S Protocol

SOILS

Version 1.1 **(updated Dec 2001)**

Aim

To classify the soils at ECN sites, to characterise and quantify their physical, chemical and mineralogical properties and to quantify temporal changes in those properties

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Rationale

Patterns of soil and vegetation are closely interlinked in areas of natural and semi-natural vegetation, the pattern of vegetation and the distribution of individual plant species both reflecting and influencing the complex interaction of the soil's chemical, physical and biological properties. Changes in soil characteristics may result in changes in vegetation, and *vice versa*. The link between soils and surface vegetation patterns is largely broken where land is under intensive agricultural management but the inherent soil properties are nevertheless major factors influencing the choice of crops and the type of land management.

Soils also exert a strong influence on surface water chemistry and flow regimes, on gas exchange between the atmosphere and the earth's surface, and on the fate of deposited pollutants. Changes in soils take place naturally over time but can also be driven by alterations in pollution climate and land management.

The soil map compiled at the start of the programme and the associated characterisation of the soils comprising the various map units provide the baseline against which changes over time can be assessed. A complete characterisation of a soil requires information on physical, chemical, mineralogical and biological attributes which change at different rates in response to changes in, for example, climate, pollution or land use. Thus, soil chemistry tends to respond more rapidly than do physical characteristics such as texture and structure. Exchangeable soil chemistry, comprising the fraction most readily available for plant uptake and loss to drainage waters, responds more readily than total soil chemistry. Similarly, various aspects of soil biological properties change at different rates. Sampling intervals and their associated analyses have been planned to take account of these differences.

Method

Soil survey and classification (SB)

An extensive survey of the whole of the ECN site is carried out, using a soil auger and spade, to provide maps at a scale of 1:10 000 for sites up to 50 km^2 and 1:25 000 for sites over 50 km^2 ; map units will be identified to soil series level, or complexes where necessary (Avery 1980), but will also be classified according to the systems of the Food and Agriculture organisation and of the US Department of Agriculture (FAO-UNESCO 1974; FAO 1986; USDA 1975). Where site soil maps already exist, these will be evaluated and the necessity for additional survey work will be assessed by the appropriate Soil Survey organisation.

An intensive survey will be carried out on a 300 m x 300 m area, with the TSS at its centre, using auger borings at 50 m grid intersections over the whole 9 ha area and at 25 m grid intersections within the TSS (Figure 6). Where possible, the TSS is oriented towards north (see LM Protocol).

Method

Soil characterisation and assessment of change (SF and SC)

Sampling frequency and location

Soil sampling will be carried out at the beginning of the programme. The soils are subsequently re-sampled at five-yearly and 20-yearly intervals, using different sets of determinands over the two periods. Both periodic samples are replicated in six blocks of which four are located adjacent to the sides of the TSS and two are close to its centre. The side blocks may be square and located

immediately outside the TSS as suggested in Figure 7, or linear and located immediately inside the TSS as suggested in Figure 8; the choice will depend on the particular conditions attaching to the TSS at a site. The arrangement suggested in Figure 7 can be used where the TSS has fragile vegetation and where it is important to avoid damage; the arrangement suggested in Figure 8 may be used where the availability of space outside the TSS is very limited. Where neither design is thought suitable for local circumstances any proposed alternative must be approved by the ECN Central Co-ordination Unit.

Each sampling block is referenced alphabetically as shown in Figures 7 and 8, the most northerly block being designated 'A' and the others logically in a clockwise direction. The corners of each block should be marked permanently with wooden posts or stone blocks and recorded on a map. Each sampling block is itself divided into 5 m x 5 m cells, of which some are used for five-yearly sampling and others for 20-yearly sampling, as shown in Figures 7 and 8. Each cell used for five-yearly sampling is assigned a number from 1 to 16; each cell used for 20-yearly sampling is assigned an identifying letter from A to I (or A-P in the case of linear blocks), as shown in Figures 7 and 8. The corners of the cells should be marked temporarily by canes for convenience in locating current sampling positions. The location of each cell used on any sampling occasion is recorded on a plan.

