

## Chapter 2

# Sampling and Analysis of Soils and Plant Tissues

## How to Take Representative Samples, How the Samples are Tested

*N. V. Hue, R. Uchida, and M. C. Ho*

Scientific approaches to crop nutrient management are based on data obtained by analyzing soil and plant tissue samples. Careful sampling techniques will ensure a representative sample. Precise analytical methods will ensure reliable data.

However, the most carefully collected sample and the most precise analytical techniques will not ensure appropriate interpretation of the data. This depends upon how well the data have been calibrated to actual conditions, which is the subject of Chapter 8.

Part of that calibration involves selecting the appropriate analytical method. For example, let's say that we have found that our best crops of corn (maize) are grown on soil having 40 ppm Modified Truog–extractable phosphorus (P). Our goal then is to determine if the soil in the field to be planted with corn has at least 40 ppm Modified Truog P and, if not, to apply sufficient fertilizer P to supply the corn with adequate P. If the Melich 3 solution (see Melich 1984) is used to extract soil P, the soil must have 100 ppm P to satisfy the crop's needs. Different methods of analysis involve different procedures for extracting the “plant-available P,” and one extraction method may be better suited from some soils than others.

This chapter is based on procedures and techniques used by the CTAHR Agricultural Diagnostic Service Center (ADSC), which have been developed over many decades of research on soils of Hawaii and interpretation of their analysis data. Not all laboratories use these same methods, and results from different methods require different interpretations.

In addition to commercial laboratory analytical services such as ADSC, test “kits” and portable analysis instruments are available on the market for use by growers to do their own analysis of soils and plant tissues. These methods are usually not as precise as the laboratory techniques described here, but they can nevertheless be useful. Growers who keep good records of inputs and yields and who sample regularly with consistent methods will develop a “calibration,” or correlation between test readings and crop performance. This will allow them a degree of “farming precision” even with analytical methods that are less precise than those used in commercial laboratories.

### Soil analysis

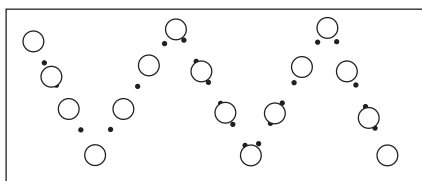
Laboratory analyses are performed on small samples of soil taken from relatively large areas of land. If the sample does not truly represent the soil you intend to treat, all the precision of the analytical process is useless. Improperly collected samples not only make test results less informative than they might be, but the results also may lead to erroneous recommendations that reduce yields, waste money and resources, and pollute the environment.

### How do I take a representative soil sample?

First, make a detailed map of your land. Divide your map into individual soil-test areas of a few (1–5) acres each. Label each area clearly on the map by using a combination of letters and numbers that make sense

and thus are easy to remember. Each test area should consist of only one soil type or variation. Areas with different slope, color, drainage, texture, or management history should be sampled separately.

Samples should be one-inch cross-sections of the soil (called cores) taken to a specified depth, normally 0–4 inches for no-till fields or established pasture and turf and 0–8 inches for conventionally tilled fields (James and Wells 1990). For trees and fruit crops, two samples at different depths should be taken wherever possible: a surface sample from 0–8 inches and a sub-soil sample from 8–24 inches. Each sample to be tested should be a thorough mix of 10–15 cores taken randomly or in a scientifically determined pattern. Although there are many sophisticated techniques (e.g., Kriging, strip sampling) to deal with variability in the field, a zigzag sampling pattern or a variant of it is often adequate for obtaining a reasonably representative soil sample (Sabbe and Marx 1987).



Collect soil samples in a zig-zag pattern

A soil “probe” is the professional’s tool for collecting soil cores, but soil cores can be collected with a garden spade or trowel. Remove a shovelful of soil to the depth you wish to sample, then cut a one-inch section from the wall of the hole you have just dug. Place it in your mixing bucket. Care should be taken that an equal amount of soil is taken at each of the sampling sites, so that the resulting composite sample will represent all the sample sites equally.

