5. Soil, Plant Tissue and Manure Analysis

Profitable crop production depends on applying enough nutrients to each field to meet the requirements of the crop while taking full advantage of the nutrients already present in the soil. Since soils vary widely in their fertility levels, and crops in their nutrient demand, so does the amount of nutrients required.

Soil and plant analysis are tools used to predict the optimum nutrient application rates for a specific crop in a specific field.

Soil tests help:
- determine fertilizer requirements
- determine soil pH and lime requirements
- diagnose crop production problems
- determine suitability for biosolids application
- determine suitability for specific herbicides

Plant tissue tests help:
- determine fertilizer requirements for perennial fruit crops
- diagnose nutrient deficiencies
- diagnose nutrient toxicities
- validate fertilizer programs

Soil analysis

Handling and preparation
When samples arrive for testing, the laboratory:
- checks submission forms and samples to make sure they match
- ensures client name, sample IDs and requests are clear
- attaches the ID to the samples and submission forms
- prepares samples for the drying oven by opening the boxes or bags and placing them on drying racks
- places samples in the oven at 35°C until dry (1–5 days) (nitrate samples should be analyzed without drying)
- grinds dry samples to pass through a 2 mm sieve, removing stones and crop residue
- moves samples to the lab where sub-samples are analyzed

What’s reported in a soil test
Commercial soil-testing laboratories offer different soil testing/analytical packages. How the laboratory reports the results will also differ between labs. It is important to select an analytical package that meets your requirements. Analyses common to almost all soil test packages include: pH, buffer pH, phosphorus, potassium and magnesium.
Soil pH is included in almost all soil tests. Although it is not a nutrient, soil acidity or alkalinity has a great influence on the availability of nutrients and on the growth of crops. The buffer pH will also be reported for acid soils to determine the lime requirement.

The main nutrient analyses reported are phosphorus, potassium and magnesium. These represent the nutrients, aside from nitrogen, most commonly applied as fertilizer. Some labs include an analysis for calcium.

Nitrate-nitrogen analysis is performed on a separate soil sample taken to a greater depth.

Micronutrient tests are not performed as frequently but are becoming more popular. Zinc and manganese have tests that are well calibrated with crop requirements, and these are performed almost routinely. Other micronutrients (copper, iron, boron) are not well calibrated, but some reports include them.

Sulphur tests are becoming more common in Ontario as atmospheric depositions of sulphur from air pollution decrease, but they are not well calibrated with crop requirements.

More labs are routinely analyzing for organic matter. It is often used as an indicator of soil quality and also tied to herbicide recommendations.

Soils with suspected excess salts can be analyzed for electrical conductivity.

See the next page for more detail on what is included in a typical soil test report.

How the numbers are reported
Soil test results are expressed in many ways, particularly when dealing with labs from outside Ontario. Most Ontario labs express results as milligrams per litre of soil (mg/L): that is, the weight of nutrient extracted from a volume of soil. This is close in value to the weight-by-weight measure of milligrams per kilogram of soil (mg/kg), which is equivalent to parts per million (ppm).

Some labs, particularly in the United States, express soil test results as pounds per acre of available nutrient, which is confusing since the soil test results don’t reflect a physical quantity. An acre-furrow slice weighs about 2 million pounds. The results can be converted back to parts per million by dividing by 2. For example, if soil test phosphorus is 120 lb/acre, divide by 2 to get 60 ppm.

Quebec results are expressed as kg/ha. Use the following formula to convert these results to ppm:

\[ \text{kg/ha} \times 0.455 = \text{ppm} \]

It is also important to know which extractants have been used to perform the soil test.
### Information found on a soil test report

#### General Information
- **Sample number** — This is provided by the grower relating the sample results to a particular field.
- **Lab number** — This is assigned by the lab, to track the sample through the various analytical steps.

#### Analytical Values
- **Soil pH** — Every report should include soil pH, measured in a soil-water paste.
- **Buffer pH** — Buffer pH is only measured on acid soils (normally where soil pH < 6.0).
- **Phosphorus (ppm)** — Ontario accredited soil tests must include the results from the sodium bicarbonate extraction (Olsen method). Some labs will also include results from Mehlich or Bray extractions. The method and the units should always be shown.
- **Potassium, magnesium (calcium, sodium) (ppm)** — The cations are measured in an ammonium acetate extract, with the results reported as mg/L of soil or ppm. Calcium and sodium are sometimes also reported.
- **Nitrate-N (ppm)** — This is not part of a regular soil test, since the interpretation of results is only valid for a deeper sample taken at planting time or before side-dressing.
- **Sulphur (S) or sulphate (SO₄-S) (ppm)** — This optional test has not been calibrated. It should be used on deeper samples, similar to nitrate.
- **Micronutrients (ppm)** — Mn and Zn are the only micronutrients with an Ontario accredited test. These are reported as a manganese index and a zinc index (see “Derived Values” below). Values may be reported for other micronutrients, but Ontario research has not shown reliable correlation to plant availability.
- **Organic matter (%)** — This is an optional test. Note carefully whether the result reported is for organic matter or organic carbon.
- **Electrical conductivity (EC) (millisiemens/cm)** — This optional test indicates the presence of excessive salts in the soil.

