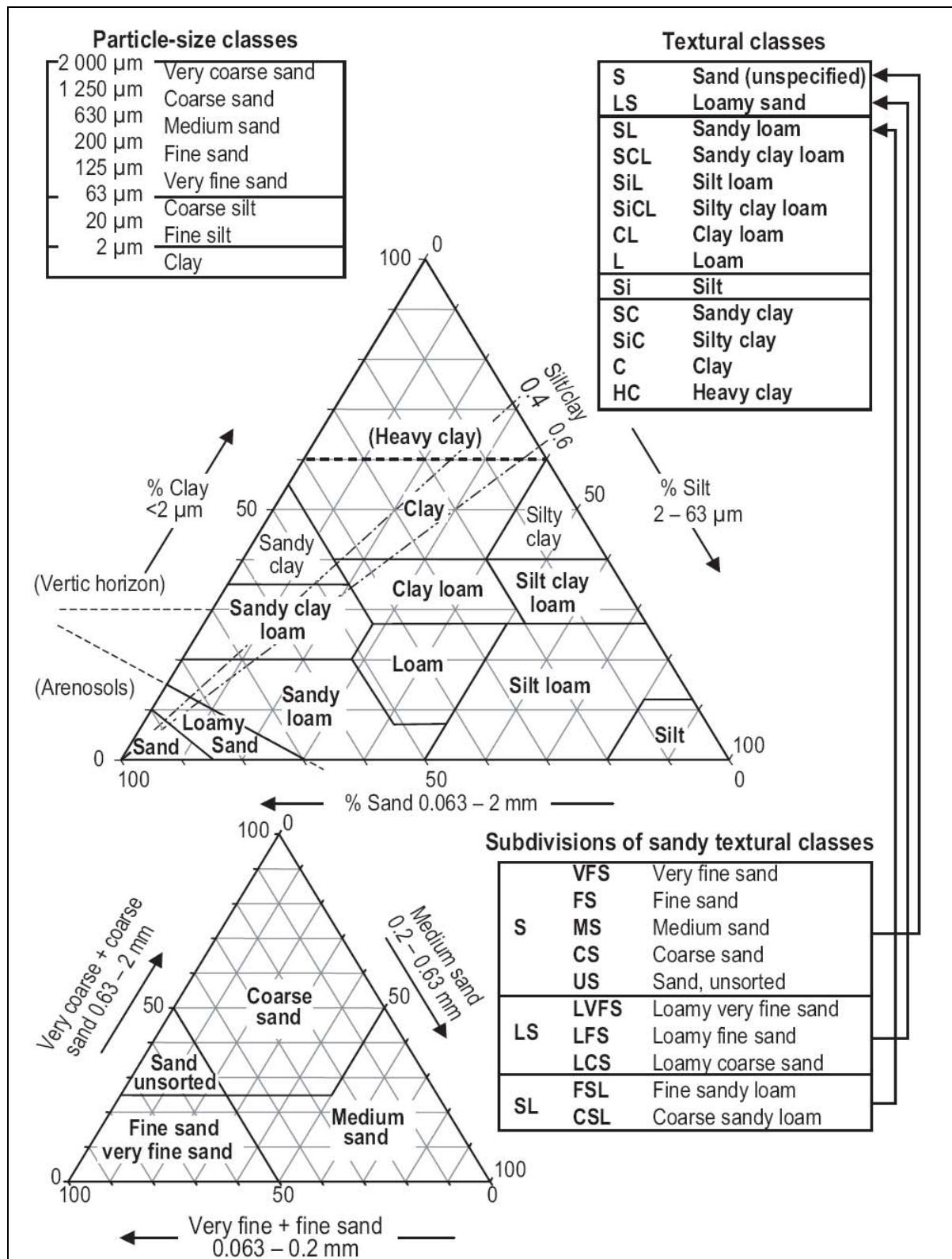
A gleyic colour pattern results from a redox gradient between groundwater and capillary fringe causing an uneven distribution of iron and manganese (hydr)oxides. In the lower part of the soil and/or inside the peds, the oxides are either transformed into insoluble Fe/Mn compounds or they are translocated; both processes lead to the absence of colours with a hue redder than 2.5 Y. Translocated Fe and Mn compounds can be concentrated in the oxidized form (Fe[III], Mn[IV]) on ped surfaces or in biopores (rusty root channels), and towards the surface even in the matrix. Manganese concentrations can be recognized by strong effervescence using a 10-percent H<sub>2</sub>O<sub>2</sub> solution.

### 3.6 Texture of the fine-earth fraction

(FAO, 1990) (to be reported in \*.PFH file)

Soil texture refers to the proportion of the various particle-size classes in a given soil volume and is described as soil textural classes (see Figure 5). The 2000 – 63 – 2 µm system for particle-size fractions is used.

The names of the textural classes, which describe combined particle-size classes, are coded as in Figure 5. With a lot of training field estimates of the texture can be made, a method for finger testing of the textural classes is presented in Annex 6. Otherwise calculating the texture class based on the analytical data from pipette texture is recommended.



**Figure 1:** The fine earth by size and defined in textural classes. Textural classes based on USDA (1951), adopted by FAO (1990) and refined by FAO (2006)

## 3.7 Rock fragments

(modified from FAO, 2006)

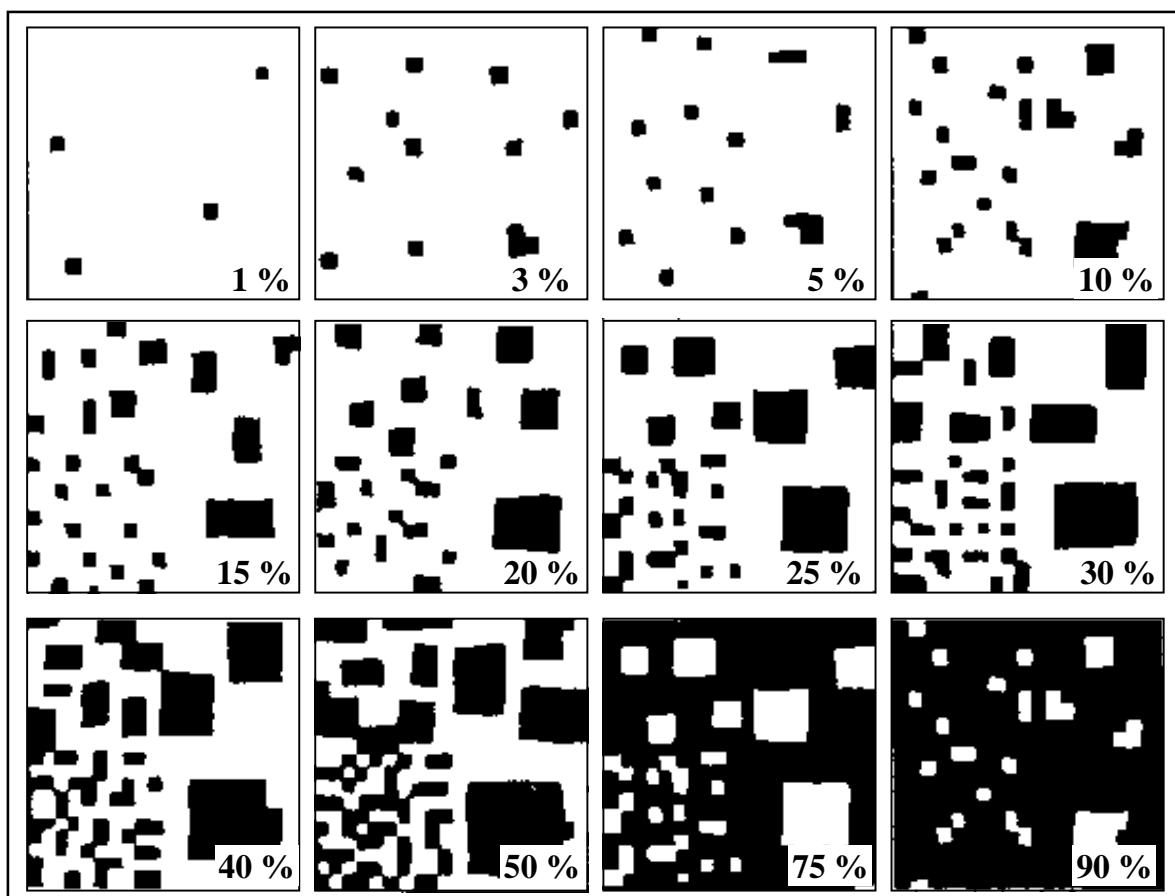
Large fragments (>2 mm) are described according to abundance, size, shape, state of weathering and nature of the fragments. The abundance classes correspond with those for surface coarse fragments and mineral nodules.

### 3.7.1 Abundance

(to be reported in \*.PFH file)

The abundance of rock fragments is estimated (Figure 6) and expressed as a percent (by volume) of the total soil. By preference, the exact figure is provided rather than abundance classes.

<b>Code</b>	<b>Description (FAO, 2006)</b>	<b>Class limits (volume%)</b>	<b>Description SGDBE (Lambert et al. 2003)</b>
9	None	0 %	No stones or gravel
1	Very few to few	0 - 5 %	Very few
2	Common	5 - 15 %	Few
3	Many	15 - 40 %	Frequent or many
4	Abundant	40 - 80 %	Very frequent, very many
5	Dominant	>80 %	Dominant or skeletal



**Figure 6:** Charts for estimating proportions of coarse fragments, mottles or other elements

### 3.7.2 Size of rock fragments and artefacts

Code	Description	Class limits
1	Fine gravel	0.2 - 0.6 cm
2	Medium gravel	0.6 - 2 cm
3	Coarse gravel	2 - 6 cm
4	Stones	6 - 20 cm
5	Boulders	20 - 60 cm
6	Large boulders	60 - 200 cm

### 3.7.3 Dominant shape of rock fragments

The shape may be described as:

Code	Description
1	Flat
2	Angular
3	Sub-rounded
4	Rounded

### 3.7.4 State of weathering of rock fragments

The state of weathering is described as:

Code	Description
9	Fresh or slightly weathered: fragments show little or no signs of weathering
1	Weathered: partial weathering is indicated by discolouration and loss of crystal form in the outer parts of the fragments while the centres remain relatively fresh; fragments have lost little of their original strength.
2	Strongly weathered: all but the most resistant minerals are strongly discoloured and altered throughout; the fragments tend to disintegrate under hand pressure.

### 3.7.5 Nature (type) of rock fragments

The nature of rock fragments is described by the same terminology as for the parent material.