Sampling - initial and five-yearly (SF)

Five-yearly sampling is carried out in each of the 16, numbered, 5 m x 5 m cells in each of the six blocks. Each 5 m x 5 m cell is subdivided into 25 subcells of 1 m x 1 m which are numbered as shown in Figure 9, with the cell orientated with the TSS. On each sampling occasion only one subcell is randomly selected from each 5 m x 5 m cell, giving a total of 16 sampling sites for each block at each five-yearly sampling. For convenience it is necessary to locate only each subcell which is to be used for sampling; the centre of the subcell should be marked with a cane. Soil sampling will be carried out within a small area around this central point. In subsequent five-yearly samples, different 1 m x 1 m subcells are selected, and thus no subcell is sampled more than once during the ECN project. The location of the subcell to be used for sampling in each block and cell will be recorded.

Sampling should be carried out using a gouge auger of a diameter suitable to provide sufficient bulked sample (about 3 kg for mineral soil and at least 6 kg for organic soil, depending on water content) for the soil type being sampled. For slightly stony soils, a narrower auger may be preferred. For some stony soils or soils with very thin horizons, it will be necessary to excavate a small inspection pit with a spade and sample from the exposed face.

Two sets of soil samples will be taken to a maximum depth of 30 cm from each sampled subcell. One set is based on depths: 0-5 cm, 5-10 cm, 10-20 cm, and 20-30 cm. The other set corresponds to horizons within the top 30 cm. It should be noted that soil horizons within the upper 30 cm can be difficult to identify clearly when using a soil auger. Horizon boundaries often merge over a depth of several centimetres and this gradation from one horizon to another may be thicker than the actual horizons. In practice, a soil layer should be designated a horizon when organic matter content or colour show a change.

The 16 subsamples by horizon and 16 subsamples by depth from each block should be bulked for each block. In some soil types with thin horizons, it may be necessary to take several auger samples within each subcell to ensure that sufficient soil is obtained.

Sampling - initial and 20-yearly (SC)

S Protocol

Profile sampling and description require excavation of the ground to expose a vertical section of soil suitable for description. Sampling and description will use

standard methods (Hodgson 1974) and will be from six pits, each located in an alphabetically labelled 5 m x 5 m cell (Figures 7 & 8) chosen at random from each block. A modified version of the Von Post scale (Avery 1980), together with the vegetation composition scheme of Troels-Smith (1955), will be used for describing the decomposition state of peats.

Profile description and sampling can be carried out at most times of the year, but preferably when the soil is at, or fairly close to, field capacity. Orientation of the profile face should be in the direction of greatest sunlight if possible but space limitations in the area of the sampling cell may preclude this. During excavation topsoil should be kept separate from subsoil and care should be taken to avoid contaminating the surface of adjoining sample cells when using the design suggested in Figure 8. It is advisable to lay a polythene sheet upon which to place the spoil; this allows a tidier re-instatement. The excavation should expose a soil profile face of suitable size and of sufficient dimensions to allow easy working. A soil profile should be exposed to 1.2 m if possible. **It should be noted that this is the maximum depth allowed by the Health and Safety Executive before shoring of the excavation sides is considered necessary.**

Using a trowel, knife or similar instrument, the profile face should be picked back to expose the soil structure from the surface downwards. This will allow identification of the soil horizons from their colour, texture and structural development and, by using a 10% solution of hydrochloric acid, the presence of excess calcium carbonate. The depth of each horizon, measured from the soil surface, should be recorded together with the location of the site, the soil surface description and the description of each horizon, following the procedures presented in the Soil Survey field handbook (Hodgson 1974). It may also be useful to record any other observations of interest which are not specified in the handbook. The description and characteristics of the site and of each horizon are recorded either by tape-recorder or in a notebook for subsequent transfer to computer and hard copy.

Samples should be collected from each soil horizon recognised in the description to about 1 m depth (or less if rock is encountered) and by standard depths of 0-5 cm, 5-10 cm, 10-20 cm, 20-40 cm, 40-60 cm, 60-80 cm, 80-100 cm, and 100-120 cm. The positions of the depth bands relative to the horizons should be recorded. Each horizon should be sampled from its full depth beginning with the lowest (deepest) so as to avoid contamination of other, higher horizons; 3-4 kg of soil should be collected from each horizon and from each depth band in each soil pit. Samples should be collected into plastic bags with the sample bag then placed within a second plastic bag and a label placed between the two bags. All samples should be stored at 4-6°C prior to drying and analysis. Samples should be stored for as short a time as possible to minimise the effect of biological activity within the cores.