#### Derived Values
- **Zinc and manganese index** — These are calculated from the analytical result and the soil pH.
- **Cation exchange capacity (CEC) and base saturation %** — These numbers are calculated from the soil pH and analytical results for K, Mg and Ca. They may be skewed in high pH soils by the presence of free lime. Ontario fertilizer recommendations are not affected by CEC or base saturation.

#### Nutrient Recommendations
- **Fertilizer and lime recommendations** — These will only be printed if information about the crop to be grown has been provided. The analytical results can be used to determine nutrient requirements for specific crops from tables in the appropriate production recommendations. Some labs will give Ontario recommendations where requested. Often the labs will provide their own recommendations.
- **Adjustments to fertilizer recommendations** — Adjustments for manure application or for a previous legume crop will be included in the fertilizer recommendations if the information is provided.
- **Notes and warnings** — Some reports will include additional information based on the crop and soil test data.

**Note:** Ratings for soil test values are based on the soil test result and the crop to be grown.
**Extractants**

Analyzing soils to determine fertilizer requirements is complicated because we are trying to estimate how much nutrient is available from a specific soil to a wide variety of crop plants throughout the entire growing season. This would be simple if soils had uniform nutrient distribution, all the nutrients were wholly available for plant uptake and there were only one method by which plants took up nutrients. However, soil is an extremely complex medium with a wide variety of physical, chemical and biological interactions occurring simultaneously. The interactions at the soil-root interface are even more complex and less well understood.

An example of this complexity is phosphorus. The most common chemical form of phosphorus in the soil is phosphate. In neutral-to-alkaline soils, phosphate will combine with calcium. In neutral-to-acid soils, it will bind to iron or aluminum. Phosphate also reacts with various clay minerals or organic compounds to form complex combinations, and it may be present in the organic fraction of the soil or the soil biomass. All these forms are available to a greater or lesser degree to plants through a variety of processes, which we try to measure with a single, rapid chemical test.

Every chemical analysis has two steps. First, the compound being analyzed is converted to a form that can be measured. Then this material is analyzed. However, because we are estimating only the available portion of the nutrient in the soil, the first step differs from a normal chemical analysis. In the case of soil tests, the soil is first treated with an extractant to remove a portion of the nutrient that is related to the amount available to plants. This extract is then analyzed to determine the amount of nutrient that was extracted.

**Choosing an extractant**

To be useful in predicting crop needs, an extractant must provide the best possible estimate of the amount of additional nutrient needed for optimum crop yields. This is complicated to measure, so the assessment of extractants is more commonly made by measuring how well the extractant estimates the nutrients available to plants in the range of soils tested in the lab or in a region. The extractant must also be relatively inexpensive and easy to use, involve as few toxic or corrosive chemicals as possible and use procedures that are reproducible from lab to lab.

No extractant pulls out the exact fraction available to plants. Each has strengths and weaknesses specific to various soils. The choice of an extractant should be governed by how appropriate it is to the soils in question and by the availability of data relating it to crop response. See Chapter 7 for more details.
First was water
The first extractant used for soil testing was water. This removed only the portion of nutrient present in the soil solution. While this fraction is immediately available to plants, it is only a tiny part of the total available nutrient in the soil. It is not well related to the total nutrient supply, since soils vary tremendously in the nutrient reserve they hold.

Researchers had noted that plant roots excrete weak acids from their surfaces, so the next step was to experiment with acid solutions. From there, the range and variety of extractants has proliferated as researchers seek better and more appropriate extractants for a wide range of soil conditions. These extractants are often named for the scientist who developed it or the main ingredient in the extracting solution.

Regionally specific
The choice of an extractant is specific to each region, since the most appropriate extractant depends to a large extent on the soils of that region.

The first step in determining an appropriate extractant or soil test method is to collect samples of a wide range of soils from across the region and then to grow plants in each soil. These plants are harvested, weighed and analyzed to find the amount of nutrient taken up by the plants from the different soils.

Different extractants are used to remove nutrients from the soils, and the extracts are analyzed. The final step compares the results of the extractions with the amount taken up by the plants, which is the measure of the nutrient-supplying capacity of the soil. The extractant that is chosen for a region is normally the one with the highest correlation (agreement) to the plant uptake.

