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# A Guide to Soil Sampling and Analysis on the National Forests of the Inland Northwest United States

Deb Page-Dumroese  
Al Harvey  
Marty Jurgensen



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## THE AUTHORS

**Deb Page-Dumroese** is a Research Soil Scientist with the Intermountain Research Station's Forestry Sciences Laboratory in Moscow, ID. She received a B.S. degree in natural resource management (1982), an M.S. degree in forest soils (1985), and a Ph.D. degree in forest soils (1988). Her main research focus is on maintaining soil long-term productivity.

**Al Harvey** is Principal Plant Pathologist with the Intermountain Research Station's Forestry Sciences Laboratory in Moscow, ID. He received a B.S. degree in biology

(1960), an M.S. degree in plant pathology (1962), a Ph.D. degree in plant pathology (1968), and spent an academic year of postgraduate work in plant pathology (1972). His current research focus is maintaining forest health and soil/ectomycorrhizae relationships.

**Marty Jurgensen** is a Professor of forest soils in the School of Forestry and Wood Products at Michigan Technological University in Houghton, MI. He received a B.S. degree in forestry (1961), an M.S. degree in silviculture (1965), and a Ph.D. degree in soil science (1967). He conducts research in forest soils and microbiology.



# Why Do We Sample Soils?

Soil samples tell us much about our National Forests: biological productivity, fire influences, changes due to global warming, differences in nutrient contents from one area to another. Comparisons of soil samples could tell us, for example, how human activities have changed—or not changed—the landscape, or how weather patterns or soil compaction influence water flow.

This guide gives general information on soil collection methods, sample analysis, and data translation. Then, for soil scientists or students working in the Inland Northwest United States, we look at particulars on sampling Andisols on National Forests in Idaho, Montana, Washington, and Oregon.

## Field Sampling

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In soil sampling, a problem is the representativeness of samples. It stretches credibility to the limit when soil is characterized over several hundred hectares (to a depth of a meter or more) using only a few dozen samples, each weighing at the most a few kilos.



In pedological field studies, the horizon is the basic sampling unit. Not all horizons, however, need to be sampled. Horizons too thin or too heterogeneous can usually be ignored, but one thing is essential in these studies: horizon boundaries must be carefully respected to avoid contamination from other horizons. This is done by sam-



pling soils from the bottom upwards, after they have been “cleaned” from the top downwards. Usually a 100 g sample is sufficient for most analyses, but more should be taken if the soil is particularly stony. Make sure that your sample represents the pedon.

When sampling a discrete volume, irrespective of horizon boundaries, it is useful to take selective samples. These samples are taken to quantify an entire site to a certain depth; usually the lower sampling depths provide the boundary where prolific rooting stops. These samples are taken with a known volume cylinder.

Whether sampling horizons or taking selective samples, always remember the specific use of the soil once you get it back to the office. Soil collected in the field must have an appropriate identification number for tracking the sample through the laboratory and back to the location from which it was collected.

## **Sample Variation**

Soil variability may be classified in either a vertical or horizontal direction and as to whether the variability is natural or human caused. Soil differences between points on the landscape present the basic challenge in designing an effective soil-sampling procedure. Soil-forming processes may cause sharply contrasting differences in the soil profile, particularly the A, E, and B horizons. These differences relate to organic matter,

pH, texture, cation exchange capacity, and, ultimately, plant nutrient availability. Soils tend to be shallower on the crest of hills and deeper on lower slopes. Avoid sampling in landslides, soil slumps, and from around the roots of overturned trees because a large amount of soil mixing will have taken place. Selection of representative soils is a key to successful sampling and analysis. Therefore, awareness of site variability is essential before planning a soil sampling scheme.

## **Sampling Methods**

**Uniform Sites**—The first problem is defining a “uniform” site. This means that on the meso- (variation in points separated by 0.05-2 m) and macro- (variation in points separated by greater than 2 m) scale, variability is nonsignificant. Sampling procedures that fully satisfy these conditions involve collection of randomly selected soil cores (of known volume) that are mixed together into one (or more) composite soil samples. More than one composite will give an estimate of soil variability. In practice, it is common to collect cores following a zigzag path where a conscious effort is made to force the path into corners and along edges as well as the central parts of the site being sampled.