In addition, core samples will be taken in triplicate from each horizon, using the methods given in Hodgson (1976), for the measurement of soil water release characteristics and of bulk density. It may be possible to take cores from depths which correspond with the fixed depth samples.

The location of each sampled cell should be marked, preferably both on the ground and on a plan.

Please note that *both* SF and SC are carried out in years 20, 40, etc.

Sample handling and storage

Soil samples should be stored in clearly marked polythene bags and kept in the dark at 4-6°C prior to drying and analysis. Grinding facilities should satisfy criteria set by the soil analysis subgroup. All bulk samples will be air-dried and sieved at 2 mm prior to analysis, except where a sample is required for analysis

in the moist state, in which case a moist subsample will be taken by an accepted method. Chemical analyses are carried out on the fine earth unless otherwise stated.

Particle size analysis

Particle size analysis should be carried out on each horizon of the profile samples collected in the initial sampling (see SC above) from the six profile pits and using the pipette/peroxidised soil method as defined in the Soil Survey laboratory handbook (Avery & Bascomb 1982) and which complies with international particle size analysis standards.

Soil mineralogy

Analyses should be on clay (<2 µm), silt (2-63 µm), and coarse silt and sand (63 µm-2 mm) fractions from each of the bulked profile horizon samples taken from the initial sampling (see SC above), using a combination of x-ray diffraction and optical microscopy techniques. Analysis of the heavy mineral fraction (SG >2.65) should also be carried out. Only clay mineral analysis will be repeated at 20-year intervals.

Soil chemistry

Each bulked horizon and depth band sample from the five-yearly (*) core samples and each horizon and depth band from the 20-yearly (†) profile samples will be analysed for:

*†	Moisture	on <2 mm soil oven dried overnight at 105°C
*†	pH	on field moist and air dry samples, on 1: 2.5 extracts in water and 0.01 M calcium chloride

Exchangeable

*†	acidity	0.5 M barium acetate pH 7
*†	sodium	1 M ammonium acetate pH 7 and unbuffered
*†	potassium	1 M ammonium acetate pH 7 and unbuffered
*†	calcium	1 M ammonium acetate pH 7 and unbuffered
*†	magnesium	1 M ammonium acetate pH 7 and unbuffered
*†	manganese	1 M ammonium acetate pH 7 and unbuffered
*†	aluminium	1 M potassium chloride pH 7

Total

*†	nitrogen	Kjeldahl digestion
*†	phosphorus	NaOH fusion (Smith & Bain 1982)
*†	sulphur	(to be decided)
*†	organic carbon	dichromate digest
*†	inorganic carbonate	manometric
†	lead	aqua regia
†	zinc	aqua regia
†	cadmium	aqua regia
†	copper	aqua regia
†	mercury	nitric acid/sulphuric acid digest
†	cobalt	aqua regia
†	molybdenum	aqua regia
†	arsenic	$Mg(NO_3)_2$ ashing, sulphuric acid/citrate extraction
†	chromium	aqua regia
†	nickel	aqua regia

Extractable

†	iron	ammonium oxalate pH 3
†	aluminium	ammonium oxalate pH 3
†	phosphorus	0.5 M sodium hydrogen carbonate

Bulk density and water release characteristics

These should be determined for each major soil horizon sampled as part of the 20-year sampling (see SC above). The method will be the Soil Survey suction table and pressure system (in triplicate) procedures in Hodgson (1976). Bulk density measurements will be carried out in conjunction with water release measurements.

Archiving of soil samples (SA)

Soil preparation before storage

The field samples must be allocated a unique ECN site, sample number and date for reference purposes. The sample is spread out thinly on a tray and dried at 25°C. Drying may take up to five days for a mineral soil and up to 30 days for a highly organic soil or peat, depending on its water content. The sample is then crushed or rolled to break up any aggregates and sieved through a 2 mm sieve. Highly organic samples are ground in a cross-beater mill. The >2 mm material which is retained after sieving generally consists of stones, roots or other organic matter and is discarded. Before any form of analysis, the <2 mm soil is homogenised by a rotary sample divider (eg Fritsch Laborrette 27) which produces eight uniform subsamples. This ensures that the analysed samples and those stored for future work are exactly similar.