Clean tools are essential for sampling soils. If specialized analyses are to be done, avoid brass, bronze, or galvanized tools, which may contaminate your samples with copper and zinc. Clean probes or shovels made of stainless steel are preferred. Mixing buckets should be durable but light (preferably made of plastic) and clean. A small amount of lime or fertilizer residue left in the bucket can severely distort your results. If analysis for boron is desired, the soil samples should not be stored in grocery (brown) paper bags, because such paper can release boron to the sample.

Collect soil samples two to three months before planting. In so doing, you will get your test results in plenty of time to plan your soil amendment and fertilizer needs. Allow adequate time (at least two weeks for ADSC) for the laboratory to complete the analysis of your soils. Soils should be retested to confirm the effects of soil amendments applied. Subsequent tests of actively managed soils should be done to warn of nutrient buildup or depletion—perhaps once every two years or even more frequently, depending on the cropping activity.

### Submitting samples and providing relevant information

After properly collecting soil samples, they may be submitted to the CTAHR Agricultural Diagnostic Service Center (ADSC) by taking them to county offices of the CTAHR Cooperative Extension Service (CES) or delivering them directly to the ADSC on the UH-Manoa campus (1910 East-West Road, Room 134; Honolulu, HI 96822). Samples must be accompanied by an ADSC Soil Sample Information Form, shown on p. 30, which is available at CES offices or from ADSC. The more complete the information provided, the better recommendation you will get. Because fertilizer and lime requirements vary with soils and crops, information about the soil’s apparent density (heavy, light, or *a‘ā* lava land), the crop to be grown, and the soil’s cropping and management history are important for making correct recommendations.

In areas outside of Hawaii, follow the protocol established by the regional soil and plant testing service.

### How are soil samples tested?

The ADSC provides all residents of Hawaii a reasonably affordable soil and plant tissue testing service. Routine analyses of soils and ornamental mixes include pH, salinity, extractable phosphorus (P), potassium (K), calcium (Ca), and magnesium (Mg). Soil organic carbon (organic matter), total nitrogen, extractable aluminum (Al), boron (B), and other micronutrients (e.g., zinc, manganese, copper) can be measured upon request. Detailed descriptions of the analytical procedures follow.

## Routine soil analyses

### **Sample preparation**

Most soil samples are air dried and sieved through a 2-mm screen. Soils derived from volcanic ash (soils classified as Andisols) and soils being analyzed for ammonium ( $\text{NH}_4$ ) are not air dried.

### **Soil pH**

A soil sample weighing 30–50 grams (g) is placed in a waxed cup, and deionized water is added to make a saturated paste. The paste is equilibrated for one hour with occasional stirring. (Equilibration time is 1.5 hours for soilless mixes). pH is measured with a pH meter.

### **Soil salinity (electrical conductivity, EC)**

A 50-g sample of soil is placed in a 100-ml disposable plastic cup; 50 ml of deionized water is added. The slurry is shaken on a reciprocating shaker for 45 minutes, then filtered. Electrical conductivity of the filtrate is read with a conductivity bridge.

### **Extractable soil nutrients**

**Extractable phosphorus in soils with pH < 7.0.** The Modified Truog procedure is used (Ayres and Hagihara 1952). An extracting solution of 0.01 M  $\text{H}_2\text{SO}_4$  (sulfuric acid) + 0.02 M  $(\text{NH}_4)_2\text{SO}_4$  (ammonium sulfate) in a soil-to-solution ratio of 1:100 with 0.5 g of soil is shaken for 30 minutes.

**Extractable phosphorus in soils with pH > 7.0.** The Olsen method is used (Olsen et al. 1954, Olsen and Sommers 1982). An extracting solution of 0.5 M  $\text{NaHCO}_3$  (sodium bicarbonate), pH 8.5, in a soil-to-solution ratio of 1:20 with 2.5 g of soil is shaken for 30 minutes.

In both the Olsen and Modified Truog methods, the slurry is filtered and phosphorus in the filtrate is measured colorimetrically using the Murphy-Riley method (Watanabe and Olsen 1965) on an auto-analyzer.

### **Extractable soil cations (Ca, K, Mg)**

Ammonium acetate (1 M, pH 7.0) is used as the extracting solution with a soil-to-solution ratio of 1:20 with 2.5 g of soil shaken 10 minutes. Calcium (Ca), magnesium (Mg), and potassium (K) in the filtrate are measured with an atomic absorption spectrophotometer (AA).