## 3.8 Andic material

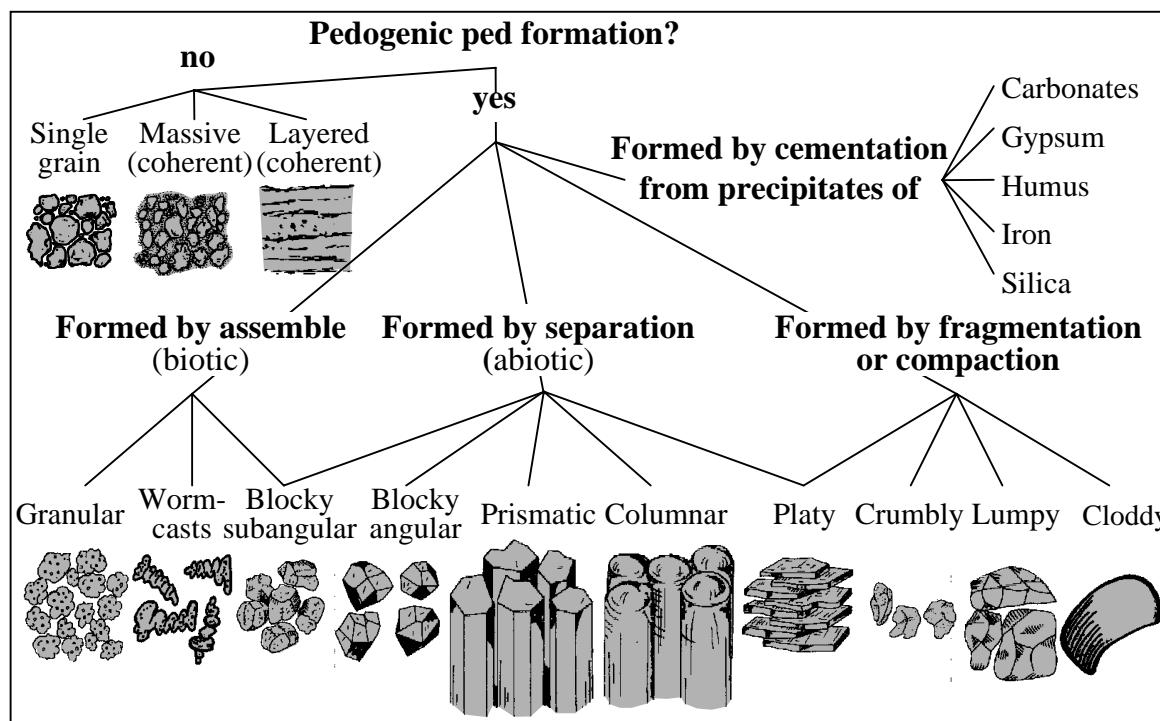
(modified from FAO, 2006)

Soils formed from young volcanic materials often have andic properties: a bulk density of 900 kg/m<sup>3</sup> or less, and <10 % clay with a smearable consistency (caused by allophane and/or ferrihydrite). Surface horizons with andic material are normally black because of the combination of allophane with humic material, or humic material immobilized by aluminium. Andic material may exhibit thixotropy, changing under pressure, or by rubbing, from a plastic solid into a liquefied stage and back into the solid condition.

Is andic material present? Yes/No/Not known (reported as: Y/N/X)

## 3.9 Soil structure (modified from FAO, 2006)

Soil structure relates to the grouping or arrangement of soil particles into discrete soil units (peds). The aggregates are separated from each other by pores or voids and are characterised primarily on basis of their dominant shape: spheroidal (granular, crumb), platy, prism (columnar-top of the prisms are rounded and prismatic- top of the prisms are level) and blocky (angular blocky and subangular blocky).



**Figure 7:** Soil structure types and their formation (FAO, 2006)

With decreasing soil humidity, the soil structure becomes increasingly pronounced. In moist or wet conditions, if no clear structure is visible, a large lump of undisturbed soil material can be dried, which will possibly reveal a specific structure. Another method is to take a large lump of soil on the spade and let it fall from about a meter height, and then to observe how the block of soil breaks into pieces. A third possibility is to use a knife to gently loosen the soil material on the profile wall. Try to loosen the soil in such a way that it breaks along the natural ped faces rather than breaking through the peds (it demands a bit of practice).

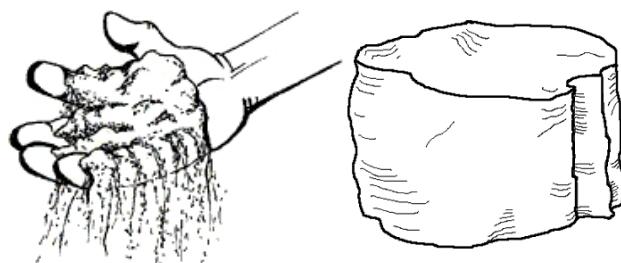
Besides the structure type, also grade and size of aggregates are recorded. When a soil horizon contains aggregates of more than one grade, size or type, the different kinds of aggregates should be described separately and their relationship indicated.

### 3.9.1 Type

(to be reported in \*.PFH file)

The soil can be structureless or shows some kind of structure. If a structure is present the degree of development and the size characteristics to record.

In structureless soil, no aggregates are observable in place and there is no definite arrangement of natural surfaces of weakness. Structureless soils are subdivided into single grain and massive (see Figure 7).

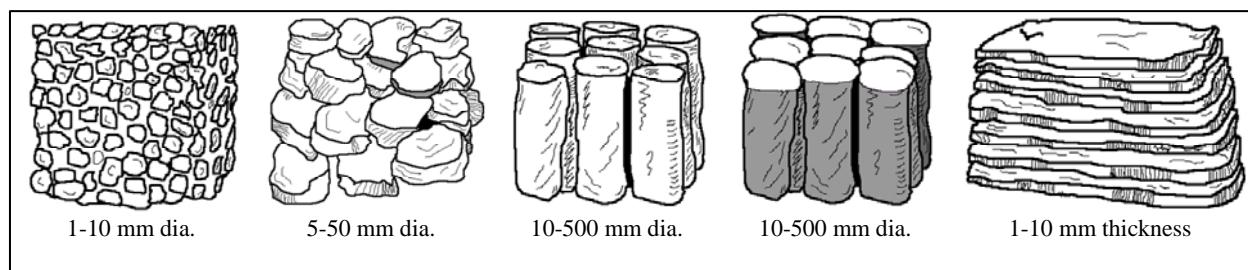


**Figure 7:** Absence of structure, either as single grain (left) or as massive (right) soil material (<http://soil.gsfc.nasa.gov>).

Structured soils:

Code	Structure name	Description
1	Platy	Flat with vertical dimensions limited; generally oriented parallel to soil surface horizontally and, usually, overlapping with other structure types.
2	Prismatic	The dimensions are greater in the vertical than horizontal direction; vertical faces well defined, having flat or slightly rounded surfaces which are casts of the faces of the surrounding aggregates. Faces normally intersect at relatively sharp angles
3	Columnar	Structure are prisms with rounded caps instead of flat surfaces.
4	Angular Blocky	Blocks or polyhedrons, nearly equidimensional, having flat surfaces which are casts of the faces of the surrounding aggregates. In an angular blocky structure, the faces intersect at relatively sharp angles.
5	Subangular blocky	Same as 4 but with rounded faces.
6	Granular	Spheroids or polyhedrons, having curved or irregular surfaces which are not casts of the faces of surrounding aggregates. Units do not fit into each other
7	Crumbs, lumps and clods	Granular like pedality but with a very high inped porosity. Mainly created by artificial disturbance (e.g. tillage) (FAO, 2006).
8	Massive (coherent)	Soil material (PT2) has normally a stronger consistence and is more coherent on rupture. Massive soil material may be further defined by consistence (see section 3.13).
9	Single grain	Soil material (PT1) has a loose, soft or very friable consistence and consists on rupture of more than 50 % discrete mineral particles.
10	Wedge-shaped	Elliptical, interlocking lenses that terminate in sharp angles, bounded by slickensides; not limited to vertic materials.
11	Nutty	Polyhedral blocky structure with many shiny ped faces which cannot or can only partially be attributed to clay illuviation
12	Rock structure	Rock structure includes fine stratification in unlithified sediment, and pseudomorphs of weathered minerals retaining their positions relative to each other and to unweathered minerals in saprolite.
13	Worm casts	A worm cast or vermicast is a structure created by worms, typically on soils such as those on beaches, that gives the appearance of multiple worms.
14	Layered (coherent)	

The natural types of structure are defined as follows (Figure 8). More than one type in one horizon is possible.



**Figure 8:** Illustrations of some of the most common types of soil structures. From left to right, these are granular, blocky, prismatic, columnar and platy (<http://soil.gsfc.nasa.gov>). The sizes indicated are the normal range, smaller or larger sizes are possible.

### 3.9.2 Size

If a structure is present, the size should be determined. The size classes vary with structure type. For granular, crumble and blocky structures the general size is measured (they are more or less equidimensional), for prismatic, columnar and wedged structures the size classes refer to the measurements of the smallest dimension of the aggregate. For platy structures the thickness of the plates are important, but it is recommended to notice the orientation as well.

Code	Size class	Structure		
		Crumby/ Blocky (mm)	Granular/Prismatic/ Columnar/Wedge-shaped (mm)	Platy (mm)
1	Very fine or thin	< 5	< 10	< 1
2	Fine or thin	5 - 10	10 - 20	1 - 2
3	Medium	10 - 20	20 - 50	2 - 5
4	Coarse or thick	20 - 50	50 - 100	5 - 10
5	Very coarse or thick	> 50	100 - 500	> 10
6	Extremely coarse	-	> 500	-

### 3.9.3 Grade

If the structure is not well developed it can be difficult to estimate the degree of development of the structure, especially if the moisture content is high. Observe if the structural units are well defined on all sides, or only on a few and how easy the units are separated from each other. Grades of structured soil materials are defined as follows:

Code	Class	Description
9	None	Structureless, such as for single grain and massive.
1	Weak	Aggregates are barely observable in place and there is only a weak arrangement of natural surfaces of weakness. When gently disturbed, the soil material breaks into a mixture of few entire aggregates, many broken aggregates, and much material without aggregate faces.
2	Moderate	Aggregates are observable in place and there is a distinct arrangement of natural surfaces of weakness. When disturbed, the soil material breaks into a mixture of many entire aggregates, some broken aggregates, and little material without aggregate faces. Aggregates' surfaces generally show distinct differences with their interiors.
3	Strong	Aggregates are clearly observable in place and there is a prominent arrangement of natural surfaces of weakness. When disturbed, the soil material separates mainly into entire aggregates. Aggregates' surfaces generally differ markedly from their interiors.

## 3.10 Consistence

(FAO, 2006)

Consistence refers to the degree of cohesion or adhesion of the soil mass - friability, plasticity, stickiness and resistance to compression. It depends on the amount and type of clay, organic matter and moisture content of the soil.

For reference descriptions, consistence is required for the soil in dry, moist and wet (both stickiness and plasticity) state. If applicable, thixotropy may be recorded. For routine descriptions, the soil consistence is described in the natural moisture condition of the profile. Wet consistence can always be described, and moist conditions if the soil is dry, by adding water to the soil sample.

### 3.10.1 Consistence when dry

This is determined by breaking the air-dried soil in the hand:

Code	Class	Description
9	Loose	Non-coherent.
1	Soft	Very weakly coherent and fragile; breaks to powder or individual grains under very slight pressure.
2	Slightly hard	Weakly resistant to pressure; easily broken between thumb and forefinger.
3	Hard	Moderately resistant to pressure; can be broken in the hands but not between thumb and forefinger.
4	Very hard	Very resistant to pressure; can be broken in the hands only with difficulty.
5	Extremely hard	Extremely resistant to pressure; cannot be broken in the hands.

### 3.10.2 Consistence when moist

This is determined by squeezing a mass of moist soil material:

Code	Class	Description
9	Loose	Non-coherent.
1	Very friable	Soil material crushes under very gentle pressure, but coheres when pressed together.
2	Friable	Soil material crushes easily under gentle pressure between thumb and forefinger, and coheres when pressed together.
3	Firm	Soil material crushes under moderate pressure between thumb and forefinger, but distinct resistance is felt.
4	Very firm	Soil material crushes under strong pressure; barely crushable between thumb and forefinger.
5	Extremely firm	Soil material crushes only under very strong pressure; cannot be crushed between thumb and forefinger.

### 3.10.3 Consistence when wet

Stickiness depends on water content and the extent to which soil structure is broken down. Wet consistence is described in terms of stickiness and plasticity. It should be assessed under standard conditions on a soil sample in which structure is completely destroyed and which contains just enough water to create maximum stickiness.

Stickiness is the quality of adhesion of the soil to other objects, assessed by observing its adherence when pressed between thumb and finger.

Code	Class	Description
9	Non sticky	After release of pressure, practically no soil material adheres to thumb and finger.
1	Slightly sticky	After pressure, soil adheres to both thumb and finger but comes off one or the other rather cleanly; it is not appreciably stretched when the digits are separated.
2	Sticky	Soil adheres to both thumb and finger and tends to stretch and pull apart rather than pulling free.
3	Very sticky	Soil adheres strongly to both thumb and finger and is decidedly stretched when they are separated.

Plasticity is the ability of soil material to change shape continuously under stress and to retain the given shape on removal of stress. It is determined by rolling the soil into a wire about 3 mm in diameter, then bending the wire.