Soil test extractants for phosphorus can be broadly divided into acidic and alkaline solutions. The acidic solutions (used in the Bray and Mehlich methods) are generally used in areas with acidic soils. In alkaline soils, these extractants underestimate the amount of available phosphorus because the acid is partly neutralized by the free lime in the soil. See Table 5–1.

The alkaline extractants (sodium bicarbonate, ammonium bicarbonate) give more consistent results over a wide range of soil pH. Potassium, calcium and magnesium are extracted using another similar cation, usually ammonium, to remove them from the cation exchange complex. Micronutrients may be extracted using a chelating agent or weak acid to remove them from the soil.
Table 5–1. Correlation of extractable P with P uptake in controlled greenhouse conditions

<table>
<thead>
<tr>
<th>Extractant</th>
<th>All soils(^1) correlation ((r^2))</th>
<th>pH&gt;7.0(^2) correlation ((r^2))</th>
<th>pH 6.1–7.0(^3) correlation ((r^2))</th>
<th>pH&lt;6.1(^4) correlation ((r^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td>sodium bicarbonate</td>
<td>0.74</td>
<td>0.79</td>
<td>0.64</td>
<td>0.87</td>
</tr>
<tr>
<td>ammonium bicarbonate</td>
<td>0.73</td>
<td>0.71</td>
<td>0.63</td>
<td>0.95</td>
</tr>
<tr>
<td>Bray-Kurtz P1</td>
<td>0.54</td>
<td>0.52</td>
<td>0.33</td>
<td>0.73</td>
</tr>
<tr>
<td>Bray-Kurtz P2</td>
<td>0.65</td>
<td>0.60</td>
<td>0.40</td>
<td>0.90</td>
</tr>
<tr>
<td>Mehlich III</td>
<td>0.66</td>
<td>0.57</td>
<td>0.40</td>
<td>0.93</td>
</tr>
</tbody>
</table>

\(^1\) n = 88 soils  
\(^2\) n = 46 soils  
\(^3\) n = 30 soils  
\(^4\) n = 12 soils

*An \(r^2\) of 1.00 is complete agreement.


Following the choice of extractant, field trials are carried out to determine the optimum fertilizer application for each soil test level with different crops. These calibrations are unique to the extractant and are expensive. Inevitably, there is resistance to changing the soil test extractant unless an alternative method has a large advantage.

**Extractant results are not interchangeable**

Different extractants will often give widely different values from the same soil. The amount of phosphorus extracted by a sodium bicarbonate solution, for example, may be one half or less of that extracted by a Bray P1 extractant. In the proper conditions, however, both could provide an index of phosphorus availability to crops. Problems arise if someone uses the numbers from one test with fertilizer recommendation tables developed for a different test.

The results from different extractants are not related perfectly to one another. While there is a trend that as the soil test level for one extractant increases, the others increase as well, there are exceptions. Even where the extractants increase consistently, the relationship between extractants is often different at low soil test values than at high soil test values. For this reason, converting values from one extractant to another should be avoided. Know which extractant is being used and use those results with fertilizer recommendation tables developed for that extractant.

**Quality control**

As with any chemical process, quality control must be used to ensure that results from each lab are accurate. This is accomplished in Ontario through an accreditation program administered by the Ontario Ministry of Agriculture, Food and Rural Affairs. The details of this program will keep changing over time, but the basic principles will remain the same.
Goals of a lab accreditation program

The goals of a lab accreditation program are to:

- ensure that participating labs complete analytical tests that fall within the range of expected results of the accreditation program
- provide consistent results from any of the accredited labs
- encourage the use of appropriate soil test extractants (see Table 5–2) for which there is a body of fertilizer response calibration data for Ontario soils
- promote the use of accredited labs
- promote the use of fertilizer guidelines based on Ontario research

Accredited labs follow a quality control program that ensures best results. Each lab has one or two standard soils that are included in each analytical run to ensure the results are consistent. Standard solutions are prepared carefully and used to calibrate the instruments and to check their calibration periodically. Recordkeeping and tracking are used for troubleshooting problems and ensuring the performance of the lab over time.

An external assessment program provides an additional check on the system. This allows comparison between labs and helps catch any problems that have been overlooked by the lab’s internal quality control.