Storage

The sieved, homogenised soils are stored in high-density plastic bottles or food-quality polycarbonate bags. Samples stored in bottles should have the sample number shown on the lid and on the container itself. Samples stored in bags should be placed inside a second, similar bag; the outer bag should carry the sample number, and the sample number should also be written on a tag placed between the inner and outer bag. Subsamples of at least 500 g or 100 g for highly organic soils, are stored in glass jars with sealed lids, and labelled.

Authors

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United States Department of Agriculture. 1975. *United States Soil Conservation Service soil taxonomy: a basic system of soil classification for making and interpreting soil surveys.* (Agriculture handbook no. 436.) Washington, DC: US Government Printing Office.

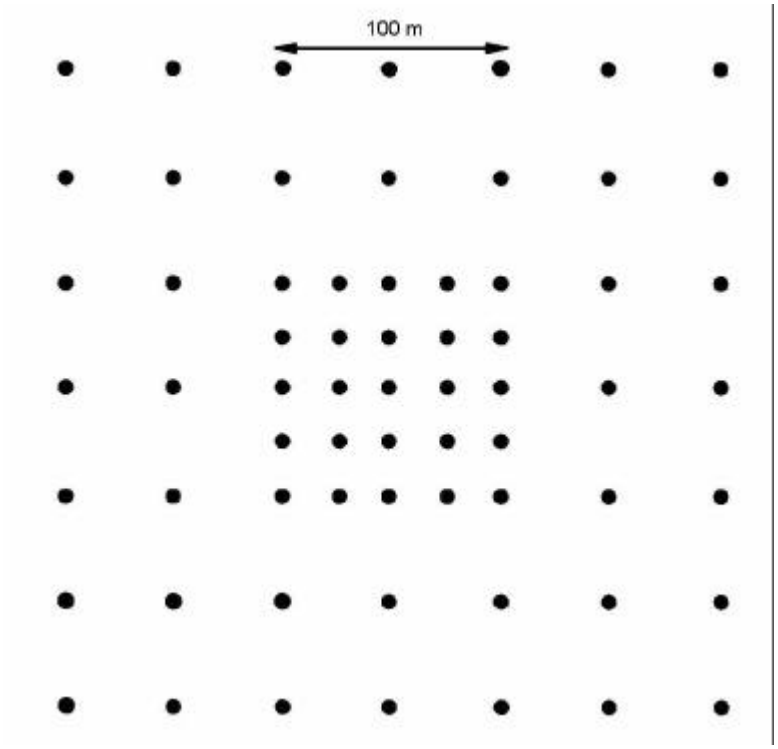

Figure 6. Distribution of auger bores for intensive soil survey of TSS and surrounding area

