

## Chapter 2

# Soil Sample Preparation and Extraction

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In the Northeast, routine testing of soil samples invariably begins with a drying step and is followed by some form of pulverization. Analysis of dried, ground, sieved soils allows for more convenient sample handling and greater reproducibility of results in comparison with analysis of field-moist soil. Sample preparation steps, although often taken for granted, have frequently been shown to significantly affect analytical results (Elk and Gelderman, 1988; Munter, 1988; Soltanpour 1976). It is therefore critical that standardized sample handling and extraction procedures be used by soil testing laboratories.

Of the major plant nutrients, potassium (K) has been shown to be significantly affected by drying (Attoe, 1947; Dowdy and Hutcheson, 1963). Whether there is a release or fixation of K upon drying appears to be determined by clay mineralogy and exchangeable K levels. Changes in available K are affected by drying-induced changes in clay because of the affinity of K for interlayer positions in certain soil clays. Extractable phosphorus has also been shown to change with drying (Pote, et al, 1999; Turner and Haygarth, 2003). Most studies indicate an increase in extractable P with drying, though not for all extraction methods. Where differences occur, they are not consistent for all soils but tend to be greater at low P test levels.

While not changed by drying, levels of soil nitrate may fluctuate in a soil sample prior to drying. Warm, moist conditions may bring about large increases in soil nitrate, especially after thawing a frozen sample. Reducing conditions in soils due to excess moisture and a resulting lack of oxygen may cause decreases in soil nitrate through denitrification reactions (Alexander, 1977).

Among the micronutrients, manganese (Mn) is most affected by drying. Generally, there is an increase in extractable Mn upon drying, caused by the reduction of insoluble manganese oxides to a soluble form (Bartlett and James, 1980). This may mask potential Mn deficiencies. Comparisons between different methods of soil crushing are not common; however in two studies conducted by Soltanpour et al. (1976 and 1979), it was shown that grinding force and grinding time both significantly affected the quantities of Fe and Zn extracted by DTPA. The authors recommended that these two factors should be standardized among laboratories to ensure both analytical accuracy and precision. Contamination of samples with micronutrient metals abraded from grinding equipment is another potential problem that should be evaluated.

After the soil samples have been dried and sieved, it is then possible to obtain representative subsamples for analysis. Because of the tendency of the bulk sample to fractionate by aggregate size, it is important to thoroughly homogenize each soil sample by some form of mixing before weighing or scooping the subsample.

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In the Northeast, there is a difference in preference among labs for scooping vs. weighing of subsamples. Scooping is the quicker method of subsampling and allows for rapid sample throughput. Scooping can also partially compensate for variations in field bulk density of soil. It is most compatible with the expression of results on a volume basis, such as mg/dm<sup>3</sup>. With both scooping and weighing, there may be a significant bias in results if the sample is not completely dry (Glenn, 1983). A standardized soil scoop and scooping procedure, such as that developed and used in many north central states (Peck, 1988), are necessary to ensure accurate, reproducible volume-basis results.

Weighing has been reported to generally provide greater precision in soil analysis. Weighing has been shown to be much more accurate in the empirical prediction of exchangeable acidity from buffer pH (Bourgoin, 1980). Relationships between quick test and established reference methods for exchangeable cations were also more precise using weighed rather than scooped subsamples (Glenn, 1983). A compromise between weighing and scooping would be to record the weight of each scooped subsample. Results may then be expressed on a weight or a volume basis. This is most feasible where weights are captured and recorded electronically. When weighing scooped soil samples, be aware that variations in texture and crumb structure of dried and sieved soils can result in a wide range of actual weights for the same volume of soil. This can affect the extraction efficiency for phosphorus and other nutrients from sample to sample, especially in methods with narrow (soil:solution) extraction ratios or shorter contact times. When considering scooping as a sampling option, time saved should be balanced against potential loss of accuracy and precision.