<table>
<thead>
<tr>
<th>Tested for</th>
<th>Testing method</th>
</tr>
</thead>
<tbody>
<tr>
<td>soil pH</td>
<td>saturated paste</td>
</tr>
<tr>
<td>lime requirement</td>
<td>SMP buffer pH</td>
</tr>
<tr>
<td>phosphorus</td>
<td>sodium bicarbonate (Olsen)</td>
</tr>
<tr>
<td>potassium, magnesium</td>
<td>ammonium acetate</td>
</tr>
<tr>
<td>zinc index</td>
<td>DTPA, modified by soil pH</td>
</tr>
<tr>
<td>manganese index</td>
<td>phosphoric acid, modified by soil pH</td>
</tr>
<tr>
<td>soil nitrate</td>
<td>potassium chloride extraction</td>
</tr>
</tbody>
</table>
History of soil test accreditation in Ontario

In 1989, it was proposed that instead of a single OMAFRA-recognized lab for soils, feed, plant tissue and greenhouse media analysis, all labs that could show proficiency in analyses for these substrates would be recognized. As a result, 33 labs showed interest in the accreditation program.

OMAFRA personnel visited each lab and provided the Ontario Soil Management Research and Service Committee methods for soil analyses. Also, staff took a list of analytical equipment and lab-tracking and quality-control methods. The labs also analyzed a number of soil samples in triplicate and had to meet standards for analytical accuracy.

To be accredited, a lab had to perform well in the areas of pH, buffer pH, phosphorus, magnesium and potassium. Optional accreditation could be obtained for zinc and manganese indices.

Three labs were accredited in 1989. In 1991, a new accreditation exercise was completed with five sets of soil, each set randomized separately. In 1998, Ontario joined the North American Proficiency Testing (NAPT) program. While this means that some of the program samples will come from areas with soils that are not representative of Ontario soil, this is more efficient and allows for greater harmonization of labs. Sample exchanges are conducted 4 times per year, with five soils per exchange that the labs analyze 3 times over 3 days. Labs must maintain acceptable accuracy in all the accredited methods to retain their accredited status.

New labs can be accredited provided they demonstrate acceptable accuracy on the NAPT exchange samples, as well as a series of independent samples with known values. In 2005, Ontario had six accredited soil labs and as of 2018, eight labs were accredited province-wide.

Soil pH

Soil pH is the measurement of the hydrogen ion activity or concentration in the soil solution. This activity affects the availability of most nutrients and controls or affects most biological processes.

The hydrogen ion concentration is measured with a pH electrode. The heart of the electrode is a glass bulb that is only porous to hydrogen ions. As the positive ions move into the electrode, a current is set up that is measured with what is essentially a voltage meter. The voltage reading of several standards is read and a graph set up. The voltage readings of the samples are then compared to the graph and given pH values.

There is some debate about what soil-to-water ratio is best for measuring pH. Usually, soil pH is measured using de-ionized water to form a saturated paste or a 1:1 or 1:2 soil-to-water ratio. Saturated paste is the accredited method in Ontario, and liming recommendations are based on this method. The measured pH tends to increase as the amount of water added to the soil increases. The difference will be greatest in the soils with the lowest buffering capacity: i.e., coarse sands.
Other methods employ calcium chloride solutions to prepare the paste or slurry, reducing the amount of interference from high salt levels. This method tends to give a lower pH reading than slurry with pure water.

The saturated paste is prepared by adding just enough water to the soil sample to completely saturate it without leaving any free water. Properly preparing a saturated paste is time-consuming and difficult, but it provides a closer approximation of the pH at the root-soil interface than the more dilute slurries.

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**The little “p” in pH**

In math, “p” is used to denote the negative log of a given value. In the case of pH, it is the negative log of hydrogen ion (H⁺) concentration in the solution.

Pure water contains some molecules that have broken apart into individual ions, either hydrogen (H⁺) or hydroxyl (OH⁻). **water (H₂O) = H⁺ + OH⁻**

In pure water, there is an equal amount of hydrogen and hydroxyl ions, and the pH is neutral (see Figure 3-1). If you were to count the number of H⁺ ions in pure water, you would find **1/10,000,000 moles of H⁺ ions per litre of water**.

In scientific notation, this is **10⁻⁷ H⁺ ions**, and the negative log of this number is the positive value of the little number on top, or 7. As the concentration of hydrogen ions increases, the value of the pH decreases and the solution becomes more acidic.

Since this is a logarithmic scale, a pH of 6 is 10 times more acid than a pH of 7. A pH of 5 is 10 times more acid than a pH of 6, and 100 times more acid than a pH of 7.

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**Buffer pH**

**Shoemaker, McLean and Pratt (SMP) method**

The measurement of soil pH is used to indicate whether a field requires lime. Depending on the crop, soils with a pH less than 6.1 need lime, and a buffer pH measurement is performed to determine how much lime is required.