When the site history is well known, this type of sampling is usually adequate for determining both physical and chemical characteristics.

**Nonuniform Sites**—Where macro-variation is large, a nonrandom soil sampling procedure is recommended. The major objective of nonrandom sampling is to understand the average field conditions, the highs and lows, and the specific locations of site extremes. By its nature, nonrandom sampling requires numerous point soil samples. To do this, a customized grid system is established prior to sampling and soil is collected at each point. Spacing between grid points will vary depending on the degree of detail needed to satisfy sampling objectives. Each point sample is analyzed and results are plotted on a site map in relation to their grid point.



If a site contains a smaller area that is of a different land type, soil classification, or habitat type, then those areas should be sampled separately. This is called a stratified random sample.

### **Effect of Time of Year on Sample Collection**

Seasonal variation in soil test values must be expected because of variations in factors that influence mineral accumulation by plants and recharge of the soil solution nutrient content of minerals as plants remove them from solution. It is difficult to quantify how much this can change soil test values at different times of the year. It is reasonable to expect a seasonal drop in pH values from spring to autumn because of the soluble salt effect resulting from mineralization of organic residues or weathering of minerals. Phosphorus and potassium are likely to drop from their high in late fall to their lowest values in mid-summer. Time of year should be noted when soil samples are collected.

### **Soil Sampling Tools**

A wide array of sampling equipment is available which will perform well, including hand tools to vehicle-mounted hydraulic drive power



probes or augers. The main consideration for tool selection is that it can be cleaned easily between samples and that it accurately samples the intended depth. In

rocky soils the tools must be rugged.

### **Sample Preparation from the Field**

**Drying**—Except under special conditions (for example, saturated soils), oven drying is unnecessary. Air drying is preferable (4 to 8 days). Crumble samples into small clods or pedes by hand and spread on a nonmetallic tray in a

well-ventilated room. Andisols and peaty soils should not be dried.

**Storing**—Once dry, each soil sample should be placed in a zip-loc type bag, permanently labeled and stored in a cool, dry location. Andisols can be stored the same way, but should be undried. These samples should be processed as soon as possible to avoid mold and mildew interference problems.

**Watch Out for Two Problems**—(1) Do not contaminate your samples by using dirty tools or metal equipment. (2) Take care not to crush fragments of fragile rock or stones such as chalk, shale, marl, limestone, or weathered granite. Rough treatment may introduce fine particles of calcium carbonate into the fine earth fraction.

**Interpretation of Results**—It is difficult to interpret analytical results for a single sample. Every measurement must be compared to others. It is particularly useful, for example, to calculate the ratio of total potassium/clay, or estimate the cation exchange capacity of the clay fractions.

For individual horizons the overall results (results of analyses plus morphological description plus other information such as physical, hydrological data, or mineralogical determinations) should be taken into account. In addition, every horizon should be considered in relation to the horizons above or below (vertical relationships) while not forgetting relationships to other soil volumes up or downslope (lateral relationships).

## Choosing Appropriate Analyses \_\_\_\_\_

Soil analyses are expensive, so they shouldn't be performed without specific objectives. There is, for example, no point in asking for an analysis if it cannot be interpreted. The choice of analyses should be guided by three questions:

**1. What do we want to find out?** Is the sample a routine one to characterize a soil for mapping? Is it a repeat to check an earlier analysis? Is it being used for detailed characterization

of a mapping unit to be published? Are you describing and analyzing the soil profile for a specific investigation? Is there a particular thematic concern? Are you using the analyses to check hypotheses in the field? What problems are you trying to solve?

## 2. What do we already know about the soil?

You need to take this into account. For example, there is no point in asking for exchangeable calcium analysis of a calcareous horizon, the carbon/nitrogen ratio for cultivated soil tells us very little,

and full particle-size analysis for all eight fractions is unnecessary for soil with a silty-clayey texture. Part of your evaluation should be to include an inventory of existing data. There may already be an analysis of similar soils available for your interpretations.