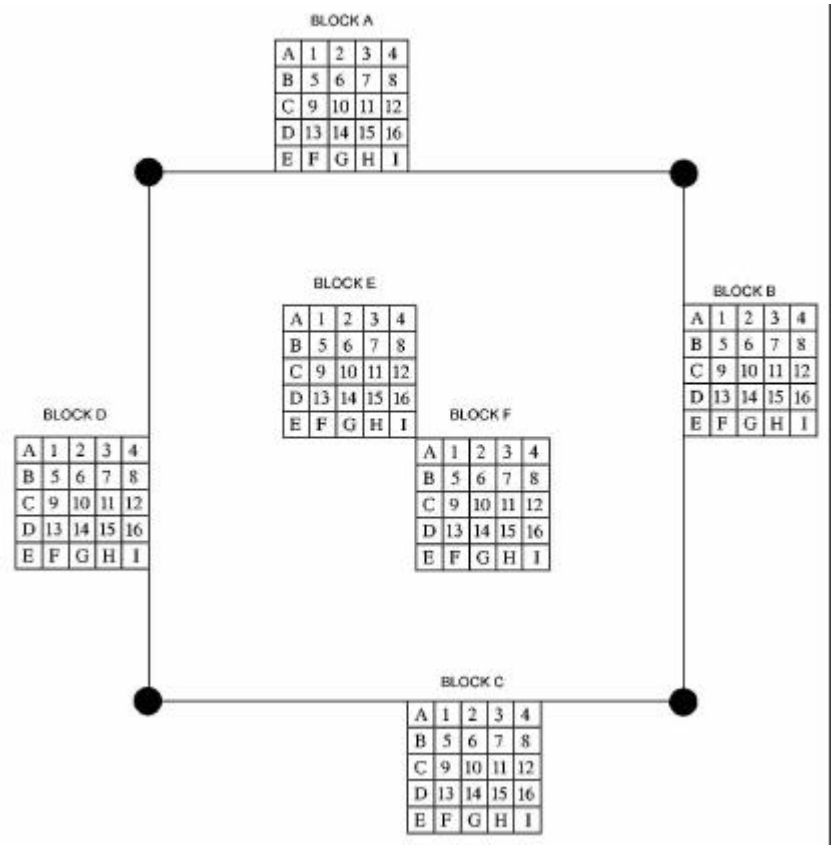

Figure 7. Suggested layout of soil sampling blocks in the TSS

Cells A - I in blocks A - F are used for 20-year sampling, each cell being 5m x 5m. On each sampling occasion one cell is selected at random from each block, resulting in a total of 6 sets of samples (one set from each block) on each sampling occasion.
Cells 1 - 16 in blocks A - F are used for five-year sampling, each cell being 5m x 5m. On each sampling occasion one sub-cell (1m x 1m) from each cell is selected at random from each block. The 16 samples from each block are bulked, resulting in a total of 6 sets of samples (one set from each block) on each sampling occasion.

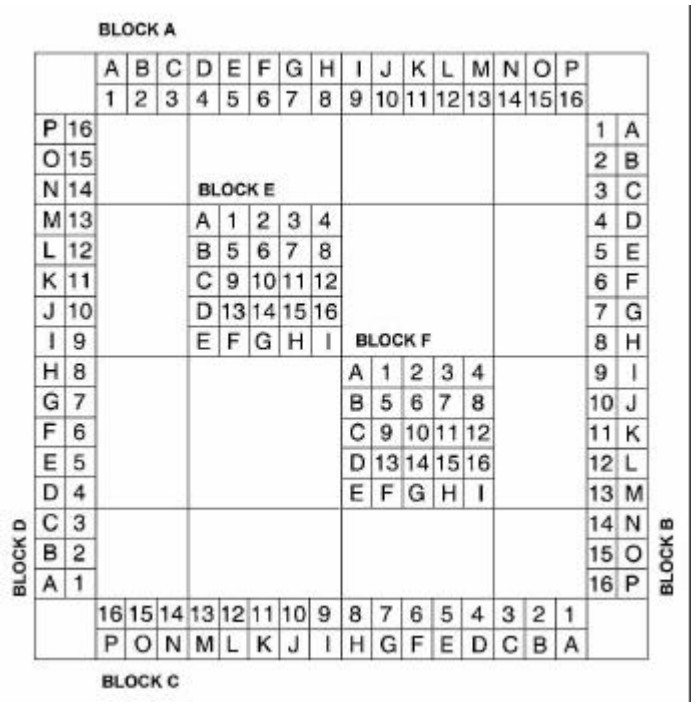

Figure 8. Alternative layout of soil sampling blocks in the TSS

Cells A - P in blocks A - D and cells A - I in blocks E and F are used for 20-year sampling, each cell being 5m x 5m. On each sampling occasion one cell is selected at random from each side block (blocks A - D) and from each inside block (blocks E and F), to give a total of 6 sets of samples (one set from each block) on each sampling occasion.

Cells 1 - 16 in blocks A - F are used for five-year sampling, each cell being 5m x 5m. On each sampling occasion one sub-cell (1m x 1m) from each cell is selected at random from each block. The 16 samples from each block are bulked to give a total of six sets of samples (one set from each block) on each sampling occasion.

1	2	3	4	5
6	7	8	9	10
11	12	13	14	15
16	17	18	19	20
21	22	23	24	25

Figure 9. Numbering the 25 1 m x 1m subcells for random selection of five-yearly samples

Specification of results and recording conventions

The measurement variables listed below are those required for each S sampling location at an ECN Site. Sites submitting data to the ECNCCU should refer to the accompanying Data Transfer documentation for the specification of ECN dataset formats, available on the restricted access Site Managers' extranet. Contact ecnccu@ceh.ac.uk if you need access to this documentation.