Code	Class	Description
9	Non plastic	Will not form a wire.
1	Slightly plastic	Wire can be formed but immediately breaks if bent; soil deformed by very slight force.
2	Plastic	Wire can be formed but breaks if bent into a ring; slight to moderate force required for deformation of the soil mass.
3	Very plastic	Wire formed and can be bent into a ring; strong force required for deformation of the soil.

## 3.11 Cutanic features

(FAO, 2006)

This section describes clay or mixed-clay illuviation features, coatings of other composition such as calcium carbonate, manganese, organics or silt; reorientations such as slickensides and pressure faces, and concentrations associated with surfaces but occurring as stains in the matrix (hypodermic coatings). All these features are described according to their type, abundance, contrast, and location.

### 3.11.1 Type

The type of coatings may be described, following Schoeneberger et al. (2002), as:

Code	Description
1	Clay
2	Iron oxides (sesquioxides)
3	Clay and iron oxides (sesquioxides)
4	Clay and humus
5	Humus
6	Silt coatings
7	Sand coatings
8	Calcium carbonate [CaCO <sub>3</sub> ]
9	Silica (opal) [SiO <sub>2</sub> ·nH <sub>2</sub> O]
10	Gibbsite [Al(OH) <sub>3</sub> ]
	Jarosite [KFe <sup>3+</sup> <sub>3</sub> (OH) <sub>6</sub> (SO <sub>4</sub> ) <sub>2</sub> ]

11	Manganese oxide	[Mn <sub>2</sub> O <sub>3</sub> ]
12	Pressure faces	
13	Shiny faces	
14	Slickensides <sup>2</sup> , predominantly intersecting	
15	Slickensides, partly intersecting	
16	Slickensides, non-intersecting	

### 3.11.2 Abundance

For coatings (cutans), an estimate is made how much of the faces of soil aggregates, stones, or pores is covered. Similarly, the proportion of the soil layer occupied by lamellae is estimated.

Code	Description	Class
9	None	0 %
1	Very few	0 - 2 %
2	Few	2 - 5 %
3	Common	5 - 15 %
4	Many	15 - 40 %
5	Abundant	40 - 80 %
6	Dominant	>80 %

### 3.11.3 Contrast

Code	Class	Description
1	Faint	Surface of coating shows little contrast with the adjacent surface. Fine sand grains are readily apparent in the coating. Lamellae are less than 2 mm thick.
2	Distinct	Surface of coating is smoother than, or different in colour from the adjacent surface. Fine sand grains are enveloped but their outlines are visible. Lamellae are between 2 and 5 mm thick.
3	Prominent	Surface of coating contrasts strongly in smoothness or colour with the adjacent surface. Outlines of fine sand grains are not visible. Lamellae are more than 5 mm thick.

### 3.11.4 Location

The location of the coatings is indicated. For pressure faces and slickensides no location is given since they are, by definition, located on the faces of structural aggregates (ped faces).

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<sup>2</sup> A slickenside, is a term describing the surfaces of the cracks produced in soils containing a high proportion of swelling clays. Slickensides are a type of cutan (<http://en.wikipedia.org/wiki/Slickenside>)

Code	Description
9	No specific location
1	Ped faces
2	Horizontal ped faces
3	Vertical ped faces
4	Bridges between sand grains
5	Lamellae (clay bands)
5	Voids
6	Coarse fragments

If the coatings are associated with coarse fragments, following subdivision can be made:

Code	Description
7	Above stones
8	Below stones
9	All around stones

## 3.12 Porosity

(simplified from FAO, 2006) (to be reported in \*.PFH file)

Voids are related to the arrangement of the primary soil constituents and aggregates. They are the results of rooting, burrowing of animals and other soil forming processes such as cracking, translocation, leaching. The term void includes all air and water-filled spaces in the soil; the term pore is often used in a more restrictive way and does not include fissures or planes.

For many purposes, a qualitative description of porosity will suffice. For reference descriptions, voids are described in terms of type, size and abundance; continuity and orientation may also be recorded.

The porosity is an indication of the total volume of voids discernible with a x10 hand lens assessed by area and recorded as the percentage of the surface occupied by pores.

Code	Description	Class limits
1	Very low	<2 %
2	Low	2-5 %
3	Medium	5 – 15 %
4	High	15 – 40 %
5	Very high	> 40 %

## 3.13 Cementation and compaction

(modified from FAO, 2006)

The occurrence of cementation or compaction, as pans or otherwise, is described according to their nature, continuity, structure, agent and degree. Cemented material does not slake after one hour of immersion in water.

### 3.13.1 Nature (type)

The cementing agent or compaction activity composes of:

<b>Code</b>	<b>Description</b>
1	Gypsum
2	Silica
3	Carbonates
4	Iron oxides
5	Iron-manganese oxides
6	Iron-organic matter
7	Organic matter
8	Others
9	Not known

### 3.13.2 Continuity

<b>Code</b>	<b>Class</b>	<b>Description</b>
1	Broken	The layer is less than 50 % cemented/compacted and appears irregular
2	Discontinuous	The layer is 50 - 90 % cemented/compacted and appears regular
3	Continuous	The layer is more than 90 % cemented/compacted, and has few cracks only

### 3.13.3 Structure

The structure (or fabric) of the cemented/compacted layer may be described as:

<b>Code</b>	<b>Class</b>	<b>Description</b>
9	None	Massive without recognizable orientation
1	Platy	The cemented/compacted parts are plate-like with more or less horizontal orientation
2	Vesicular	The layer has large, equidimensional voids which may be filled with uncemented material
3	Pisolithic	The layer is composed of cemented, spherical nodules
4	Nodular	The layer is composed of cemented nodules or concretions of irregular shape

### 3.13.4 Degree

<b>Code</b>	<b>Class</b>	<b>Description</b>
9	Non-cemented and non-compacted	No compaction/compaction is observed (slakes in water)
1	Compacted	Compacted soil material is harder or more brittle than non-compacted soil material. Non-cemented.
2	Weakly cemented	Cemented mass is brittle and hard, but can be broken in the hands
3	Moderately cemented	Cemented mass cannot be broken in the hands but is discontinuous (less than 90 % of soil mass)
4	Cemented	Cemented mass cannot be broken in the hands and is continuous (more than 90 % of soil mass)
5	Indurated	Cemented mass cannot be broken by body weight (75 kg standard soil scientist) (more than 90% of soil mass)

### 3.14 Nodules and other mineral concentrations

(FAO, 2006)

Mineral nodules cover a large variety of secondary concentrations. There are gradual transitions with mottles. Nodules are described according to their kind, type, abundance, size, shape, hardness and colour, as well as their presence within the horizon:

#### 3.14.1 Kind

Code	Class	Description
1	Crystal	
2	Concretion	A discrete body with a concentric internal structure, generally cemented
3	Soft segregation	Differs from the surrounding soil mass in colour and composition but is not easily separated as a discrete body
4	Nodule	Discrete body without an internal organization
5	Pore infillings	Including pseudomycelium of carbonates and opal
6	Crack infillings	
7	Residual rock fragment	Discrete body still showing rock structure

#### 3.14.2 Type

Nodules are described according to their composition or impregnating substance. Examples:

Code	Description
1	Gypsum
2	Silica
3	Carbonates
4	Carbonates-silica
5	Salt
6	Clay
7	Clay-oxides
8	Manganese oxides
9	Iron-manganese oxides
10	Iron oxides
11	Sulphur
12	Not known

#### 3.14.3 Abundance (by volume)

Code	Description	Class limits
9	None	0 %
1	Very few	0 - 2 %
2	Few	2 - 5 %
3	Common	5 - 15 %
4	Many	15 - 40 %
5	Abundant	40 - 80 %
6	Dominant	> 80 %

### 3.14.4 Size

Code	Description	Class limits
1	Very fine	< 2 mm
2	Fine	2 - 6 mm
3	Medium	6 - 20 mm
4	Coarse	> 20 mm

### 3.14.5 Shape

Code	Description
1	Rounded (spherical)
2	Elongated
3	Flat
4	Irregular
5	Angular

### 3.14.6 Hardness

Code	Class	Description
1	Hard	Cannot be broken between the fingers
2	Soft	Can be broken between forefinger and thumb nail
3		Both hard and soft

### 3.14.7 Colour

General colour names are usually sufficient to describe nodules, in the same way as mottles:

Code	Description
1	White
2	Yellow
3	Yellowish red
4	Reddish yellow
5	Red
6	Yellowish brown
7	Reddish brown
8	Brown
9	Green
10	Blue
11	Bluish-black
12	Grey
13	Black
14	Multicoloured

## 3.15 Roots

### 3.15.1 Abundance

(FAO, 2006) (to be reported in \*.PFH file)

Presence/absence of roots is the most essential information to take notice of. A qualitative description of the size and the abundance of roots is important. Remember the abundance of roots should only be compared within the same size class.

Abundance (number of roots/dm<sup>2</sup>) per size class:

Code	Size class	Very fine	Fine	Medium	Coarse
		<0.5 mm	0.5-2 mm	2-5 mm	>5 mm
9	None	0	0	0	0
1	Very few	1 - 20	1 - 20	1 - 2	1 - 2
2	Few	20 - 50	20 - 50	2 - 5	2 - 5
3	Common	50 - 200	50 - 200	5 - 20	5 - 20
4	Many	>200	>200	>20	>20

### 3.15.2 Distribution

If there is a sudden change in the quantity and/or size of the roots it is very important to explain why. Possible root limiting factors are: compaction (check the bulk density), cementations, discontinuous pore system, etc. Sometimes it may be useful to record additional information, such as an abrupt change in root orientation.

Code	Description
1	Continuous
2	in the space of cracks
3	in the space of vughs and channels
4	concentrated in nests

## 3.16 Other biological features

(FAO, 2006)

Krotovinas (an animal burrow which has been filled with material from another horizon), termite burrows, insect nests, worm casts, burrows or other disturbances of larger animals are described in terms of abundance and kind. In addition, specific locations, patterns, size, composition or any other characteristic may be recorded.

### 3.16.1 Kind

Examples of biological features are the following:

Code	Description
1	Burrows (unspecified)
2	Open large burrows
3	Infilled large burrows
4	Earthworm channels
5	Termite or ant channels and nests
6	Other insect activity
7	Pedotubules <sup>3</sup> (voids filled with soil material by faunal and floral activity, for further info read the footnote)
8	Charcoal

### 3.16.2 Abundance

Abundance of biological activity is recorded as a percentage of the exposed surface:

Code	Description	Class limits
1	Few	<5%
2	Common	5-15%
3	Many	15-40%
4	Abundant	>40%

### 3.16.3 Continuity

Abundance of biological activity is recorded as a percentage of the exposed surface:

Code	Description
1	Continuous
2	Discontinuous
3	Patchy
4	Locally
5	Other (explain)

## 3.17 Carbonates

(modified from FAO, 2006)

The presence of calcium carbonate ( $\text{CaCO}_3$ ) is indicated by adding some drops of 10% HCl to the soil. Following information should be collected per horizon:

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<sup>3</sup> The term pedotubules is proposed for a group of pedological features which have a tubular external form and which are distinguished from cutans by their complex internal composition and fabric. Pedotubules are classified according to their internal fabric and composition, details of external form, distinctness, and by a comparison of their fabric and composition with that of the horizons of the soil profile. Their general morphology suggests their origin as voids caused by faunal and floral (root) activity which have been filled, or partially filled, with soil material. Since little is known of the details of the effects of faunal activity on soil materials, such interpretations are tentative (Brewer and Sleeman, 1963)

Is the matrix calcareous or non-calcareous (the exact quantity of carbonates will be tested in the laboratory). If traces are found in at least one horizon of the profile, the presence/absence should be recorded for all horizons.

Is the carbonate at least partly secondary (pedogenic).