## 3. Are there external constraints? What is the cost of analyses in relation to importance of the final data, available finances, and the volume of sample available?



## Coarse Fragments

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The term "coarse fragments" embraces all mineral (lithic) fragments, stone, larger rock, soil concretions, and nodules of various kinds greater than 2 mm in diameter. Stoniness is determined by weighing the residue left on a 2 mm sieve when preparing samples. Stoniness results are expressed as:

$$\text{Coarse fragments by weight (\%)} = \left( \frac{\text{Weight not passing a 2 mm sieve}}{\text{Weight of total soil sample}} \right) * 100$$

It is often useful to convert this to an expression by volume to estimate. For example: the water storage capacity of stony soils. The bulk density of both the coarse fragments and the fine earth must be determined to convert to volume measurements.



### Usefulness...

It is useful to know stoniness, particularly when they are abundant and likely to play a part in:

- reducing the available water capacity
- providing protection against evaporation
- soil warming
- obstacles to harvest/site preparation
- give a clue to parent material
- indicate very old pedogenesis
- soil history

### Particle-Size Analysis

This analysis determines (by weight) the distribution of mineral particles less than 2 mm according to size class. Once the limits of various particle-size classes have been defined it becomes a matter of determining the distribution of particles in the sample. Particle-size analysis is a laboratory operation that involves complete dissociation of the soil material into its individual particles and the total destruction of aggregates.

The most widely used methods (simple and easy to use) are based on Stokes' Law. According to Stokes' Law, the larger a particle, the faster it will fall through water, taking into account that the water temperature affects speed.

For Andisols, normal particle-size analysis is not appropriate because of the destruction of cements, so the dispersion of the particles arising is completely different. We recommend that samples for this analysis not be dried in air and that the fine earth should be sieved in moist condition. To obtain optimum dispersion, prior treatment with ultrasound is effective. Organic matter must then be destroyed by repeated treatments with hydrogen peroxide.

### Usefulness...

Some folks subscribe to the theory that "if only one analysis can be made, then choose particle-size analysis. The best way of describing a horizon by means of a single figure is to give its clay

content." In fact, particle-size composition is not the only variable strongly correlated with other analytical data, but it directly and closely determines how a horizon will behave and function. Besides, all other analytical data cannot be validly interpreted without explicit reference to the particle-size composition, and particularly the clay content. However, the clay content does not convey or explain everything. The structure of the horizon, its history, its organic matter content, and the cations held on the exchange complex are also of considerable importance.

## **Bulk Density**

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Bulk density is the ratio between the mass and apparent volume of a given sample.

$$BD = \text{mass (g)} / \text{volume (cc)}$$

Most commonly, small steel cylinders of known volume are driven vertically or horizontally into



the soil by percussion. This method is fairly easy and can be used to characterize thin horizons. Replicate sampling is possible. However, driving in the cylinder can compress the

sample, especially when stones or roots are present or if the soil is hard or dry.

### **Usefulness...**

Bulk density is an essential item for converting any weight/weight value into a weight/volume value.

## **Characteristic Water Contents**

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Field capacity is the maximum amount of water that a soil can hold against the force of gravity alone. Field capacity is used to indicate water

storage capacity and therefore to estimate water reserves. Also, field capacity provides a good estimate of the maximum contents of water storage formed by soil when the macropores are occupied by air. It can be determined in the laboratory, but with two cautions: (1) gravity alone has a hard time acting on a small sample, and (2) a sample removed from the natural environment may behave differently in the laboratory.

Permanent wilting point is generically described as the point when a plant is unable to extract additional water from the soil. Field capacity and permanent wilting point are needed to determine soil water availability.

A cumbersome but useful tool is water retention curves. They provide identification of the water retention properties of soil. This involves plotting the water content curve by weight as a function of the value of the matric potential. For Andisols, water content is naturally large (about 200 percent for recently obtained samples). Measurement of water content of these soils is only valid for fresh samples because sample drying is largely irreversible.