## Special soil analyses (by request)

### **Organic carbon**

A modified version of the Walkley-Black method (Heanes 1984) is used.

### **Total nitrogen**

The micro-Kjeldahl method (Bremner and Mulvaney 1982) is used.

### **Extractable aluminum**

Aluminum extraction uses 50 ml of 1M KCl in a soil-to-solution ratio of 1:10 with 5 g of soil, shaken for 30 minutes. Aluminum in the filtrate is measured with an inductively coupled plasma spectrophotometer (ICP).

### **Extractable boron**

Hot water is used to extract boron from 10 g of soil mixed with 20 ml water and boiled for 5 minutes. Boron in the filtrate is measured colorimetrically using the azomethine-H method (Wolf 1974, Mahler et al. 1984).

### **Extractable micronutrients**

The DTPA method (Lindsay and Norvell 1978) is used. A 10-g soil sample is mixed with 20 ml DTPA (0.005 M, adjusted to pH 7.3 with TEA), then shaken for 2 hours before filtering. The micronutrients are measured with an AA spectrophotometer.

## Soil test results and fertilizer recommendations

Within two to three weeks you should receive from the ADSC the result of your soil test along with fertilizer recommendations, if requested. The results for pH, P, K, Ca, and Mg are interpreted as either very low, low, sufficient, high, or very high. Fertilizer recommendations provided include amounts of lime (given in lb/1000 ft<sup>2</sup> or tons/acre) and its estimated cost and fertilizer formulation options (for example, 21-0-0, 21-0-32, 10-30-10) and their amounts and costs.

## Plant tissue analysis

As with soil analysis, sampling and sample preparation of plant tissues are often the weakest steps in the testing process. The sample should represent the overall plant population in the field, otherwise all the careful and usually costly analytical work is wasted.

## How do I take a representative plant tissue sample?

Different plant species may require different tissue parts for meaningful sampling and interpretation. A list of recommended sampling procedures (Jones and Case 1990, Jones et. al 1971) is reproduced in Table 2-1.

To ensure a representative sample, sample as many plants as practical. Generally, the youngest fully matured leaves on main branches or stems are sampled. They should be taken just prior to or at the onset of flowering.

Do not collect tissue that is covered with soil or dust. Do not collect from plants that are damaged by insects, mechanically injured, or diseased. Dead plants or senescent tissues should not be sampled. Also, sampling is not recommended when plants are under moisture or temperature stress.

Samples must be protected from dirt and fertilizer materials and should be placed in clean plastic bags. The form used by ADSC to obtain information on plant tissues submitted for analysis is reproduced on page 65.

## How are plant-tissue samples tested?

### Sample preparation

Samples are cleaned, placed in a forced-draft oven at 55°C (or 70°C in a gravity oven) for at least 12 hours, then ground to pass a 2-mm sieve with a Wiley mill. A 0.50-g sample is dry-ashed in a porcelain crucible for 4–6 hours at 500°C in a muffle furnace. (If the ashing is judged incomplete, then the ash is cooled, dissolved in 1 M nitric acid, evaporated to dryness, then ashed again for 1 hour.) The residue is dissolved in 25 ml of 1 M hydrochloric acid.

### Routine plant tissue analyses

Analyses for P, K, Ca, Mg, Fe, Cu, Zn, Mn, Mo, Al, and Na are done on the ash solution, using an ICP spectrophotometer. Boron is measured by the azomethine-H colorimetric method.

### Total nitrogen

A 0.250-g sample of dried, ground plant material is mixed with approximately 2 g of Na<sub>2</sub>SO<sub>4</sub> and 7 ml of a digestion mixture of concentrated sulfuric acid, salicylic acid, and selenium. After a minimum of 2 hours, 3–4 drops of a sodium thiosulfate solution are added. The mixture is allowed to stand for 45 minutes, then 4 ml of 30% hydrogen peroxide is added. The mixture is

then digested at 410°C until a clear liquid is obtained (approximately 45 minutes). The liquid is cooled, then diluted with water. Nitrogen (NH<sub>4</sub>) in this solution is measured colorimetrically by an auto-analyzer.