### **3.17.1 Presence of carbonates**

Following categories apply:

Code	Description
9	No presence of carbonates
1	Matrix is non-calcareous, presence of secondary carbonate
2	Matrix is calcareous, no evidences of secondary carbonate
3	Matrix is calcareous, presence of secondary carbonates

### **3.17.2 Type of secondary carbonates**

The type of secondary carbonates should be described. Following categories has been defined, more can be defined where applicable:

Code	Description
1	Capping
2	Coatings
3	Nodules
4	Pendants
5	Pseudomycelia
6	Others (define)

## **3.18 Gypsum**

(modified from FAO, 2006)

Gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) may occur as residual, gypsous parent material or as newly formed features such as pseudomycelia, coarse crystals (commonly as nests, beards or coatings, or as elongated groups of fibrous crystals), or loose to compact powdery accumulations.

Is gypsum present in the horizon? If so, where is it present (in the matrix, as nodules... ...) and in which form is it present (crystals, powder,...; primary or secondary etc.). If gypsum is present in the soil, recommendations should be made for the laboratory on which samples the content of gypsum should be measured. The presence/absence of gypsum is explained applying following categories:

### 3.18.1 Abundance of gypsum

<b>Code</b>	<b>Abundance (by volume)</b>
9	0%
1	0 - 5%
2	5 - 15%
3	15 - 25%
4	25 - 60%
5	> 60%

### 3.18.2 Abundance of secondary gypsum

<b>Code</b>	<b>Abundance (by volume)</b>
9	0%
1	0 - 5%
2	> 5%

## 3.19 Readily soluble salts (modified from FAO, 2006)

Readily soluble salts are more soluble than gypsum; the most common salts are chlorides. The salt content of the soil can be estimated from the electrical conductivity (EC in dS/m = mS/cm) measured in a saturated soil paste or a more diluted suspension of soil/water.

If salts are observed during fieldwork, the electric conductivity of a saturated paste should be analysed for all horizons in the soil.

Is salt present?

- Y      Evidences of soluble salts
- N      No evidences of soluble salts
- X      Not known

## 3.20 Man-made materials

(simplified from FAO, 2006)

The areas dominated or significantly changed by human activity are rapidly extending, especially in urban and mining areas. Of particular importance are the man-made materials found in soils; their age, amount, state and composition determine their durability and environmental impact. Any human impact on the soil should be recorded. Examples are:

- Evidences of past agriculture
- Presence of artefacts (e.g. ceramics)
- Remains of past structures (e.g. postholes)
- Other features of possible human origin (e.g. charcoal, brick fragments)

### 3.21 Human-transported material (simplified from FAO, 2006)

This is any material brought onto the site. This may be for agricultural purposes (e.g. large-scale terracing, mine spoil...), for human settlement, or simply to dispose of material (e.g. dredged sediment). It is a soil parent material in the same way as alluvium.

### 3.22 Soil horizon designation

The term horizon indicates a soil layer presumed to bear the imprint of soil forming processes, as opposed to layers that are laid down by sedimentation, volcanic activity or other geological events.

Horizons are identified by symbols that consist of one or two capital letters for the master horizon and lower case letter suffixes for subordinate distinctions, with or without a figure suffix. The detailed definitions of the different horizon symbols and rules that apply are found in Annex 7. This annex also explains the use of symbols to express lithologic discontinuities and the vertical subdivision when two horizons have the same set of master and subordinate symbols.

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### **Annex 3: Recommendations for locating and describing a soil profile**



## Location of the soil profile

The location of the soil profile should be as representative for the level plot as possible. Obviously only surface characteristics can help us in this search. Factors of importance can be:

1. Composition of ground vegetation, e.g. if most of the experimental plot hosts no ground vegetation do not locate the profile where vegetation is present
2. Composition of tree stand; dig the profile under the canopy of dominant tree species.
3. Avoid areas of strong human influence, such as ditches, earth banks, forest roads, tracks from tree harvesting machines...
4. Avoid micro-lows and micro-highs, as they will allow more or less litter to accumulate, which will have an influence on the biological activity and hydrology.
5. On experimental plots with steeper slopes, it is important to locate the profile as representative as possible with respect to the general slope inclination. If the general slope is concave or convex, then try to locate the profile at the level of the plot centre with respect to the slope (meaning in the zone immediate outside the experimental plot to the left or the right of this plot centre: not down- or upslope).
6. Other factors such as surface stoniness, rock outcrop, different land-use practice etc. should also be taken into consideration so that the profile location is as representative as possible.

## Orientation of the soil profile

Factors to take into consideration are:

7. If the slope inclination is such that it will have an important impact on the hydrology, then the profile should be oriented with its long axe in the slope direction.
8. If the slope inclination is not important, the profile is oriented in such a way that by the time the profile is to be studied the light should be equally distributed over the complete profile wall (e.g. if you start to dig at 10 AM and you estimate that it takes 2h to dig the profile, and 30min to clean it for taking photos, then orient the profile towards SSW (180-200°). By the time you can take photos the profile will have a perfect angle towards the sun. A wall that is partly shaded partly with sunshine is impossible to describe optimally, and no quality photos can be produced.
9. If the slope and light is not a problem, then other factors can help with the orientation of the profile, such as microtopography, vegetation, etc. For example, if the experimental plot is characterised by drainage ditches, orienting the profile with its long axis perpendicular to the ditch will result in a profile where on the side walls it will be possible to observe the changes from a wetter soil closest to the ditch towards a drier soil between the ditches.

## Observations to be made while digging the profile

10. If the field work is organised in such a way that the person that will do the profile description will be present when the profile is dug, it is advisable to make some first observations during this process. Following aspects of a standard profile description can be substantially improved if already considered during the profile digging, simply because the

observations are made not only based on a two dimensional profile wall, but based on about one cubic metre of soil that is removed:

11. the rock fragments: description of the abundance, size, shape, and type is considerably improved, and special features such as presence/absence of pendants/cappings on the stones are better observed.

- I. Soil structure, especially type and degree of development
- II. Cutanic features, especially if the quantity is low working in a three dimension will improve the chance to observe such features
- III. Presence of cementations and compactations will undoubtedly be discovered when the profile is worked with the spade, shovel and pickaxe
- IV. If carbonate is present, observing if at least part of the carbonate is secondary will be easier
- V. Quantification and size estimates of roots, as well as the total root depth is more accurately observed

## Observations in three dimensions

12. A standard profile is typically 80-100 cm wide, 180-200 cm long and should have a depth of 200 cm. The wideness and length can be reduced if e.g. the soil is very stony, but be careful not to diminish the profile beyond the size where proper use of spade, shovel, pickaxe is restricted. The profile depth can be limited by a series of factors, such as:

- I. The groundwater table. If the permeability of the soil is low, digging below the groundwater table is possible and even soil sampling and/or making a few observations such as colour, reductomorphic properties, ... is possible. Remember to measure the actual groundwater depth
- II. Bedrock, either continuous or discontinuous rock that prevents further digging
- III. Cementations of any kind, For example a Petrocalcic, Petroduric, Petrogypsic, Petroplinthic or Pisoplithic cemented horizon which makes any further digging impossible.
- IV. Parent material. If the C horizon material is reached at a shallower depth than 200 cm, further digging can be stopped. It is though recommended to continue 20-50 cm to control that it indeed is the C-horizon that has been reached.

13. When the profile in its full length has reach a depth of about 100-120 cm, further digging is restricted to the 80-100 cm of the profile closest to the front wall. This creates a soil pit with 2 or more steps.

14. After digging the profile, it is essential to clean the profile walls.

- I. This is done by e.g. a knife, trowel, or another scraping tool with an metal blade, make sure that the metal blade has rounded edges to avoid sharp scraping lines on the profile wall.
- II. While the profile is cleaned, the soil is carefully observed, with respect to colours, presence absence of roots, biogalleries, stones and other characteristics that might be important to outline the horizons.
- III. Take the first round of photos (see paragraph 3.2 of Annex 2): Of the profile, details of the profile, and the surrounding landscape
- IV. Draw on the profile wall with the knife the horizon boundaries. The most important feature to delineate horizons from each other is change of colour. Other feature that can be used to differentiate genetic horizons from each other are a relatively sharp change over (vertical)

- distance of mottles, texture, coarse fragments, structure, porosity, cementation, compaction, nodules, roots or carbonates.
- V. A second round of photos ((see paragraph 3.2 of Annex 2) is taken of the profile with and without the horizon boundaries indicated, and of particular details such as mottles, involutions, biogalleries, disturbances etc.
15. If a soil is composed of well developed relatively uniform horizons, focussing on the front wall for the profile description is usually sufficient. If on the other hand the profile is more irregular and/or the horizons are less developed, it might be necessary to study also the side walls. This should appear from the profile description. For example the horizon depths in soil profiles located on a strong slope should be measured perpendicular to the surface, which is easiest on the side wall.
16. For certain pedological features, it might be useful to study them on a horizontal surface. This is for example possible while digging the profile, or when the first or second stair has been made at the correct depth. If necessary, a new sub-profile is dug on the sidewall to the depth(s) where a horizontal section is needed.



## **Annex 4: Field equipment**



<b>Equipment for profile description:</b>	<b>Number</b>	<b>Further information</b>
Spade, pickaxe	1	
Shovel	2	
Bucket	1	
Scraper	1	Make the corners rounded to avoid sharp lines on the cleaned profile wall
Trowel	1	Sharpen the edges to allow a better cutting in the soil
Knife	1	The blade should be straight (e.g. to cut bulk density samples straight) and as long as possible (recommended is 15-20 cm)
Field umbrella	1	To shade for sun and rain
Pruner or horticulture sheer	1	To cut roots
Small painting brush	optional	To highlight certain special features e.g. slickensides, stones, etc.
Munsell Colour Chart	1	Try to avoid dirt on the colour chips
Folding ruler or measuring tape	2	Two coloured ones are preferred
Note book	1	Large enough e.g. A5 format, with squared lines on the paper to facility profile drawings
Writing pen/pencil	2	
Marker pen	2	For sample labelling, black colours are more resistant to sun light
Sample labels	sufficient	
Hand lens	1	Magnifications x10 (4 +6 or 2 + 8)
Soil thermometer	optional	Especially if the soil at or below 0°C
Penetration rod	1	Required if the soil is compacted, cemented or is stony
Clinometer or Abney-level	1	To measure slope inclinations
GPS system	1	Measuring coordinates of key points on the plot
Compass	1	For orientation of e.g. slope direction
Auger handle	1	
Auger heads	selection	Selection in accordance to soil type, see table below
Extension rods	1	With one extension a depth of 225 cm can be reach
Photo camera and tripod	1	
Water bottle	1	
Water sprayer	1	
<b>For sampling</b>	<b>Number</b>	<b>Further information</b>
Sampling tray	optional	
Sample recipients	sufficient	Quality bags (plastic, cloth..) or boxes
Sample labels	sufficient	
Sample frame	1 - 3	To sample the organic layer
<b>For pF and BD</b>	<b>Number</b>	<b>Further information</b>
Bulk density cylinders (volume between 100 cm <sup>3</sup> and 250 cm <sup>3</sup> ) and lids	sufficient	Take care not to destroy the cutting edge of the ring while inserting it into the soil
Ring holder	optional	Needed if the soil is hard (e.g. due to dryness and/or a high clay content)
Impact free hammer	1	Eventually a geological hammer if fieldwork is done in

		mountainous regions
Wood piece	1	To distribute the hammer impact equally
Hammering head	optional	For very hard soils, to be used with the guide cylinder
Cylinder guide	optional	
Small iron saw	1	To cut the edges of the sampling rings
Liquids:	Number	Further information
Distilled water*	±100 cl	To test moist colours; testing for water repellence
Concentrated H <sub>2</sub> O <sub>2</sub> *	±100 cl	Reacts to manganese (charcoal and organic matter not)
10% HCl*	±100 cl	Reacts to carbonates
α, α dipyridyli*	±100 cl	Reddish colour reaction if Fe <sup>2+</sup> is present Read also: <a href="http://soils.usda.gov/use/hydric/ntchs/tech_notes/note8.html">http://soils.usda.gov/use/hydric/ntchs/tech_notes/note8.html</a>

\* The liquids are by preference stored in bottles, which allow drop wise application (like eye drops). If this is not possible, bring along plastic pipettes for careful application.