## **Organic Carbon and Organic Matter** \_\_\_\_\_

“Organic carbon” is the term used for soil carbon other than the carbon in carbonates, expressed as a percentage or weight (g/100 g or g/kg of the fine earth fraction). The total organic carbon content is obtained by a medium-temperature resistance furnace. Organic matter is obtained by loss-on-ignition in a muffle furnace. Generally, organic matter content varies greatly depending on the nature of the vegetation and the kind of humus and depth. Normally, C horizons contain little organic matter and carbon compared to overlying horizons.

### **Usefulness...**

Determination of organic content and organic matter is useful in two circumstances:

- When checking that the total chemical analysis of the sample adds up to 100 percent.



- For horizons which are very rich in poorly decomposed organic matter.

These analyses are relatively easy to obtain, but values are always approximate.

## Soil pH

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The pH measurement of a soil suspended in water takes into account the concentration of dissociated  $H_3O^+$  ions in the supernatant liquid. These ions are in equilibrium with those present in the non-dissociated state, which are fixed to particular soil solids, such as clay minerals, organic matter, and compounds within which aluminum is associated with water molecules and  $OH^-$ . Through their ability to fix  $H^+$  or  $OH^-$ , these solid components cause changes in the soil pH. The resistance they offer to change is called buffering capacity. As cation exchange capacity (CEC) increases, so does the buffering capacity.

The pH is expressed on a scale from 0 to 14. Low values indicate acidity, values  $>7$  indicate a basic character. It is always worthwhile to specify the fine earth/water ratio. Given the great spatial and temporal variability of the equilibria governing soil pH, it is pointless to express pH with a greater accuracy than one-tenth of a unit.

The expression "pH of a soil" has no physico-chemical significance other than the pH measured in a solution in equilibrium with a soil sample suspended in water (or KCl) using a specified soil/solution ratio.

Equilibrium in water does not take into account all the acid ions (protons and aluminum ions) which are fixed on exchange sites. The exchange acidity at the pH of the soil can be displaced by exchange with an ion such as  $K^+$  in a normal unbuffered KCl solution. It should be understood that  $KCl\ pH < water\ pH$ .

The pH determinations are useful, inexpensive, and easy: they can be carried out with simple equipment. Water pH is of little interest in calcareous soils. It is mainly useful in neutral or acid soils. One major threshold must be

pointed out: below a water/soil pH value of 5.0, the presence of exchangeable aluminum gives rise to risk of aluminum toxicity. Generally, the lower the pH of a horizon, the lower the base saturation. Determination of pH alone is not enough for good interpretation. It must be supplemented by measurement of the cation exchange capacity and exchangeable cations (including  $\text{Al}^{3+}$  in the case of very acid soils, less than 5.0).

## Cation Exchange Capacity \_\_\_\_\_

The cation exchange capacity of a sample is the total number of cations it can retain on its adsorbent complex at a given pH. Cation exchange capacities are currently expressed as centimoles per kg. The centimole unit is used especially for cation exchange capacity. It represents one-thousandths of a gram equivalent. According to official standards it will be better in the future to use the "mole" which defines the same quantity as the equivalent. Thus:

$$1 \text{ me}/100 \text{ g} = 1 \text{ mmol}^+/100 \text{ g} = 1 \text{ cmol}^+/\text{kg}$$

### Usefulness...

Cation exchange capacity determination is quite essential for assessing interpretation of soils data. It is most useful to indicate the potential fertility of soils. In addition, it provides an overall idea of the nature and amount of the clay minerals. Some average cation exchange capacities are:

Silty soils	35 me/100 g
Bulk soil	22 me/100 g
Clayey soils	50 me/100 g

Allophanes in Andisols pose special problems because they have a cation exchange capacity that varies depending on the pH of the exchange solution. Cation exchange capacity values in these soils are very high and are at a maximum at alkaline pH's. The standardized measurements at pH 7 are therefore insignificant to such soils. Measurements should be made at several pH values, using buffered ammonium acetate.