The buffering capacity of the soil is its ability to resist changes in pH. In an acid soil, this ability to resist change is due to the reserve acidity. This reserve acidity is due to hydrogen, aluminum and other cations that are held on the cation exchange complex. The greater the reserve acidity, the more lime is required to bring the pH into optimal range.

This reserve acidity is measured by adding a buffer solution (SMP) to the soil sample and reading the pH of the soil and buffer mixture after a half hour. This buffer resists change in pH and starts out at a pH of 7.5, but the soil acidity reduces the pH of the buffer in proportion to the amount of reserve acidity in the soil. If the pH of this mixture is low, the soil has a high reserve acidity and requires a large amount of lime to neutralize it.

The lime requirement is calculated according to formulas in Table 5–3.
Table 5–3. Calculating lime requirements

<table>
<thead>
<tr>
<th>pH to which soil is limed</th>
<th>Equation</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.0</td>
<td>lime (t/ha)* = $334.5 - 90.79 \text{pH}<em>{B} + 6.19 \text{pH}</em>{B}^2$</td>
</tr>
<tr>
<td>6.5</td>
<td>lime (t/ha) = $291.6 - 80.99 \text{pH}<em>{B} + 5.64 \text{pH}</em>{B}^2$</td>
</tr>
<tr>
<td>6.0</td>
<td>lime (t/ha) = $255.4 - 73.15 \text{pH}<em>{B} + 5.26 \text{pH}</em>{B}^2$</td>
</tr>
<tr>
<td>5.5</td>
<td>lime (t/ha) = $37.7 - 5.75 \text{pH}_{B}$</td>
</tr>
</tbody>
</table>

* Lime requirement is calculated at tonnes of lime per hectare with an agricultural index of 75 (see Chapter 3, Table 3–2, for more details).

** $\text{pH}_{B}$ = buffer pH

Example calculation. Determine the lime requirement for a soil with a buffer pH ($\text{pH}_{B}$) of 6.5 in order to achieve a desired pH of 7.0:

$$334.5 - (90.79 \times 6.5) + 6.19 \times (6.5)^2 = 5.9 \text{ t/ha lime required}$$

Soluble salts

Soluble salts in soils can result from excessive applications or spills of fertilizers and manures, runoff of salts applied to roads and chemical spills. There can also be high salt levels in areas affected by brine seeps or spills from recent or historical oil and gas exploration. High concentrations of soluble salts in or near a fertilizer band can restrict plant (root) growth severely without seriously affecting the salt concentrations in the rest of the soil. It is difficult to identify excess salts in a starter fertilizer band because of the limited volume of soil affected and because the excess salts can dissipate quickly into the surrounding soils with rainfall.

Soluble salts also interfere with the uptake of water by plants. A given amount of salt in a soil provides a higher salt concentration in soil water if the amount of water is small. Plant growth is most affected by soluble salts in periods of low moisture supply (drought) and in soils with low water-holding capacity (e.g., sands and gravels).

Soluble salts can be measured in the lab by measuring the electrical conductivity of a soil-water slurry. The higher the concentration of water-soluble salts, the higher the conductivity. Table 5–4 provides an interpretation of soil conductivity reading for Ontario field soils in a 2:1 water:soil. This slurry is prepared by mixing one volume air-dried soil with two volumes of water.

For greenhouse soils, the OMAFRA-accredited soil test uses a larger soil sample and measures conductivity on a saturation extract.
Table 5–4. Interpreting soil conductivity readings in field soils

<table>
<thead>
<tr>
<th>Conductivity “salt” reading millisiemens/cm</th>
<th>Rating</th>
<th>Plant response</th>
</tr>
</thead>
<tbody>
<tr>
<td>0–0.25</td>
<td>L</td>
<td>suitable for most if recommended amounts of fertilizer used</td>
</tr>
<tr>
<td>0.26–0.45</td>
<td>M</td>
<td>suitable for most if recommended amounts of fertilizer used</td>
</tr>
<tr>
<td>0.46–0.70</td>
<td>H</td>
<td>may reduce emergence and cause slight to severe damage to salt-sensitive plants</td>
</tr>
<tr>
<td>0.71–1.00</td>
<td>E</td>
<td>may prevent emergence and cause slight to severe damage to most plants</td>
</tr>
<tr>
<td>1.00</td>
<td>E</td>
<td>expected to cause severe damage to most plants</td>
</tr>
</tbody>
</table>

**Testing for nitrate-nitrogen**

Nitrate-nitrogen content of the soil at planting time can be used to fine-tune nitrogen fertilizer applications for corn and spring barley or for nitrogen applications to corn at sidedress timing (pre-sidedress nitrate test or PSNT). Extensive calibration work has not been carried out in Ontario for other nitrogen-using crops such as wheat, canola or most horticultural crops. Work has been done with potatoes and tomatoes, but results did not lead to definite recommendations.