Soil augers:

Soil texture	Moisture condition	Type of Edelman augers	Riverside auger	Gouge auger
	wet	sand	-	-
Sandy	moist	combi, sand	(+)	+
	dry	sand	-	(+)
	wet	Combi	-	+
Loamy/silty	moist	Combi	-	+
	dry	-	+	-
	wet	Clay	-	+
Clayey	moist	Clay	-	+
	dry	-	+	-
	wet	Gravel	-	-
Stony	moist	Gravel	-	-
	dry	-	+	-
Frozen soil		(gravel)	(+)	-

"-" : not suitable

"(+)" : possibly suitable

"+" : suitable (recommended)

Clay-auger: thin blades, good for clayey and/ sticky soils when moist and wet

Combi-auger: all round auger best for medium textured soils when moist and wet

Sand-auger: has wide blades, so the sand stays in the auger even if the soil is relatively dry

Gravel-auger: with two cutting blades at the end that can drill and remove small stones

Riverside-auger: closed with drilling blades, the only auger that can be used in dry loamy or dry clayey soils. It should be avoided when these soils are moist or wet! Sometimes also useful in frozen soils

Gouge-auger: for non-stony soils preferentially moist or wet. It can take undisturbed samples. Mode length variable, mostly 50 to 100 cm long.

For the sampling of the organic layer a frame of 25 by 25 cm is recommended, but alternatives with a minimum total surface of 500 cm<sup>2</sup> are acceptable. For mor humus, an auger with a diameter of 8 cm can be used.



## **Annex 5: Additional information on colour measurements**



This annex provide additional information on the colours, particular for optimal measurements for classification purposes (IUSS WG on WRB 2006, update 2007)

Colour should be determined, if possible, under uniform conditions out of direct sunlight. Early morning and late evening readings are not accurate. Determination of colour has proven to be often inconsistent between individuals or, even for the same person. Since colour is significant with respect to many soil properties and for soil classification, routine cross-checks are recommended. To save time in the field and to improve the colour measurements, it is recommended to measure the colours in the laboratory. Let fragments of the soil air-dry before the dry colours are measured, then gently add vaporised water to moisten the sample to field capacity. The wet colour can be measured after saturation of the sample with water. Colours for waterlogged soils should be measured immediately before the soil gets oxidised.

For all horizons, at least the moist colours should be recorded (if the soil is arid or wet, respectively dry and wet colours may replace the moist ones). In those soils where one or more diagnostic horizons or properties might be present, additional colours are mandatory as summarised in the table. For example, for a soil with a spodic horizon (diagnostic for Podzols), information on the moist crushed colour is required according to the WRB.

**Table 4:** Required colour measurements.

Soil moisture condition	Measured colour	Horizons for which colours are required				
		O, H	A	E	B	C
Dry	Matrix (broken)		2a, 2b	4	5	3
	Crushed		2a, 2b			
Moist	Matrix (broken)	1	1, 2a, 2b	1	1	1
	Crushed		2a, 2b		6	
Wet	Matrix	1b	1b	1b	1b	1b

1. Moist colours should always be recorded for all horizons in the profile, except for those soils that 1b) naturally are wet (e.g. Gleysols, Histosols). For those soils, the wet colours are measured instead of moist colours. The matrix colour can be considered the same as the broken colour.
2. 2a) Broken colours are mandatory to record if the soil has a dark topsoil with colours of 3.5/3.5 (value/chroma) or less when moist, and a value of 5.5 or darker when dry. 2b) If the soil may have aridic properties (relative low organic carbon content, evidences of aeolian activity, high base saturation...) these colour measurements are required too.
3. Should be measured if the topsoil has colours of 3.5/3.5 (value/chroma) or less when moist, and a value of 5.5 or darker when dry (see 2a). If no C-horizon is present, the colour should be measured for the horizon immediately underlying the surface horizon(s).
4. If the eluvial horizon has a colour value of 4 or more and a chroma of 4 or less, then measure also the dry colour.
5. If the moist colour is redder than 5YR (3.5YR or redder) then also the dry colour should be measured (required for the rhodic qualifier).
6. If the soil may have a spodic horizon (required for Podzols), then the crushed colour is mandatory to record. The colour required for a spodic horizon (moist) is 10YR or redder with value of 3 or less and chroma of 2 or less. If the colour is 7.5YR or redder the value required is 5 or less and the chroma 4 or less.



## **Annex 6: Guidelines for field estimates of textural classes**



The textural class can be estimated in the field by feel. For this, the soil sample must be moist (as close as possible but not exceeding the field capacity). Fragments >2 mm must be removed.

Clay:	Soil can be rubbed, is sticky, can be moulded but is stiff (high plasticity), smeared surface is shiny
Silt:	Soil can be rubbed, is non-sticky, and feels floury (like talc)
Sand:	Cannot be moulded, does not soil fingers and feels gritty

### Key to the soil texture classes (adapted from Schlichting et al., 1995)

y	<b>Cannot be rolled into a ball</b>	% clay
	Not dirty, not floury, no fine material adhering to fingers: <b>sand</b>	<b>S</b> <5
	If grain sizes are mixed: <b>Unsorted sand</b>	<b>US</b> <5
	If most grains are very coarse (>0.5 mm): <b>very coarse and coarse sand</b>	<b>CS</b> <5
	If most grains are of medium size (0.25-0.5 mm): <b>medium sand</b>	<b>MS</b> <5
	If most grains are of fine size (<0.25 mm) but still grainy: <b>fine sand</b>	<b>FS</b> <5
	If most grains are of very fine size (<0.1 mm), almost floury: <b>very fine sand</b>	<b>VFS</b> <5
2.	<b>Can be rolled into a ball but not into a wire</b>	
	Not floury, grainy, scarcely any fine material sticking to fingers, forms a ball weakly, adheres slightly to the fingers: <b>loamy sand</b>	<b>LS</b> <12
	As 2.1 but floury: sandy loam	<b>SL</b> <10
3.	<b>Can be rolled into a wire of about 3 to 7 mm in diameter (about half the diameter of a pencil) but breaks when trying to bend into a ring, sticks to the fingers</b>	
	Floury and not cohesive	
	Some grains to feel: <b>silt loam</b>	<b>SiL</b> <10
	Floury and not cohesive, no grains to feel: <b>silt</b>	<b>Si</b> <12
	Cohesive, sticks to the fingers, has a rough and smeared surface after squeezing between fingers	
	Is very grainy and not sticky: <b>sandy loam</b>	<b>SL</b> 10-25
	Neutral feel, neither sticky, nor gritty, nor floury: <b>loam</b>	<b>L</b> 8-27
	Is not grainy but distinctly floury and somewhat sticky: <b>silt loam</b>	<b>SiL</b> 10-27
	Is very grainy and not sticky: <b>sandy loam</b>	<b>SL</b> 10-25
	Sticky and grainy to very grainy: <b>sandy clay loam</b>	<b>SCL</b> 20-35
4.	<b>Can be rolled into a wire smaller than 3 mm in diameter (less than half of that of a pencil) and bent to form a ring of about 2-3 cm in diameter, cohesive, sticky, shiny smeared surface</b>	
	Very grainy: <b>sandy clay</b>	<b>SC</b> 35-55
	Grains can be seen and felt: <b>clay loam</b>	<b>CL</b> 25-40
	No grains to see and to feel, low plasticity: <b>silty clay loam</b>	<b>SiCL</b> 25-40
	No grains to see and to feel, high plasticity: <b>silty clay</b>	<b>SiC</b> 40-60
5.	<b>Shiny smeared surface and high plasticity</b>	
	Some grains to see or to feel, grates between teeth: <b>clay</b>	<b>C</b> 40-60
	No grains to see or to feel, does not grate between teeth: <b>heavy clay</b>	<b>HC</b> >60



## **Annex 7: Detailed definitions, rules and conventions on master and subordinate horizon symbols**



## 1. Vocabulary

**Organic material (OM)** (IUSS Working Group WRB, 2007) (from Greek *organon*, tool) consists of a large amount of organic debris that accumulates at the surface under either wet or dry conditions and in which the mineral component does not significantly influence the soil properties.

**Diagnostic criteria.** Organic soil material must have one of the two following:

1. 20 percent or more organic carbon (by mass) in the fine earth; **or**
2. if saturated with water for 30 consecutive days or more in most years (unless drained), one or both of the following:
  - a.  $(12 + [\text{clay percentage of the mineral fraction} * 0.1])$  percent or more organic carbon in the fine earth (by mass), **or**
  - b. 18 percent or more organic carbon (by mass) in the fine earth.

### Organic horizons

The organic horizons (codes: OL, OF, OH) are formed by dead organic matter (OM), mainly leaves, needles, twigs, roots and, under certain circumstances, materials such as mosses and lichens. This OM can mainly be transformed in animal faeces. An organic horizon contains 20 % or more organic carbon (by mass) in dry samples, without living roots.

### Hemorganic horizons

The hemorganic horizons (code: A) are formed near the soil surface, generally beneath the organic horizons. Coloured by organic matter, these horizons are generally darker than the underlying mineral part of the soil profile. It is generally accepted that in the soil fraction < 2mm of the A horizon, the organic carbon has to be less than 20% by mass (following WRB FAO 2006, Broll et al. 2006).

### Recognisable remains

Recognisable remains within an organic or hemorganic horizon are organic materials like leaves, needles, roots, bark, twigs and wood, fragmented or not, whose original organs are recognizable by the naked eye or with a 5-10 X magnifying hand lens. Fresh litter generally consists for 100% of recognizable remains (Zanella et al. 2010)

### Humic components

Humic components of an organic or hemorganic horizon are small particles of organic remains and/or grains of organic or organo-mineral matter mostly comprised of animal droppings of different sizes. The original organs which compose the litter and generate the small particles (free or incorporated in animal faeces) are not recognizable by the naked eye or with a 5-10 X magnifying hand lens. Bound mineral particles can be visible within the mass.

Well decomposed organic substrate generally consists for 100% out of humic components. However, the generated humic component can also be in the hemorganic (A) and organic (OL,

OF, OH) horizons. Thus, an A horizon made of anecic and endogeic earthworm hemorganic faeces as well as a totally, finely decomposed and mostly organic OH horizon resulting from enchytreid and microarthropod activities, can both be composed for 100% of humic components, despite differences in the animals responsible for the structure of the horizons (Zanella et al. 2010).

### Fibric component

Non-decomposed or very weakly decomposed hygrophilous plant remains like sphagnum species, sedges, rushes, reeds... Whole plants, parts of them and/or free plant organs (leaves, needles, twigs, wood, roots...) sometimes lying in more or less dark coloured layers.

### Sapric component

Homogeneous dark organic and organo-mineral matter comprised of well decomposed plant remains partly mixed with mineral particles. Plant structures are not visible to the naked eye or with a 5-10 X magnifying hand lens. Animal droppings are possible in periodically drained horizons and can be abundant in drained peats.

### Mineral components

Mineral components of an organic or hemorganic horizon are mineral particles of different sizes, free or very weakly bound to humic components and visible by the naked eye or with a 5-10 X magnifying hand lens.

### Zoogenic transformed material

Zoogenic transformed materials are recognizable remains and humic components processed by animals (i.e. leaves, needles and other plant residues more or less degraded by soil animals, mixed with animal droppings. A fine, powdered and/or grained structure (less than 1 mm) is typical in a terminal stage of faunal attack in an organic horizon. At this last level of biotransformation, the substrate (OH horizon) is essentially comprised of organic animal droppings of varying size (droppings of epigeic earthworms, macro-arthropods such as millipedes, woodlice and insect larvae, micro-arthropods such as mites and springtails and enchytraeids dominate). Within hemorganic horizons, animal activity leads to different types of A horizons, depending on the animals' ability to dig into the mineral soil and thoroughly mix organic and mineral matter.