## Chemical Analyses

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According to requirements and financial constraints many different chemical analyses can be requested. Generally, the six most important elements are: N, K, Mg, Ca, Na, and P. The S and Mn may also be requested. All other chemical elements occur in small amounts and their determination is outside the scope of routine analysis and are expensive to obtain.

Nitrogen analyses—including ammonium ( $\text{NH}_4^+$ ), nitrate ( $\text{NO}_3^-$ ), and total N—are usually the most important and can be used, for instance, to determine the C/N ratio. Nitrogen in most forested ecosystems is the most limiting element and therefore of great concern to managers.



The inorganic combined N in soils is predominately  $\text{NH}_4^+$  and  $\text{NO}_3^-$ . Inorganic N may represent more than a small fraction of soil total N. Exchangeable  $\text{NH}_4^+$  is extractable at room temperature in a 2 N KCl solution. Determination of  $\text{NH}_4^+$  and  $\text{NO}_3^-$  in soils is complicated by rapid biological transformations that may occur, changing the amounts and forms of inorganic N in the sample. Ideally, soil samples taken to determine inorganic forms of N should be refrigerated and analyzed as soon as possible for valid results.

However, some delay is nearly impossible to avoid, because samples must be transported to the laboratory, sieved, and perhaps subsampled before analysis. If the samples cannot be run within 3 weeks, the soil should be either frozen at  $0^\circ\text{C}$  or dried at laboratory temperatures. Air-drying can lead to small but significant increases in exchangeable  $\text{NH}_4^+-\text{N}$  and  $\text{NO}_3^- - \text{N}$ . Suitable precautions for air-drying of soil samples include drying at low temperatures (elevated temperatures such as greater



than 55 °C can lead to marked increases in exchangeable  $\text{NH}_4^+-\text{N}$  and storing in closed plastic or glass containers. Drying in paper bags also results in significant increases in exchangeable  $\text{NH}_4^+-\text{N}$ . Nitrogen mineralization (the potential of a soil to supply N to plant when conditions are ideal for mineralization) may be the most diagnostic analysis when trying to relate N to tree growth.

Total N is obtained by a medium-temperature resistance furnace. Total N analysis is complicated by the lack of knowledge concerning N forms present and by the low N content of the material under analysis. Total soil N content ranges from less than 0.02 percent in subsoils and greater than 2.5 percent in peats.

Exchangeable K is another important soil analysis that can be used to relate soil K to tree growth. The Intermountain Forest Tree Nutrition Cooperative suggests that when the ratio of exchangeable K/mineralizable N is less than 6 to 10 then K is in short supply or there is too much mineralizable N.

Results from a laboratory are expressed either as elements or oxides. Check this point carefully. They are also frequently expressed as percentages or parts per million (or thousands). The sum of the oxide form and the loss-on-ignition organic matter value should be approximately 100 percent, provided that these values have been expressed in the same way and loss-on-ignition was determined on the same sample.

### Usefulness...

Chemical analysis of one or more elements allows samples to be compared, thus allowing one to trace the pedological differences within a solum or changes across the landscape.

Examples:

- Potassium analysis: A high K concentration can indicate abundant micas or potassium feldspars (orthoclase) in the sand and silt fractions.

- **Magnesium analysis:** In limestone country a relative abundance of this element can indicate the presence of dolomite.
- **Calcium analysis:** In soils without carbonate or dolomite, low concentrations of Ca can be attributed to feldspars.

Soil scientists working on National Forests in the Inland Northwest will find in table 1 information on soil nutrients that could be helpful in their own analyses.

## Salinity and Electrical Conductivity

Any soil containing an abnormally high quantity of soluble salts is regarded as "saline." Two methods can be used to extract soluble salts. The first method saturates the soil to its Atterberg "liquidity limit" and gives a fine earth fraction/water ratio which varies according to texture. The second method mixes the fine earth fraction with water so that the soil/water ratio is constant regardless of the nature of the sample and its particle-size distribution.

The overall salinity is determined by measuring its electrical conductivity, which is the conductance

**Table 1**—Average selected soil nutrients for the Inland Northwest United States. Values are for the surface 0 to 10 cm of mineral soil.