Routine nitrogen analysis is not done on soil samples because nitrate contents vary greatly from week to week; nitrate-nitrogen samples are taken to a greater depth than standard soil tests; and samples must be handled carefully to prevent changes in the soil nitrate content.

Some users request analysis for ammonium nitrogen as well as nitrate, even though it is not used for recommendations. The same extraction method is used, although a different analytical procedure is used on the extract. If the sample is to be analyzed for ammonium, it should be refrigerated. Drying the sample will invalidate the ammonium nitrogen test (see Chapter 4, Sample collection section).

**Methods**

Nitrate-nitrogen is present in the soil almost exclusively within the soil solution and is extracted easily. The standard extractant used is a potassium chloride solution.

A sample of the soil is mixed with the potassium chloride solution at a ratio of 1 part soil to 5 parts extracting solution, shaken for half an hour and then filtered. The extract is analyzed using an auto-analyzer, which measures the intensity of colour produced after mixing the extract with specific chemicals.

Portable field sensors are becoming available commercially. Careful operation and calibration by the user needs to be fully understood. These sensors do provide rapid on-site analysis and reduce the costs of couriering samples to commercial labs. It is advised to participate in a check sample program to verify equipment performance and verify results.
Comments

- This method produces highly reproducible results and is relatively straightforward.
- Soil nitrate values generally increase by 30% from early May (pre-plant timing) to early June (pre-sidedress timing). Ensure that the soil test lab is aware of your sample timing.
- Interpretation of the soil nitrate test is complicated by the variability of soil nitrate contents within the field.
- Soil nitrate content may underestimate the amount of available nitrogen where organic sources of nitrogen have been applied (e.g., livestock manure, sewage sludge, legume plowdown) and have not had a chance to mineralize. Research is under way to develop soil tests for the easily mineralizable portions of soil and added organic matter.

Phosphorus

The three common methods for extracting available phosphorus are Olsen (sodium bicarbonate), Bray P1 and Mehlich III (see Table 5–5).

Whatever methodology is used, the next step is to determine the concentration of phosphorus in the extract. Several analytical methods can be used, some of which are related to a specific extractant. The most common involves adding molybdenum as a colour reagent. It will form a blue colour when combined with phosphorus. The greater the concentration of phosphorus, the more intense the colour.

The Olsen extractant is very alkaline, so it tends to react differently with the colour complex than the Bray or Mehlich do. As well, the Bray or Mehlich extracts tend to have higher phosphorus concentrations than the Olsen, so the standards used in the analysis are different.

<table>
<thead>
<tr>
<th>Method</th>
<th>Extracting solution</th>
<th>Solution pH</th>
<th>Where it’s used</th>
</tr>
</thead>
<tbody>
<tr>
<td>sodium bicarbonate</td>
<td>0.5 M NaHCO₃ solution, 1 part soil to 20 parts solution, shaken for 30 min at room temperature</td>
<td>8.5</td>
<td>Ontario, Iowa, most western states</td>
</tr>
<tr>
<td>Bray P1 (weak Bray)</td>
<td>0.025 M HCl + 0.03 M NH₄F, 1 part soil to 10 parts solution, shaken for 5 min</td>
<td>2.5</td>
<td>Michigan, Ohio, Indiana, Illinois, eastern states</td>
</tr>
<tr>
<td>Bray P2 (strong Bray)</td>
<td>0.1 M HCl + 0.03 M NH₄F, 1 part soil to 10 parts solution, shaken for 5 minutes</td>
<td>2.5</td>
<td>early 1960s in Ontario before sodium bicarbonate</td>
</tr>
<tr>
<td>Mehlich III</td>
<td>0.2 M CH₃COOH + 0.25 M NH₄NO₃ + 0.015 M NH₄F + 0.013 M HNO₃ + 0.001 M EDTA, 1 part soil to 10 parts solution</td>
<td>2.5</td>
<td>Quebec, Maritime provinces, Pennsylvania, southeastern states</td>
</tr>
</tbody>
</table>
**Sodium bicarbonate method (Olsen)**

The sodium bicarbonate method (also called the Olsen method) is the one recommended for use in Ontario. This extracting solution has a pH of 8.5 and so is best used with a soil pH range from 6.0–8.0. The calcium phosphates in the soil and some of the organic phosphates are dissolved by the sodium bicarbonate. The sodium bicarbonate method will predict the relative available phosphorus in a wide range of soil types.