### Non zoogenic transformed material

Non zoogenic transformed materials are recognizable remains and humic component processed by fungi or other non-faunal processes (i.e. leaves, needles and other plant residues more or less fragmented and transformed into fibrous matter by fungi. Recognizable and recent animal droppings are absent or not detectable by the naked eye in the organic horizons; fungal hyphae can be recognized as white, brown or yellow strands permeating the organic or hemorganic substrates; traces of animal activity (old bite marks, mucus) may sometimes be detectable but are always marginal. In the last stage of biodegradation of an organic horizon, non zoogenic substance may essentially be composed of brown, dry plant residues more or less in powder form or tiny fragments (OF and OH horizons), or be massive like a dark wet plastic clay (OH or very organic A horizons).

By the naked eye or with a 5-10 X magnifying hand lens, each topsoil horizon appears to be composed of recognizable remains, humic and mineral components (fig. 1). At the soil surface,

the animals or plant cover shed litter (remains) on and within the soil. Animal remains exist too, but they are quite negligible compared to plant remains and often overlooked by the neophyte. Litter is made up of recognizable residues and humic components (because of soil biological activity). In the other direction, the geological substrate "leaks" fragments of rock, which in the topsoil become mineral (free grains) and humic (grains incorporated in faeces) components. The mineral component can also be increased by surface erosion. The process perceived by the naked eye hides a more complex world of chemical-physical-biological processes. In unfavourable conditions for soil fauna the process is mainly dominated by fungi. Therefore, the new definitions of zoogenic and non zoogenic transformed material have been coined to distinguish zoogenic biological transformation from mycogenic transformation. The aim of a naked eye examination is to collect initial data and information for the purposes of a more detailed research on the same system. The vocabulary terms have been selected with regards to a dynamic interpretation of current knowledge on the topsoil.

## 2. Soil horizon designation

### 2.1 Master horizons and layers

#### 2.1.1 The Organic horizons

##### 2.1.1.1 O horizon or layer

The O horizon or layer is dominated by organic material, consisting of fresh, partially or completely decomposed litter (such as leaves, needles, twigs, mosses, and lichens) that has accumulated on the surface; it may be on top of either mineral or organic soil. It is not saturated with water for prolonged periods. The mineral fraction of such material is only a small part of the volume of the material and generally is much less than half of the mass.

An O layer may be at the surface of a mineral soil or at any depth beneath the surface if it is buried. However, a horizon formed by illuviation of organic material into mineral subsoil is not an O horizon (FAO, 2006).

A subdivision of the organic O-layers is made according to the following definitions (Zanella et al. 2010):

**OL-horizon** (Litter, Förna): this organic horizon is characterised by an accumulation of mainly leaves/needles, twigs and woody materials (including bark), fruits etc.

This sublayer is generally indicated as litter (Klinka et al., 1981, Green et al., 1993, Jabiol et al., 1995, Delecour, 1980). It must be recognized that, while the litter is essentially unaltered, it is in some stage of decomposition from the moment it hits the floor and therefore it should be considered as part of the humus layer.

There may be some fragmentation, but the plant species can still be identified. So most of the original biomass structures are easily discernible. Leaves and/or needles may be discoloured and slightly fragmented. The humic components amounts to less than 10 % by volume; recognisable remains 10% and more, up to 100% in non-decomposed litter.

OL types (suffixes: n, v, t)

OLn: new litter (age < 1 year), neither fragmented nor transformed/discoloured leaves and/or needles;

OLv: old litter (aged more than 1 year, vetust, verändert, verbleicht), slightly altered, discoloured, bleached, softened up, glued, matted, skeletonized, sometimes only slightly fragmented leaves and/or needles.

OLT: transitional (t) litter made of leaf petioles, twigs, bark and surface cast accumulation in very active mull forms (eumull generally in mixed hardwood forest – described in ash/beech stands).

Note: The passage from OLn to OLv can be very rapid (1 to 3 months) or very slow (more than a year) according to types of litter (plant species composition), climate, season and level of soil biological activity

**OF-horizon** (fragmented and/or altered) is a zone immediately below the litter layer. This organic horizon is characterised by an accumulation of partly decomposed (i.e. fragmented, bleached, spotted) organic matter derived mainly from leaves/needles, twigs and woody materials. The material is sufficiently well preserved to permit identification as being of plant origin (no identification of plant species). The proportion of humic components is between 10 % and 70 % by volume. Depending on humus form, decomposition is mainly accomplished by soil fauna (OFzo) or cellulose-lignin decomposing fungi (OFnoz). Slow decomposition is characterised by a partly decomposed matted layer, permeated by hyphae.

Note: this is the **fragmented layer** in non-saturated soils (Klinka et al., 1981, Green et al., 1993, Jabiol et al., 1995, Delecour, 1980)

OF types (suffixes: zo, noz)

OFzo = content in zoogenic transformed material > 10% of the volume of the horizon;

OFnoz = content in non zoogenic transformed material 90% or more of the volume of the horizon;

**OH-horizon** (humus, humification): characterised by an accumulation of dark, well-decomposed, amorphous organic matter. It is partially coprogenic, whereas the F horizon has not yet passed through the bodies of soil fauna. The humified H horizon is often not recognized as such because it can have friable crumb structure and may contain considerable amounts of mineral materials. It is therefore often misinterpreted and designated as the Ah horizon of the mineral soil and not as part of the forest floor as such. To qualify as organic horizon, it should fulfil the FAO requirement, as described above. The original structures and materials are not discernible. Humic components amounts to more than 70 % by volume. The OH is either sharply delineated from the mineral soil where humification is dependent on fungal activity (mor) or partly incorporated into the mineral soil (moder).

Note: This horizon coincides with what is called the **humus layer** (Klinka et al., 1981, Green et al., 1993, Jabiol et al., 1995, Delecour, 1980)

OH types (suffixes: zo, noz, r, f)

OHzo = content in zoogenic transformed material > 30% of the volume of the horizon;

OHnoz = content in non zoogenic transformed material 70% or more of the volume of the horizon;

Concerning the organic layers, a distinction is made between the water saturated organic layers, designated as 'H', and the aerated organic materials indicated as 'O'.

### 2.1.1.2 H horizon or layer

The H horizon or layer is dominated by organic material, formed from accumulations of fresh or partially decomposed organic material at the soil surface (which may be under water). All H horizons are saturated with water for prolonged periods or were once saturated but are now drained. A H horizon may be on top of mineral soils or at any depth beneath the surface if it is buried (FAO, 2006).

Distinction of subhorizons in the organic H-layers (Zanella et al., 2010):

### Hf (from Histic and fibric) horizon

Histic organic horizon consisting almost entirely of almost unchanged plant remains. Fibric component  $\geq 90\%$ , sapric component  $< 10\%$  of horizon volume. Content of rubbed fibres  $\geq 40\%$  of soil by dry weight ( $105^{\circ}\text{C}$ )<sup>4</sup>. Von Post scale of decomposition: 1 to 3 (4, 5 possible)<sup>5</sup>.

Remarks: Plant remains from mosses like Sphagnum species, sedges, rushes and reeds are recognizable. Fibric horizons are quite common in bogs and oligotrophic parts of isolated fens. These horizons are mainly composed of remains of Sphagnum and Eriophorum species. In mesotrophic fens, the Hf-horizon is mainly composed of remains of sedges and rushes. Fibric horizons in eutrophic fens are less common because of the fast decomposition in those environments. A further differentiation could be made on the base of the origin of the plant material (oligotrophic mosses, mesotrophic sedges, mesotrophic sedges and reeds). This could be adapted to national, regional and local circumstances.

Note: this horizon coincides with what is classified as **fibric** (Klinka et al., 1981, Green et al., 1993) or **fibrist** (Delecour, 1980).

### Hfs (from Histic, between fibric and sapric)

Histic organic horizon consisting of half decomposed organic material not fitting the definition of fibric (Hf) or sapric (Hs). Fibric component 10% to 70%, sapric component 90% to 30% by volume (Figure 9). Content of rubbed fibres 10 to 40% of soil by dry weight (soil dried at  $105^{\circ}\text{C}$ ), Von Post scale of decomposition: 4 to 7 (8 possible)<sup>6</sup>.

Note: This horizon coincides with what is classified as **mesic** (Klinka et al., 1981, Green et al., 1993) or **hemist** layer in saturated soils;

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<sup>4</sup> The content (by mass) of the total organic fraction is generally more than 80%. When saturated, this fibric horizon can have a water content of far more than 850% of the oven-dry weight (Soil Taxonomy 1975).

<sup>5</sup> von Post scale: (1) Undecomposed; plant structure unaltered; yields only clear water coloured light yellow brown; (2) almost undecomposed; plant structure distinct; yields only clear water coloured light yellow brown; (3) very weakly decomposed; plant structure distinct; yields distinctly turbid brown water, no peat substance passes between the fingers, residue not mushy.

<sup>6</sup> von Post scale: (4) weakly decomposed; plant structure distinct; yields strongly turbid water, no peat substance escapes between the fingers, residue rather mushy; (5) Moderately decomposed; plant structure evident, but becoming indistinct; yields much turbid brown water, some peat escapes between the fingers, residue very mushy; (6) strongly decomposed; plant structure somewhat indistinct, but more evident in the squeezed residue than in the undisturbed peat; about one-third of the peat escapes between the fingers, residue strongly mushy; (7) Strongly decomposed; plant structure indistinct, but recognizable; about one-half of the peat escapes between the fingers.

**Hs (from Histic and sapric)**

Histic organic horizon in advanced stage of decomposition. Sapric content  $\geq$  70% of the horizon volume; fibric component less than 30% (Figure 9). Content of rubbed fibres < 10% of soil by dry weight (soil dried at 105 °C). Von Post scale of decomposition: 8 to 10<sup>7</sup>.

Remarks: Sapric horizons of brook valley systems and around wells have mostly a higher mineral fraction than those in fens or bogs. Although at first sight quite similar, the horizons can differ in structure, pH, nutrient content and base saturation due to differences in water quality, vegetation and soil organisms.

Note: This horizon coincides with what is classified as **humic** (Klinka et al., 1981, Green et al., 1993) or **saprist** (Delecour, 1980).

The following three horizons can be seen as special cases of the Hs horizon:

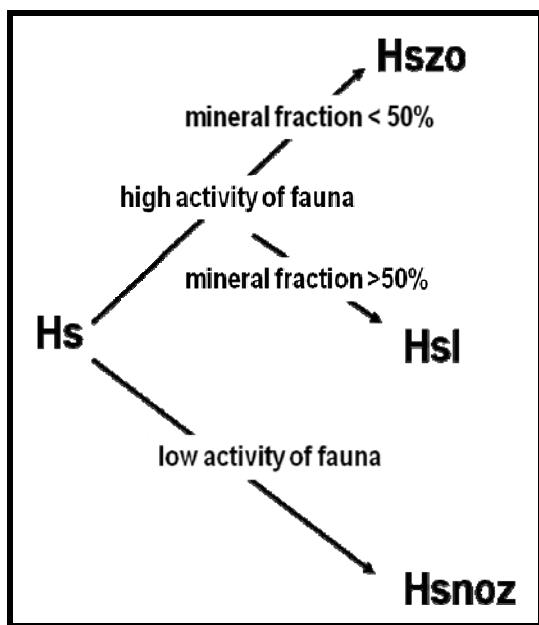
**Hszo** = Meso or macrostructured Hs horizon with a high activity of soil animals, especially earthworms. The mineral fraction is less than 50% (Figure 10). Typically present in drained semiterrestrial humus forms (both naturally and artificially drained). Activity of earthworms is high. The mineral fraction (clay, loam and/or sand) is commonly high compared to that of fibric horizons;

**Hsnoz** = Massive Hs horizon with low activity of soil animals. Common around bogs and rain fed ponds. Humification mainly results from the activity of microorganisms, which is typical of oligotrophic environments. Complexes of humic substances are acid and relatively poor in nutrients and bases and subject to eluviation when drained. The mineral fraction is variable;

**Hsl** = Hs horizon with a high percentage of mineral particles (clay, silt and sand). The mineral fraction is more than 50%. The mineral component may occur in the form of thin layers. The bioactivity is comparable to Hszo.