Expression of analytical results in pounds of nutrient per acre of soil, using either subsampling system, is normally based on the assumption that the tillage layer contains two million pounds of soil per acre. Scooping assumes an average weight of a volume of soil, whereas a weighed subsample is directly converted to a two million pound basis by multiplying the results in ppm of soil (mg•kg<sup>-1</sup>) by two. Both systems suffer from the common problem of not taking the volume or weight of coarse fragments into account. In the glacial soils of the Northeast, where up to 35% or more of the volume of the plow layer may be composed of coarse fragments (Rourke and Bangs, 1975), this can constitute a significant bias. It has been the convention, however, to use the two-million-pound conversion factor as a common reference point for indexing available nutrient levels. Ultimately, selection of scooping or weighing as the subsampling technique should depend primarily upon the method that was used in field calibration trials or yield-response studies conducted in the state or region.

Many factors affect the analytical results of a nutrient extraction procedure. These factors range from the most basic principles of laboratory operation (e.g. equipment cleanliness, quality control) to temperature, shaking speed, size or shape of extracting vessel, and soil:solution contact time.

Where large numbers of samples are analyzed using automated or mass production procedures, contamination of laboratory equipment can be a significant source of analytical error. For example, ions adsorbed on extraction flasks or sample bottles in contact with

extracted solutions may be a source of contamination for subsequent analyses using the same containers. Because of this potential for contamination, many laboratories rinse glassware with either a dilute acid solution or with an aluminum chloride solution (Tucker, 1985). Rinsing glassware with a cleaning solution is particularly important for the analysis of micronutrients and trace metals, provided that the acid anion is not one of the analytes. Acids have the disadvantage of being corrosive to equipment and potentially hazardous in the work environment. An alternative rinsing solution, aluminum chloride ( $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ , 2 g/L) has been used successfully in several states. In studies in Maine,  $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$  was shown to remove high concentrations of Ca, K, Mg, and P from glassware (Hoskins, unpublished data). Aluminum chloride is also less corrosive and safer than most acid rinses, but may not be effective for removing trace elements to sub-part per million levels. Aluminum chloride may also significantly interfere in the determination of Al or Cl.

The north central states conducted a study into the effects of temperature, shaking speed, and extracting vessel type on potassium (K) and phosphorus (P) extraction from soils, using the standard procedures of the region. No significant differences were found in extractable P or K levels as a result of changing shaking speed or flask size or shape. An increase in temperature was shown to significantly increase extractable P levels using the Bray P1 procedure. Similarly, Soltanpour et al. (1976) found significant effects of extraction ratio (soil:solution), shaker speed, shaker type (rotary vs. reciprocal), and extraction vessel shape (Erlenmeyer vs. square bottle) on extractable Fe and Mn. Shaker speed also affected the extraction of Zn from soils. In general, the more thorough the mixing of soil and solution, the higher the solution concentration of Fe and Mn. No effect of temperature was found on the extraction of K by 1N ammonium acetate (Munter, 1988). As a result of this study, the North Central Regional Soil Testing Committee (NCR-13) recommended that soil samples should be extracted for P and K by shaking for five minutes at speeds of 160-210 excursions per minute. Erlenmeyer flasks (50 mL) were cited as the preferred extraction vessel, with Wheaton bottles given as an acceptable alternative. This was consistent with the recommendations of the Southern Regional Soil Testing and Plant Analysis Group (SRIEG-18), that recommended the use of 50 mL extraction bottles, and a shaking speed of 180 oscillations per minute. Although a similar study has not been conducted in the northeast, similar effects would be expected with northeastern soils and extracting procedures, since the same exchange and desorption mechanisms are involved. An ideal laboratory temperature range (24 to 27°C) should perhaps be adopted as part of recommended nutrient extraction procedures.