**Comments**

- Requires a longer shaking time than the Mehlich or Bray (a half hour, as opposed to 5 minutes).
- The sodium bicarbonate method is very sensitive to temperature, pH and shaking times, so that uniform conditions are required throughout the analysis to ensure consistent results. Olsen found that the extractable phosphorus can increase almost 0.5 ppm for a 1°C increase in temperature of the extracting solution between 20°C and 30°C.

**Bray P1**

The Bray extraction solution contains hydrochloric acid and ammonium fluoride, which form an acidic solution. This tends to simulate an acid soil environment. This test is better for acidic than for alkaline soils.

The Bray extractant tends to extract more phosphorus than the sodium bicarbonate method. At high pH values, the acid nature of the extracting solution may dissolve the calcium phosphates, over-estimating the available phosphorus. However, the free lime in the soil may also neutralize the acid nature of the extracting solution, making it less effective. These two situations indicate that the Bray P1 extraction provides unpredictable results under alkaline conditions.

A modified Bray P2 (strong Bray) extractant was used in Ontario during the 1960s, using a more concentrated acid to overcome the neutralizing effect of alkaline soils. It was replaced by the sodium bicarbonate extractant, which was more consistent over the range of soils in Ontario.

**Mehlich III**

The Mehlich III is a multi-element extracting solution composed of acetic acid, ammonium fluoride, ammonium nitrate and the chelating agent ethylene diamine tetra-acetic acid (EDTA). It combines chemicals from Bray P1, ammonium acetate and DTPA extracting solutions.

Mehlich III extracts phosphorus with acetic acid and ammonium fluoride. It extracts potassium, magnesium, sodium and calcium with ammonium nitrate and nitric acid and extracts zinc, manganese, iron and copper with EDTA.
This method is often used because of the savings in analysis time. When used with an inductively coupled plasma (ICP) machine capable of running simultaneous elements, this method is appealing for soil labs. The value measured using an ICP may be different from the value measured by a colour reaction, although the reasons for this are not clear. These should be considered to be two separate tests, with different interpretations for making fertilizer recommendations.

Because of its acidic nature, the Mehlich III solution is best suited to acidic soils and is routinely used in Quebec and the Maritimes. The relatively high acid concentration in this extractant means it will perform adequately in slightly alkaline soils, but inconsistently in soils with high carbonate (free lime) content.

**Potassium, calcium, magnesium, sodium**

Potassium, magnesium, calcium and sodium are positively charged. They are all cations. They can all be extracted by the same solution, since the mechanism is to flood the soil with another cation to displace them from the exchange complex.

Potassium and magnesium are the cations that most often limit crop production, and they are measured routinely in Ontario. Potassium is absorbed by the plant in larger quantities than any other element except nitrogen.

Calcium supply is generally adequate if the soil pH is suitable for crop growth, so it is not measured by all labs. Calcium contents are often high enough that extra dilutions are required to bring the extract within the operating range of the lab equipment, adding extra time and inconvenience.

Sodium is not an essential nutrient for crop production and is analyzed only where environmental contamination is suspected.

The presence of free lime in calcareous soils complicates the measurement of calcium and magnesium. This free lime is partly dissolved by the ammonium acetate solution and causes extra calcium and magnesium to show up in the extract. The amount of lime dissolved will depend on the pH of the extracting solution and the ratio of soil to extracting solution, so it is important for labs to follow analytical procedures exactly.

**Ammonium acetate**

The most common cation used for extracting soil cations is ammonium from ammonium acetate.

The availability of potassium is influenced by the drying temperature of the soil. Temperatures higher than 35°C tend to cause the potassium to be bound up on the exchange sites. This is the reason that at least two days of lab time is spent drying. Speeding up the process would either leave water in the soil, affecting the final concentration of the nutrients,
or over-heat the soil, making the readings for potassium inaccurate.

After extraction, the cations in the ammonium acetate solution are measured.

**Mehlich III**

The Mehlich III extractant can be used for potassium and other cations as well. The ammonium ions from ammonium nitrate and ammonium fluoride behave the same way as the ammonium from ammonium acetate, displacing the cations from the exchange sites. The concentration of the cations are then measured in the extract.

The Mehlich III method extracts amounts of potassium from the soil comparable to the amounts extracted by the ammonium acetate method.

**Sulphur**

There is no standard accepted sulphur soil test or calibrated sulphur fertilizer recommendations for Ontario. Soil test labs in Ontario have not routinely analyzed for sulphur in the past. Sulphur concentration in the soil is affected by leaching and mineralization, which make it difficult to correlate soil test values to plant uptake. It is likely that sulphur test results will be more meaningful from a 30 cm sample rather than a 15 cm sample.