Nutrient	Central Idaho	Northern Idaho	Montana	Central Washington	Northeast Oregon	Northeast Washington
Total N (percent) <sup>1</sup>	0.15	0.17	0.14	0.10	0.23	0.17
Mineralizable Soil N (ppm) <sup>1</sup>	28	38	35	25	48	35
Available P (ppm) <sup>1</sup>	60	29	60	75	98	50
Exchangeable Ca (ppm) <sup>1</sup>	6	9	13	7	10	8
Exchangeable Mg (ppm) <sup>1</sup>	1.8	1.3	2.4	1.1	4	1.2
Exchangeable K (ppm) <sup>1</sup>	610	310	250	290	410	290
Carbon (percent) <sup>1</sup>	1.7	1.7	2.4	1.6	4	1.2

<sup>1</sup>From data collected by the Intermountain Forest Tree Nutrition Cooperative.

of the solution measured between electrodes of 1 cm<sup>2</sup> surface area 1 cm apart. Conductance can be expressed in mhos and conductivity in mhos/cm. (In SI units siemens, S, replaced mhos, so that 1 mmho/cm is equivalent to 1 dS/m.) Electrical conductivity values range from 0 to 16 mmho/cm. Above 8 dS/m the yields of most cultivated plants are affected by salinity. Only specialized plants can prosper above an electrical conductivity of 16 dS/m.

## Glossary

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**Active acidity**—The activity of the hydrogen ion in the aqueous phase of a soil. It is measured and expressed as a pH value.

**Aggregate** (of soil)—Many fine soil particles held in a single mass or cluster, such as a clod, crumb, block, or prism. Many properties of the aggregate differ from those of an equal mass of unaggregated soil.

**Alkaline soil**—Any soils whose pH is greater than 7.0.

**Cation exchange capacity**—The sum total of exchangeable cations that a soil can absorb. Sometimes called the total-exchange capacity, base-exchange capacity, or cation-adsorption capacity.

**Exchange acidity**—The titratable hydrogen and aluminum that can be replaced from the adsorption complex by a neutral salt solution. Usually expressed as milliequivalents/100 g of soil.

**pH, soil**—The negative logarithm of the hydrogen-ion activity of a soil. The degree of acidity (or alkalinity) of a soil as determined by means of a glass, quinhydrone, or other suitable electrode or indicator at a specified moisture content or soil-water ratio, and expressed in terms of the pH scale.

**Saline soil**—A nonsodic soil containing sufficient soluble salt to impair its productivity. The electrical conductivity of the saturation extract is >2 mmhos/cm.

**Sodic soil**—(1) A soil containing sufficient exchangeable-sodium to interfere with the growth of most plants. (2) A soil in which the sodium adsorption ratio of the saturation extract is 15 or more.

**Solum**—The set of genetic horizons developed by soil-forming factors. The upper and most weathered part of the soil profile (usually the A and B horizons).



## Conversion Factors for SI and Non-SI Units

To convert column 1 into column 2, multiply by	Column 1 SI unit	Column 2 non-SI unit	To convert column 2 into column 1, multiply by
<b>Concentrations</b>			
1	centimole per kilogram, $\text{cmol kg}^{-1}$ (ion exchange capacity)	milliequivalents per 100 grams, $\text{me } 100 \text{ g}^{-1}$	1
0.1	gram per kilogram, $\text{g kg}^{-1}$	percent, %	10
1	milligram per kilogram, $\text{mg kg}^{-1}$	parts per million, ppm	1
<b>Electrical conductivity</b>			
10	siemen per meter, $\text{S m}^{-1}$	millimho per centimeter, $\text{mmho cm}^{-1}$	0.1

## For More Information...

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This guide details soil collection methods, sample analysis, and data translation. It outlines what field soil scientists need to make accurate interpretations of site information. Included are instructions for sampling typical Andisols found on National Forests of the Inland Northwest United States.

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Keywords: Andisols, soil variability, coarse fragments, particle size, bulk density, organic carbon, organic matter, soil pH

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## INTERMOUNTAIN RESEARCH STATION

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