## Special plant tissue analyses (by request)

Nitrate, sulfur, and silicon in plant tissues can be determined upon request.

## References

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**Table 2-1. Suggested plant tissue sampling procedures for some crops (Jones et al. 1971).**

<b>Crop</b>	<b>Stage of growth</b>	<b>Plant part to sample</b>	<b>No. of plants to sample*</b>
<b>Field crops</b>			
Corn	Seedling stage (<12 inches)	All the aboveground portion	20–30
	Prior to tasseling	The entire leaf fully developed below the whorl	15–25
	From tasseling and shooting to silking	The entire leaf at the ear node (or immediately above or below it)	15–25
Soybean or other bean	Seedling stage (<12 inches)	All the above-ground portion	20–30
	Prior to or during initial flowering (Sampling after pods begin to set is not recommended)	2 or 3 fully developed leaves at the top of the plant	20–30
Small grains (including rice)	Seedling stage (<12 inches)	All the aboveground portion	50–100
	Prior to heading (Sampling after heading is not recommended)	The 4 uppermost leaves	50–100
Hay, pasture or forage grasses	Prior to seed head emergence, or at the optimum stage for best quality forage	4th uppermost leaf blade	40–50
Alfalfa	Prior to or at $\frac{1}{10}$ -bloom stage	Mature leaf blades taken about one-third of the way down the plant	40–50
Clover and other legumes	Prior to bloom	Mature leaf blades taken about one-third of the way down the plant	40–50
Sugarbeets	Mid-season	Fully expanded and mature leaves midway between the younger center leaves and the oldest leaf whorl on the outside	40–50
Tobacco	Before bloom	Uppermost fully developed leaf	8–12
Sorghum-milo	Prior to or at heading	2nd leaf from top of plant	15–25
Peanuts	Prior to or at bloom	Mature leaves from both the main stem and either cotyledon lateral branch	40–50
Cotton	Prior to or at first bloom or when first squares appear	Youngest fully mature leaves on main stem	30–40

. . . continued

\*Number of plants to sample per acre. For smaller areas, the minimum number of plants to sample should not be less than five.

**Table 2-1 (continued) *continued.***

<b>Crop</b>	<b>Stage of growth</b>	<b>Plant part to sample</b>	<b>No. of plants to sample*</b>
<b>Vegetable crops</b>			
Potato	Prior to or during early bloom	3rd to 6th leaf from growing tip	20–30
Head crops (cabbage, etc.)	Prior to heading	1st mature leaves from center of whorl	10–20
	At heading	Wrapper leaf	10–12
Tomato (field)	Prior to or during early fruit set	3rd or 4th leaf from growing tip	20–25
Tomato (greenhouse)	Prior to or during fruit set	Young plants: leaves adjacent to 2nd to 3rd clusters Older plants: leaves from 4th to 6th cluster	20–25
Bean	Seedling stage (<12 inches)	All the aboveground portion	20–25
	Prior to or during initial flowering	2 or 3 fully developed leaves at the top of the plant	
Root crops (carrots, onions beets, etc.)	Prior to root or bulb enlargement	Center mature leaves	20–30
Celery	Mid-growth (12–15 inches tall)	Petiole of youngest mature leaf	15–30
Leaf crops (lettuce, spinach, etc.)	Mid-growth	Youngest mature leaf from the top of the plant	35–60
	At heading	Wrapper leaf	10–12
Peas	Prior to or during initial flowering	Leaves from the 3rd node down	30–60
Sweet corn	Prior to tasseling	The entire fully mature leaf below whorl	20–30
	At tasselling	The entire leaf at the ear node	
Melons (watermelon, cucumber melon, muskmelon)	Early growth prior to fruit set	Mature leaves near the base portion of plant on main stem	20–30
<b>Fruits and nuts</b>			
Apple, apricot, almond, prune, peach, pear, cherry	Mid-season	Leaves near base of current year's growth or from spurs	50–100
Strawberry	Mid-season	Youngest fully expanded mature leaves	50–75
Pecan	6 to 8 wks after bloom	Middle pair of leaflets from mid-portion of terminal growth	30–45
Walnut	6 to 8 wks after bloom	Middle pair of leaflets from mature shoots	30–35