The relatively short extraction times used by soil testing laboratories to facilitate sample throughput can indirectly lead to another source of error. In contact time experiments at the University of Maine Analytical Laboratory, it has been shown that after only 5 minutes of contact, solution concentrations are still increasing at a rapid rate. Soltanpour et al. (1976) also noted that shaking time can affect the extraction of micronutrients Cu, Fe, Mn, and Zn. Even a moderate deviation from the prescribed shaking time for an extraction method, therefore, has the potential to significantly change the final extract concentration. The type of filter paper used will affect filtration speed and influence overall contact time. Since results from interlaboratory sample exchanges will be dependent upon contact time, soil:solution ratio, and filter paper speed

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as well as the type of extractant used, it is critical to standardize these factors for consistency between laboratories in the region, a primary goal of this bulletin.

### **Recommended Method for Drying Soils**

The exact procedure for drying is not critical as long as contamination is minimized and excessive temperatures are avoided. The recommended drying procedure for routine analysis is to dry the samples overnight, using forced air at ambient temperatures. Supplemental heating can be used, but Elk and Gelderman (1988) recommended that soil samples to be used for routine analyses not be dried at greater than 36°C. Microwave drying may alter the analytical results (Thien et al., 1978) and should be avoided.

If the sample is to be analyzed for nitrate, drying should be as rapid as possible, but no longer than overnight. This can easily be done by drying small sample volumes or spreading the sample out in thin (< 1 cm) layers. Drying for nitrate analysis can be done at temperatures as high as 105°C, but these samples should not be used later for routine nutrient analysis. It is also important to consider the recent history of samples submitted for nitrate analysis. If they have been stored at ambient temperatures for a considerable amount of time (e.g., delays in mailing samples to the laboratory), the nitrate results may be erroneously high.

### **Recommended Method for Crushing and Sieving Soils**

Because soil is defined as having a particle size of less than 2 mm, this sieve size (10 mesh) is recommended for routine soil testing. Various soil grinders and crushers are available commercially and are typically large, motorized, mortar and pestles, hammer-mills, or roller-crushers. The amount of coarse fragments common in some samples limits the use of some of these. In general, it is desirable to get most of the sample to less than 2 mm with the least amount of grinding. If the sample is to be analyzed for micronutrients, all contact with metal surfaces should be avoided during the crushing and sieving procedures unless it has been clearly demonstrated that the metal is not a source of contamination.

Cross-contamination between samples can be avoided by minimizing soil-particle carryover on the crushing and sieving apparatus. For macronutrient analysis, removal of particles by brushing or jarring should be adequate. If micronutrient or trace element analysis is to be performed, a more thorough cleaning of apparatus by brushing or wiping between samples may be required.

## **Recommended Methods for Subsampling Soils**

### **Mixing**

The bulk soil sample should be thoroughly homogenized by mixing with a spatula, stirring rod, or other implement. As much of the sample as possible should be loosened and mixed together. No segregation of the sample by aggregate size should be apparent after mixing. Dip into the center of the mixed sample to obtain a subsample.

### **Weighing**

Subsamples should be weighed into a tared vessel with a minimum precision of +/- 1% (e.g., 5.0 +/- 0.05 g). If a separate container is used to weigh and transfer subsamples to extraction vessels, it should be brushed out between samples to avoid cross-contamination. Recommended soil weights for routine testing procedures are provided in each section of this manual.

### **Scooping**

Dip into the center of the homogenized bulk sample with a standard soil scoop, filling it heaping full without pushing against the side of the soil container. Holding the scoop firmly, tap the handle three times with a spatula, two or three inches from the sample. Hold the spatula perpendicular to the top of the scoop and strike off the excess soil (Peck, 1988). If weight basis results are needed, empty the scoop contents into a tared weighing vessel and record the weight.

## **Recommended Method for Cleaning Glassware and Plasticware**

All containers that are to be reused for extraction, filtration, or solution analysis should be initially rinsed in warm tap water to remove any residual solution and soil particles. They should then be filled to the top or rinsed with an aluminum chloride solution (2 g  $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ /liter) (Tucker, 1985). Avoid using anhydrous  $\text{AlCl}_3$ , due to its violent reaction with water. A 4% HCl solution can also be used if Cl<sup>-</sup> is not to be analyzed. The rinsing solution can then be poured out and the containers rinsed and drained twice in distilled or deionized water and allowed to dry for reuse.

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