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<sup>7</sup> von Post scale: (8) very strongly decomposed; plant structure very indistinct; about two-thirds of the peat escapes between the fingers, residue almost entirely resistant remnants such as root fibres and wood; (9) Almost completely decomposed; plant structure almost unrecognizable; nearly all the peat escapes between the fingers; (10) Completely decomposed; plant structure unrecognizable; all the peat escapes between the fingers.



## 2.1.2 The mineral soil layer and its master horizons and layers

### 2.1.2.1 The A horizon

A mineral horizon formed at the surface or below an O horizon, in which all or much of the original structure of the parent material has been obliterated and characterized by one or more of the following:

An accumulation of humified organic matter intimately mixed with the mineral fraction and not displaying properties characteristic of E or B horizons (see below);

Properties resulting from cultivation, pasturing, or similar kinds of disturbance;

A morphology that is different from the underlying B or C horizon, resulting from processes related to its surface position.

If a surface horizon has properties of both A and E horizons but the dominant feature is an accumulation of humified organic matter, it is designated an A horizon.

Where the climate is warm and arid, the undisturbed surface horizon may be less dark than the underlying horizon and contains only small amounts of organic matter. It has a morphology distinct from the C layer, though the mineral fraction may be unaltered or only slightly altered by weathering; such a horizon is designated A because it is at the surface. Examples of surface horizons which may have a different structure or morphology due to surface processes are Vertisols, soils in pans or playas with little vegetation, and soils in deserts.

Recent alluvial, colluvial or aeolian deposits that retain fine stratification are not considered to be an A horizon unless cultivated.

The different diagnostic A horizons are identified in the field observing the soil mass by the naked eye or with 5-10X magnifying hand lens, assessing the structure (Soil Survey Manual (1993) and FAO Guidelines 2006) and consistence, and measuring the acidity (pH<sub>water</sub>).

Zanella et al. (2010) distinguishes the following five diagnostic A horizons:

**maA: biomacrostructured A horizon = A aneci-endovermic**

**General characteristic:** mixed biogenic organo-mineral peds dominate

**Diagnostic criteria:**

To be identified as a biomacrostructured A horizon, a layer must have at least four of the following:

structure grade, observable in place in undisturbed soil: never weak, never lack of structure;

presence of peds, observable in place in undisturbed soil as well as in the palm of the hand after applying a weak-moderate pressure on a sample of soil: all sizes of peds are present, but the volume of peds larger than 4 mm is greater than the volume of all other peds or units of soil;

structure (FAO and USDA) - grade: moderate or strong; size if granular shape: medium (2-5 mm) and/or coarser; size if subangular blocky shape: fine (5-10 mm) or fine (5-10 mm) and very fine (< 5 mm);

living earthworms, or earthworm galleries and/or casts;

earthworm galleries within underlying horizon;

pH in water >5.

**Origin:**

Biological: the whole horizon is made of anecic and endogeic earthworm faeces (the limit of 4 mm is rarely reached by droppings of arthropods and epigeic earthworms); roots and fungal hyphae (visible or not) also play an important role in the formation and stability of aggregates. Living earthworms or their galleries and casts are always present within the horizon.

**meA: biomesotstructured A horizon = A endo-epivermic**

**General characteristic:** composed of coloured organic (dark) or/and organo-mineral biogenic peds

**Diagnostic criteria:**

The biomesotstructured A horizon has all the following properties:

structure grade, observable in place in undisturbed soil: never weak, never lack of structure;

presence of peds, observable in place in undisturbed soil as well as in the palm of the hand after applying weak pressure on a sample of soil: all sizes of peds are present, but the volume of the peds larger than 1 mm and smaller than 4 mm is greater than the volume of all the other peds or parts of soil;

structure (FAO and USDA) - grade: moderate or strong (rarely weak); size if granular shape: fine (1-2 mm) and/or medium (2-5 mm); size if subangular blocky shape: very fine (<5 mm).

living earthworms, arthropods or enchytraeids or their droppings.

**Origin:**

Biological: earthworms (mostly epigeic and small endogeic), enchytraeids and arthropods are responsible for the structure; roots and fungal hyphae are also involved. Anecic and large endogeic earthworm droppings, classified typically as biomacro peds, are generally larger than 4 mm.

**NB.:** Green et al. (1993) described a rhizomull characterized by an A horizon which could be a biomesostructured A horizon. In this case, the thick mat of fine roots of grasses plays a major role in determining the type of structure of the topsoil.

### **miA: biomicrostructured A horizon = A enchy-arthropodic**

**General characteristic:** composed of fine mineral grains mixed with fine organic particles and dark-coloured biogenic peds (holorganic or hemiorganic)

**Diagnostic criteria:**

The biomicrostructured A horizon has at least five of the following properties:

absence of peds > 4 mm; observable both in situ, in undisturbed soil, and in the palm of the hand after applying a slight pressure on a sample of soil: peds of varying size can be present, but the volume of peds smaller than 1 mm is greater than the volume of all other peds or parts of the soil; gently squeezing the soil, almost all large peds easily reduce into smaller units;

structure (FAO and USDA) - grade: moderate, strong; shape: granular; size: very fine (< 1 mm);

presence of (generally uncoated) mineral grains (mineral component > 10%);

> 10% organic particles and dark-coloured biogenic peds (holorganic or hemiorganic = humic component)

living arthropods, enchytraeids or their droppings;

pH in water <5.

**Origin:**

**Biological:** the horizon has an important amount of faecal pellets, droppings of enchytraeids (potworms) (larval stages, insects, spiders, mites, springtails...), micro-arthropods and particles of organic matter (remains of decomposed litter). Hyphae and roots are also very common.

**Field identification:**

Take a sample of A horizon rich organic soil material. If squeezed gently in the palm of the hand, the sample breaks up into units composed of organic and organo-mineral peds (single or bound droppings, droppings bound to mineral grains), organic particles and mineral grains. Observing the soil with a magnifying hand lens (5-10X) reveals a lot of complex holorganic and hemiorganic peds. Their mean size is less than 1 mm but the structure of the soil is clearly expressed and never weak or absent. The appearance can be very similar to that of the OH horizon.

Observed on sandy or loamy substrate (acid or non calcareous). The large amount of quartz grains (> 50%) seems to prevent the formation of a larger size structure or a massive one.

### **sgA: single grain A horizon**

**General characteristic:** single grained structure, biological aggregation absent or involving less than 5% of the soil volume

**Diagnostic criteria:**

To be identified as a single grain A horizon, a layer must have at least four of the following:

an unbound loose consistence (undisturbed soil mass);

structure (FAO and USDA): single grain;  
presence of clean (= uncoated) mineral grains;  
<10% of fine organic particles and/or dark-coloured biogenic (holorganic or hemorganic) peds;  
pH in water < 5.

**Origin:** Mineral grains coated with organic matter indicate a process of podzolisation in places. Faecal pellets of micro-arthropods or enchytraeids are sometimes present but irrelevant (< 10%).

**Field identification for structure and consistency:**

In undisturbed soil - Structure: single grain. Sub-units of soil do not appear bound together or are weakly bound in a casual manner. Sometimes, in a relatively organic sample, very small peds (< 1 mm) are detectable in the mass (animal pellets), because of their dark colour and organic composition in a light mineral mass. At other times the horizon looks like a brownish-red coloured nearly uniform fluffy mass. In this case, it is very difficult to separate mineral from scarcely present organic components.

In a sample of soil in the palm of the hand - When squeezing gently with the fingers, the sample breaks up progressively into large then fine, artificial units. The fine units are mostly mineral, more or less coloured by organic matter in coatings. Animal pellets are absent or in traces (less than 10%). The sample could be wrongly classified as weak medium granular structured but grains, never zoogenic, break easily into micro units because they are very weakly attached together in variable manner and size (no apparent soil structure).

**Horizon designation:** Because of observable processes of eluviation or podzolisation, the horizon could be classified as EA (or E) or AB following its similarity to mineral horizons.

**msA: massive A horizon**

**General characteristic:** massive structure, biological aggregation absent or involving less than 5% of the soil volume

**Diagnostic criteria:**

To be identified as a massive A horizon, a layer must have at least three of the following:

heterogeneous but one-piece matrix;  
structure (FAO and USDA): massive;  
presence of clean (= uncoated) mineral grains;  
pH in water < 5.

**Origin:** Presence of mineral grains coloured by organic matter in coatings. Cohesion forces among parts of soil seem equally distributed in the soil, as they depend mostly on physical or chemical conditions rather than biological aggregation (peds originated by animals < 5%). Past biological activity (incorporation of organic matter) could also be involved in the process of formation of the horizon, but traces of current biological activity are never visible. Organic or hemorganic pellets of microarthropods or enchytraeids are present (< 5% of the soil volume). A 5-10 X magnifying hand lens is necessary to detect the composition of the pellets or grains, the size of the most common biostructured units being less than 1 mm.

**Field identification for structure and consistency:**

In undisturbed soil - Structure: massive. The units of soil are bound together in a relatively compact manner. No planes or zones of weakness are detectable in the mass, which appears as a heterogeneous coloured layer of organo-mineral soil.

If the soil is dry, when applying a moderate to strong pressure with the fingers, the soil sample progressively breaks up into finer artificial units. These fine units have a varying composition: mineral, organo-mineral and organic. If the soil is moist, the shape of the sample can be modified as in a tender, plastic, non-elastic matter.

**Horizon designation:** Because of observable processes of eluviation or initial podzolisation, the horizon could be classified as AE (or EA), following its resemblance to a mineral E horizon.

**Zoogenic and non zoogenic A horizon**

**Azo** = zoogenic A horizon.

**Azo** = maA (implied maAzo) or meA (meAzo) or miA (miAzo).

**Anoz** = A horizon considered as non zoogenic. To the naked eye, or with the help of a hand lens, this horizon does not show relevant (mass) signs of animal activity (absence of galleries; droppings, mucus, animal remains etc., < 5% of the soil volume). Zoological agents are not involved in the soil aggregation. Fungal structures can be visible.

**Anoz** = sgA (implied sgAnoz) or msA (msAnoz).

**2.1.2.2 E horizon:**

A mineral horizon in which the main feature is loss of silicate clay, iron, aluminium, or some combination of these, leaving a concentration of sand and silt particles, and in which all or much of the original structure of the parent material has been obliterated.

An E horizon is usually, but not necessarily, lighter in colour than an underlying B horizon. In some soils, the colour is that of the sand and silt particles but, in many soils, coatings of iron oxides or other compounds mask the colour of the primary particles. An E horizon is most commonly differentiated from an underlying B horizon in the same soil profile by colour of higher value or lower chroma, or both; by coarser texture; or by a combination of these properties. An E horizon is commonly near the surface, below an O or A horizon and above a B horizon, but the symbol E may be used without regard to position in the profile for any horizon that meets the requirements and that has resulted from soil processes.

**2.1.2.3 B horizon:**

A horizon formed below an A, E, H or O horizon, and in which the dominant features are the obliteration of all or much of the original structure of the parent material, together with one or a combination of the following:

Illuvial concentration of clay, iron, aluminium, humus, carbonates, gypsum, silica or some combination of these;

Evidence of removal of carbonates;

Residual concentration of iron and aluminium oxides;

Coatings of humus and/or oxides that make the horizon conspicuously lower in value, higher in chroma, or redder in hue than overlying and underlying horizons;

Alteration that forms silicate clay or liberates oxides or both, and that forms a granular, blocky, or prismatic structure if volume changes accompany changes in moisture content; Brittle consistency.

All kinds of B horizons are, or were originally, subsurface horizons. Included as B horizons are layers of illuvial concentration of carbonates, gypsum, or silica (these horizons may or may not be cemented) and brittle horizons that have other evidence of alteration, such as prismatic structure or illuvial accumulation of clay.

Examples of layers that are not B horizons are layers in which clay films either coat rock fragments or are on finely stratified unconsolidated sediments, whether the films were formed in place or by illuviation; layers into which carbonates have been illuviated but that are not contiguous with an overlying pedogenetic horizon; and layers with gley colours but no other pedogenetic changes.