<b>Crop</b>	<b>Stage of growth</b>	<b>Plant part to sample</b>	<b>No. of plants to sample*</b>
<b>Ornamentals</b>			
Lemon, lime	Mid-season	Mature leaves from last flush or growth on nonfruiting terminals	20–30
Orange	Mid-season	Spring cycle leaves, 4 to 7 months old from nonbearing terminals	20–30
Grapes	End of bloom period	Petioles from leaves adjacent to fruit clusters	60–100
Raspberry	Mid-season	Youngest mature leaves on lateral or “primo” canes	20–40
Ornamental trees, shrubs	Current year’s growth	Fully developed leaves	30–100
Turf	2–3 weeks after mowing	Leaf blades; clip by hand to avoid contamination with soil or other material	30–40
Roses	During flowering	Upper leaves on the flowering stem	20–30
Chrysanthemums	Prior to or at flowering	Upper leaves on flowering stem	20–30
Carnations	Unpinched plants	4th or 5th leaf pairs from base of plant	20–30
	Pinched plants	5th and 6th leaf pairs from top of primary laterals	20–30
Poinsettias	Prior to or at flowering	Most recently mature fully expanded leaves	15–20

\*Number of plants to sample per acre. For smaller areas, the minimum number of plants to sample should not be less than five.



## Agricultural Diagnostic Service Center

Sherman Laboratory, Rm. 134  
 1910 East-West Rd., Honolulu, HI 96822  
 Ph: (808) 956-6706; Fax: (808) 956-2592  
 E-mail: adsc@ctahr.hawaii.edu

### Sample Information Form (soil or water)

Name \_\_\_\_\_ Phone \_\_\_\_\_  
first, middle initial, last

Mailing address \_\_\_\_\_ Fax \_\_\_\_\_

City \_\_\_\_\_ State \_\_\_\_\_ Zip code \_\_\_\_\_ E-mail \_\_\_\_\_

#### Sample description

Identification label: 1. \_\_\_\_\_ 4. \_\_\_\_\_

**(The sample identification label should be written on the sample container. This form may be used for up to six samples. When information is given below, be sure to clearly note by number [1–6] the sample that is being referred to. If this cannot be clearly done, use separate forms.)**

2. \_\_\_\_\_ 5. \_\_\_\_\_

3. \_\_\_\_\_ 6. \_\_\_\_\_

This sample is:  accompanied by plant tissue sample/s [provide tissue sample ID label: \_\_\_\_\_]  
 a follow-up sample, related to a sample previously analyzed  
 [provide sample ID label from previous analysis report: \_\_\_\_\_]

Soil series or mapping unit: \_\_\_\_\_  
(Optional information; can be obtained from the *Soil Survey of the State of Hawaii*, available at local libraries.)

Describe location, condition, and problem: \_\_\_\_\_

\_\_\_\_\_  
 \_\_\_\_\_

(If more space is needed, use the back of this form)

Apparent soil density:  heavy  light  a'ā lava

Can you till in fertilizer 4–6 inches?  yes  no

Soil management history: type or formulation quantity applied how often applied date of last application

lime \_\_\_\_\_

manure \_\_\_\_\_

fertilizer \_\_\_\_\_

other \_\_\_\_\_

#### Plant/s to be grown:

##### Vegetable crop:

- lettuce  cabbage
- onion  bean
- tomato
- other \_\_\_\_\_  
(specify)

##### Orchard crop:

- coffee  macadamia nut
- papaya  guava
- avocado  banana
- other \_\_\_\_\_  
(specify)

##### Field crop:

- wetland taro  dryland taro
- corn  soybean
- other \_\_\_\_\_  
(specify)

- Mixed garden or general landscaping
- Turfgrass
- Container plant/s (specify) \_\_\_\_\_
- Pasture:  improved pasture  natural rangeland
- Forage:  grass  legume (specify plant/s) \_\_\_\_\_
- Other crop category (specify plant/s) \_\_\_\_\_

Special reporting instructions: \_\_\_\_\_

- Only nutrient levels and adequacy diagnosis are needed (no fertilizer recommendation needed).

Other comments or instructions: \_\_\_\_\_

<b>ADSC use only:</b>	Job Control no. _____	Date received _____ <small>month / day / year</small>
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