The first 4 key parameters uniquely identify a sample or recording occasion in space and time, and must be included within all datasets:

• Site Identification Code (e.g. T05)	Unique code for each ECN Site
• Core Measurement Code (e.g. PC)	Unique code for each ECN 'core measurement'
• Location Code (e.g. 01)	Each ECN Site allocates its own code to replicate sampling locations for each core measurement (e.g. for different surface water collection points)
• Sampling Date (/time)	Date on which sample was collected or data recorded. This will include a time element where sampling is more frequent than daily

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Core measurement: soils (S Protocol)

Soils - baseline recording (SB)
The following information is recorded in the first year of monitoring:
- Soil map at 1:10 000 scale (or 1:25 000 for larger sites)
- Soil typologies at 50 m intervals around TSS and 25 m intervals within TSS from auger borings

Soils fine-grain recording (SF)
The following variables are recorded for the initial and five-yearly samplings.

Variable	Units	Precision of recording
Site Identification Code		
Core Measurement Code		
Location Code		
Sampling date		
Block code	1 character code[1]	
For each horizon (by block)		
Horizon code	character code[2]	
Horizon lower average depth	cm	1
Horizon average thickness	cm	1
For each depth band (by block)		
Depth code	numeric code[3]	
Then, for each depth and each horizon sample		
Soil moisture	%	0.1
pH	pH scale	0.1
Exchangeable		
Acidity	$mmol_c\ kg^{-1}$	0.1
Sodium	$mmol_c\ kg^{-1}$	0.01
Potassium	$mmol_c\ kg^{-1}$	0.01
Calcium	$mmol_c\ kg^{-1}$	0.01
Magnesium	$mmol_c\ kg^{-1}$	0.01
Manganese	$mmol_c\ kg^{-1}$	0.01

Aluminium (assume all Al is Al_3^+)	$mmol_c\ kg^{-1}$	0.01
Total		
Nitrogen	%	0.01
Phosphorus	$mg\ kg^{-1}$	10
Sulphur	$mg\ kg^{-1}$	0.01
Organic carbon	%	0.1
Inorganic carbon	%	0.1

Soils coarse-grain recording (SC)

The following variables are recorded for the initial and 20-yearly samplings for the each soil block.

Variable	Units	Precision of recording
Site Identification Code		
Core Measurement Code		
Location Code		
Sampling date		
Block code	1 character code[1]	
Cell code	1 character code[4]	
Altitude	m	1
Slope	degrees	1
Aspect	degrees	1
Land use	numeric codes[5]	
Decomposition of peats	character code[6]	
For each horizon		
Horizon code	character code[2]	
Horizon lower average depth	cm	1
Horizon average thickness	cm	1
Description	See note 7	
Particle size analysis	% in each size class[8]	1
Soil mineralogy	% in each mineral class[9]	1
Soil water release	% volume held at different tensions[10]	1
Bulk density	$kg\ m^{-1}$	1
For each depth band		
Depth code	numeric code[3]	
Then, for each depth or horizon sample		
Soil moisture	%	0.1
pH	pH scale	0.1
Exchangeable		
Acidity	$mmol_c\ kg^{-1}$	0.1
Sodium	$mmol_c\ kg^{-1}$	0.01
Potassium	$mmol_c\ kg^{-1}$	0.01
Calcium	$mmol_c\ kg^{-1}$	0.01
Magnesium	$mmol_c\ kg^{-1}$	0.01
Manganese	$mmol_c\ kg^{-1}$	0.01
Aluminium (assume all Al is Al_3^+)	$mmol_c\ kg^{-1}$	0.01
Total		
Nitrogen	%	0.01
Phosphorus	$mg\ kg^{-1}$	10
Sulphur	$mg\ kg^{-1}$	0.01
Organic carbon	%	0.1
Inorganic carbon	%	0.1
Lead	$mg\ kg^{-1}$	0.01
Zinc	$mg\ kg^{-1}$	0.01
Cadmium	$mg\ kg^{-1}$	0.01
Copper	$mg\ kg^{-1}$	0.01

Mercury	mg kg^{-1}	0.01
Cobalt	mg kg^{-1}	0.01
Molybdenum	mg kg^{-1}	0.01
Arsenic	mg kg^{-1}	0.01
Chromium	mg kg^{-1}	0.01
Nickel	mg kg^{-1}	0.01
Extractable		
Iron	%	1
Aluminium	%	1
Phosphorus	mg kg^{-1}	1

Recording forms

The forms supplied in Hodgson (1974) should be used for field survey.