#### **2.1.2.4 C horizon:**

A horizon, excluding hard bedrock, that is little affected by pedogenetic processes (lacks properties of H, O, A, E, or B horizon). The material of C layers may be either like or unlike that from which the soil is presumed to have formed. A C layer may have been modified even if there is no evidence of pedogenesis. Plant roots can penetrate C layers, which provide an important growing medium.

Included as C layers are sediments, saprolite, and unlithified geological materials that, commonly, slake within 24 hours when air-dry chunks are placed in water and, when moist, can be dug with a spade. Some soils form in material that is already highly weathered; such material that does not meet the requirements of A, E or B horizons is designated C. Changes not considered pedogenetic are those not related to overlying horizons. Layers having accumulations of silica, carbonates, or gypsum, even if indurated, may be included in C layers, unless the layer is obviously affected by pedogenetic processes; then it is a B horizon.

#### **2.1.2.5 R layer:**

Hard bedrock underlying the soil. Granite, basalt, quartzite and indurated limestone or sandstone are examples of bedrock that are designated R. Air-dry or drier bits of an R layer, when placed in water, will not slake within 24 hours and are resistant to pressure with the fingers. The R layer is sufficiently coherent when moist to make digging with a spade impractical, although it may be chipped or scraped. Some R layers can be ripped with heavy power equipment. The bedrock may be fissured, but few roots can penetrate. The cracks may be coated or filled with clay or other material.

#### **2.1.2.6 I layer:**

Ice lenses and wedges that contain at least 75% ice (by volume) and that distinctly separate organic or mineral layers in the soil.

In areas affected by permafrost, ice bodies may form lenses of wedges that separate entire soil layers. Where such ice concentrations occur within the depth of soil description, they can be designated as I layer.

## 2.2 Transitional horizons

There are two kinds of transitional horizons: those with properties of two horizons superimposed and those with the two properties separate.

For horizons dominated by properties of one master horizon but having subordinate properties of another, two capital letter symbols are used, such as AB, EB, BE and BC. The master horizon symbol that is given first designates the dominant properties: an AB horizon, for example, has characteristics of both an overlying A horizon and an underlying B horizon, but is more like the A than like the B.

In some cases, a horizon can be designated as transitional even if one of the master horizons to which it is apparently transitional is not present. A BE horizon may be recognized in a truncated soil if its properties are similar to those of a BE horizon in a soil in which the overlying E horizon has not been removed. An AB or a BA horizon may be recognized where bedrock underlies the transitional horizon. A BC horizon may be recognized even if no underlying C horizon is present; it is transitional to assumed parent material. A CR horizon can be used for weathered bedrock which can be dug with a spade though roots cannot penetrate except along fracture planes.

Horizons or layers in which distinct parts have recognizable properties of two kinds of master horizons are indicated as above, but the two capital letters are separated by a stroke (/), as E/B, B/E, B/C or C/R. Commonly, most of the individual parts of one component are surrounded by the other material.

## 2.3 Subordinate characteristics within master horizons and layers

Designations of subordinate distinctions and features within the master horizons and layers are based on characteristics observable in the field. Lower case letters are used as suffixes to designate specific kinds of master horizons and layers, and other features. The list of symbols and terms is explained more in detail below:

Suffix	Description	Use for
a	Evidence of cryoturbation: Irregular or broken boundaries, sorted rock fragments (patterned ground), or organic matter in the lower boundary between the active layer and permafrost layer.	No restriction
b	Buried horizon: Used in mineral soils to indicate identifiable buried horizons with characteristics that were formed before burial. Horizons may or may not have developed in the overlying materials which may be either like, or unlike, the assumed parent material of the buried soil. The symbol is not used in organic soils or to separate an organic layer from a mineral layer, in cryoturbated soils, or with C layers.	Mineral horizon, not cryoturbated
c	Concretions or nodules: In mineral soil it indicates a significant accumulation of concretions or of nodules. The nature and consistence of the nodules is specified by other suffixes and in the horizon description.	Mineral horizon
d	Dense layer: Used in mineral soils to indicate a layer of relatively unaltered, mostly earthy material that is not cemented but that has such bulk density or internal organization that roots cannot enter except in cracks; the symbol is not used in combination with the symbols m (cementation) and x (fragipan).	Mineral horizon
f	Frozen soil: Designates a horizon or layer that contains permanent ice or is perennially colder than 0°C. It is not used for seasonally frozen layers or for bedrock (R). Dry frozen soil layers may be labelled (f).	Not in I and R horizons
g	Stagnic conditions: Designates a horizon with a distinct pattern of mottling that reflects alternating conditions of oxidation and reduction of sesquioxides, caused by seasonal surface waterlogging. If aggregates	No restriction

	are present, the interiors of the aggregates show oxidising colours and the surface parts reducing colours.	
<b>h</b>	Accumulation of organic matter: Designates the accumulation of organic matter in a mineral horizon. The accumulation may occur in a surface horizon or in subsurface horizons (through illuviation).	Mineral horizon
<b>i</b>	Slickensides: In mineral soils, denotes the occurrence of slickensides, i.e. oblique shear faces caused by the shrink-swell action of clay; wedge-shaped polished pedes and seasonal surface cracks are commonly present.	No restriction
<b>j</b>	Jarosite: Indicates the presence of jarosite (straw-yellow) mottles, coatings or hypodermic coatings.	No restriction
<b>k</b>	Accumulation of pedogenetic carbonates: Indicates an accumulation of alkaline earth carbonates, commonly calcium carbonate.	No restriction
<b>l</b>	Capillary fringe mottling: Indicates mottling caused by ascending groundwater. If aggregates are present, the interiors of the aggregates show reducing colours and the surface parts oxidising colours.	No restriction
<b>m</b>	Strong cementation or induration: In mineral soils, indicates continuous or nearly continuous cementation - used only for horizons that are more than 90 % cemented, though they may be fractured. The layer restricts rooting to fracture planes. The single predominant or co-dominant cementing agent may be indicated using defined letter suffixes single or in pairs. If the horizon is cemented by carbonates km is used; by silica, qm; by iron, sm; by gypsum, ym; by both lime and silica, kqm; by salts more soluble than gypsum, zm.	Mineral horizons
<b>n</b>	Pedogenetic accumulation of exchangeable sodium.	No restriction
<b>o</b>	Residual accumulation of iron/aluminium oxides: Indicates residual accumulation of sesquioxides, as opposed to the symbol s, which indicates illuvial accumulation of oxides or organic and oxide mixture.	No restriction
<b>p</b>	Ploughing or other artificial disturbance: Indicates mixing of the surface layer by ploughing or other tillage practices. A disturbed organic horizon is designated Op or Hp. A disturbed mineral horizon, even though clearly originally an E, B or C, is designated Ap.	No restriction; E, B or C as Ap
<b>q</b>	Accumulation of pedogenetic silica: If silica cements the layer and cementation is continuous or nearly continuous, qm is used.	No restriction
<b>r</b>	Strong reduction: Indicates presence of iron in reduced state. If r is used with B, pedogenetic change in addition to reduction is implied; if no other change has taken place, the horizon is designated Cr.	No restriction
<b>s</b>	Illuvial accumulation of iron/aluminium oxides: Used with B to indicate the accumulation of illuvial, amorphous, dispersible organic matter-oxide complexes if the value and chroma of the horizon are more than 3. The symbol is also used in combination with h as Bhs if both the organic matter and oxide components are significant and both value and chroma are approximately 3 or less.	B horizon
<b>t</b>	Accumulation of clay: Used with B or C to indicate an accumulation of clay that either has formed in the horizon or has been moved into it by illuviation, or both. At least some part should show evidence of clay accumulation in the form of coatings on ped surfaces or in pores, as lamellae, or as bridges between mineral grains.	B and C horizon
<b>u</b>	Urban and other man-made materials: Used to indicate the dominant presence of man-made materials.	H, O, A, E, B and C horizons
<b>v</b>	Plinthite: Indicates the presence of iron-rich, humus-poor material that is firm or very firm when moist and that hardens irreversibly when exposed to the atmosphere. When hardened, it is no longer called plinthite but a hardpan, ironstone, a petroferric or a skeletic phase – in which case v is used in combination with m.	No restriction
<b>w</b>	Development of colour or structure in B: Indicates development of colour or structure, or both, in B horizons lacking other diagnostic	B horizons

	characteristics. It is not used to indicate a transitional horizon.	
x	Fragipan: Brittle consistency or high bulk density attributed to pedogenetic processes.	No restriction
y	Pedogenetic accumulation of gypsum.	No restriction
z	Pedogenetic accumulation of salts more soluble than gypsum.	No restriction

### Conventions for using letter suffixes

Many master horizons and layers that are symbolized by a single capital letter will have one or more lowercase letter suffixes. More than three suffixes is cumbersome. The following rules apply:

Letter suffixes should immediately follow the capital letter;

A B horizon that has significant accumulation of clay and also shows evidence of development of colour or structure, or both, is designated Bt (t has precedence over w, s and h);

Suffixes are listed alphabetically.

## 2.4 Vertical subdivisions

A horizon or layer designated by a single combination of letter symbols can be subdivided using arabic numerals following the letters. Within a C, for example, successive layers could be C1, C2, C3, etc.; or if the lower part is gleyed and the upper part is not, the designations could be C1-C2-Cg1-Cg2 or C-Cg1-Cg2-R.

These conventions apply whatever the purpose of subdivision. A horizon identified by a single set of letter symbols may be subdivided on the basis of morphology, such as structure, colour, or texture. These subdivisions are numbered consecutively. The numbering restarts with 1 at whatever level in the profile. Thus Bt1-Bt2-Btk1-Btk2 is used, not Bt1-Bt2-Btk3-Btk4.

The numbering of vertical subdivisions within a horizon is not interrupted at a discontinuity (indicated by a numerical prefix) if the same letter combination is used in both materials: Bs1-Bs2-2Bs3-2Bs4 is used, not Bs1-Bs2-2Bs1-2Bs2. A and E horizons can be subdivided similarly, for example Ap1, A1, A2, Ap2, A3; and E1, E2, Eg1, Eg2.

## 2.5 Discontinuities

In mineral soils, arabic numerals are used as prefixes to indicate discontinuities. Wherever needed, they are used preceding A, E, B, C and R. They are not used with I, although this symbol clearly indicates a discontinuity. These prefixes are distinct from arabic numerals used as suffixes to denote vertical subdivisions.

A discontinuity is a significant change in particle size distribution or mineralogy that indicates a difference in the material from which the horizons formed or a significant difference in age, or both -unless that difference in age is indicated by the suffix b. Symbols to identify discontinuities are used only when they will contribute substantially to the reader's understanding of relationships among horizons. The stratification common in soils formed in alluvium is not designated as discontinuities - unless particle size distribution differs markedly from layer to layer - even though genetic horizons have formed in the contrasting layers.

Where a soil has formed entirely in one kind of material, no prefix is used (the whole profile is material 1). Similarly, the uppermost material in a profile having two or more contrasting materials is understood to be material 1, but the number is omitted. Numbering starts with the second layer of contrasting material, which is designated 2. Underlying contrasting layers are

numbered consecutively. Even though a layer below material 2 is similar to material 1, it is designated 3 in the sequence. The numbers indicate a change in the material, not the type of material. Where two or more consecutive horizons formed in one kind of material, the same prefix number applies to all of the horizon designations in that material, e.g. Ap-E-Bt1-2Bt2-2Bt3-2BC. The number suffixes designating subdivisions of the Bt horizon continue in consecutive order across the discontinuity.

If an R layer is below a soil that formed in residuum and the material of the R layer is judged to be like that from which the material of the soil weathered, the arabic number prefix is not used. If the R layer would not produce material like that in the solum, the number prefix is used, as in A-Bt-C-2R or A-Bt-2R. If part of the solum formed in residuum, R is given the appropriate prefix: Ap-Bt1-2Bt2-2Bt3-2C1-2C2-2R.

In organic soils, discontinuities between different kinds of layers are not identified. In most cases the differences are shown by the letter suffix designations, if the different layers are organic, or by the master symbol if the different layers are mineral.