Notes

1. The six soil blocks are coded A-F. Refer to the Protocol for a description of the sampling layout.
2. Horizons should be coded according to the notation described in Hodgson (1974). Codes must be unique within each profile; horizons qualifying for the same letter notation and occurring in vertical sequence are denoted by numerals placed after the letter designation (eg Ah1,Ah2).
3. Depth bands should be coded as follows:

0-5 cm	1	20-40 cm	5
5-10 cm	2	40-60 cm	6
10-20 cm	3	60-80 cm	7
20-30 cm	4*	80-100 cm	8
		100-120 cm	9

   * Bands 1-4 are used for five-yearly sampling (SF). Band 4 is excluded from 20-year sampling (SC), being replaced by band 5.
4. Cells for 20-year sampling are coded from A to P. Refer to the Protocol for a description of sampling layout.
5. Land use should be described using the numeric codes given in Hodgson (1974); these codes are also given here under Section 1.9, note 5, page 167.
6. Decomposition of peats: Table 3 is taken from Avery (1980). Please use the decomposition codes H1-H10 given in the Table.
7. Horizon descriptions should follow the procedures given in Hodgson (1974).
8. Particle size classes are as follows:

Clay (<2 µm)	1
Fine silt (2-63 µm)	2
Coarse silt (63-106 µm)	3
Fine sand (106-212 µm)	4
Med sand (212-600 µm)	5
Coarse sand (600-2 mm)	6

9. Soil mineral categories are as follows:

Sand	Quartz
	Potassium feldspar
	Plagioclase feldspar
	Carbonate minerals
	Ferro-magnesium minerals: garnets, olivines, calcium and magnesium amphiboles, sodium-rich amphiboles, pyroxenes, biotite mica, muscovite, chlorite, epidotes
Silt	Quartz
	Potassium feldspar
	Plagioclase feldspar
	Carbonate minerals
	Ferro-magnesium minerals
Clay	Smectite
	Kaolinite
	Illite
	Chlorite
	Inter-stratified minerals
	Iron-oxide minerals

Aluminium hydroxide minerals

10. Water release tension categories are as follows:

50 millibars	1
100 millibars	2
400 millibars	3
2 bars	4
15 bars	5

Table 3. **Modified version of the Von Post scale for assessing the degree of decomposition of peat (source: Avery 1980)**

	Nature of liquid expressed on squeezing	Proportion of peat extruded between fingers	Nature of plant residues	Description
H1	Clear, colourless	None	Plant structure unaltered; fibrous, elastic	Undecomposed
H2	Almost clear, yellow-brown	None	Plant structure distinct; almost unaltered	Almost undecomposed
H3	Slightly turbid, brown	None	Plant structure distinct; most remains easily identifiable	Very weakly decomposed
H4	Strongly turbid, brown	None	Plant structure distinct; most remains identifiable	Weakly decomposed
H5	Strongly turbid, contains a little peat in suspension	Very little	Plant structure clear, but becoming indistinct; most remains difficult to identify	Moderately decomposed
H6	Muddy, much peat in suspension	One-third	Plant structure indistinct, but clearer in the squeezed residue than in the undisturbed peat; most remains unidentifiable	Well decomposed
H7	Strongly muddy	One half	Plant structure indistinct but recognisable; few remains identifiable	Strongly decomposed
H8	Thick mud, little free water	Two-thirds	Plant structure very indistinct; only resistant remains such as root fibres and wood identifiable	Very strongly decomposed
H9	No free water	Nearly all	Plant structure almost unrecognisable; practically no identifiable remains	Almost completely decomposed
H10	No free water	All	Plant structure unrecognisable; completely amorphous	Completely decomposed

(In this field test a sample of wet peat is squeezed in the closed hand and the colour of the liquid that is expressed between the fingers, the proportion of the original sample that is extruded, and the nature of the plant residues are observed)