Labs will do sulphur analyses on request. The most common technique is to extract sulphur from the soil using a calcium phosphate solution. The amount of sulphate in the extract is measured by adding barium to form barium sulphate crystals and measuring the turbidity of the resulting suspension or by reducing the sulphate to sulfide and measuring it through a colour-forming reaction. Other labs may analyze sulphate-S in the calcium phosphate extract or in a Mehlich III extract using an ICP.

**Micronutrients**

Because micronutrients are generally found in extremely low levels in the soil, estimates of their concentrations are generally less reliable than the measurement of macronutrients.

Micronutrient tests are difficult to correlate with plant uptake because:

- the concentrations in the extracting solutions may be near the detection limit of the equipment
- there is potential for contamination of the sample from sampling tubes, pails or dust
- soil pH, organic matter, clay content and mineralogy can affect both the extractions and the plant availability of micronutrients

In Ontario, tests have been accredited for zinc and manganese. The other micronutrients are not well enough correlated to be used for fertilizer recommendations. Tissue analysis should be the primary tool in diagnosing deficiencies of these elements. The soil test can be useful, however, as a secondary tool.
**Micronutrient extraction**

Most of the micronutrients are chemically active and would form insoluble compounds with an extracting agent, making them difficult to measure. Chemists get around this by using chelates or weak acids to extract micronutrients. Chelates are organic compounds that “complex” the micronutrient metal ions, binding to the ion at more than one point and wrapping themselves around it. This keeps the ions in the solution and allows them to be separated from the soil for measurement.

The most common chelating agents are diethylene triamine penta-acetic acid (DTPA) and ethylene diamine tetra-acetic acid (EDTA). While both behave similarly, they have slightly different affinities for different metal ions.

By varying the pH, chelating agents can be adjusted to extract specific nutrients. DTPA is adjusted to a pH of 7.3 for most soil extractions. Triethanolamine is added to the extracting solution to buffer it against pH changes during the extraction. Calcium chloride is also added to prevent the calcium carbonate in calcareous soils from dissolving.

**Zinc**

**DTPA extraction**

For this extraction, the soil is mixed with a 0.005 M DTPA solution at a ratio of 1 part soil to 2 parts solution and shaken for 1 hr. The zinc in the soil is complexed by the DTPA and held in the solution.

Following extraction and filtering, the zinc content in the extract is measured.

**Comments**

- The extraction process does not reach equilibrium, so it is necessary to maintain strict procedures with regard to shake time, speed and filtering for the tests to be consistent.
- The high soil-to-solution ratio (1:2) makes it difficult to filter out adequate sample sizes. Filtration may take several hours to overnight.
- The long shake and filtration time makes DTPA extraction one of the most time-consuming processes in the lab.
- This analysis is susceptible to contamination during the soil sampling process. In the field, be sure to use only plastic or stainless-steel equipment. The use of galvanized or iron implements will contaminate the sample with zinc or iron.
Chapter 5. Soil, Plant Tissue and Manure Analysis

**Zinc Availability Index**
The availability of zinc is influenced more by soil pH than by the amount of nutrient in the soil. Soil tests in Ontario for zinc report an availability index instead of, or in addition to, the nutrient analysis.

Formula to calculate the zinc index:
Zinc index = 203 + (4.5 x DTPA extractable zinc in mg/L soil) – (50.7 x soil pH) + (3.33) x (soil pH)^2

Interpreting the Index
- greater than 200 — suspect contamination of the sample or field
- 25–200 — adequate for most field crops
- 15–25 — adequate for most field crops but bordering on deficiency for corn
- less than 15 — likely deficient for corn and zinc fertilizer should be applied

**Mehlich III extraction**
The EDTA in the Mehlich extractant behaves much like DTPA. There has not, however, been as much work done with the Mehlich extractant in Ontario, so its results should be used with caution.

**Manganese**

**Phosphoric acid extraction**
In Ontario, a weak phosphoric acid solution is used as an extracting solution with a 1:10 soil-to-water ratio. Other areas may use the DTPA extractant, but the phosphoric acid method has given more consistent results in Ontario.

**Manganese Availability Index**
The availability of manganese is influenced much more by soil pH than by the amount of nutrient in the soil. Soil tests in Ontario for this nutrient report an availability index instead of, or in addition to, the nutrient analysis.

The values are indices of manganese availability based on phosphoric acid extractable soil manganese and soil pH.

Where soil pH ≤ 7.1:
Mn Index = 498 + (0.248 x phosphoric acid extractable Mn in mg/L soil) – (137 x soil pH) + (9.64) x (soil pH)^2

Where soil pH > 7.1:
Mn Index = 11.25 + (0.248 x phosphoric acid extractable Mn in mg/L)

Interpreting the Index
- greater than 30 — adequate for field crops
- 15–30 — adequate for most field crops but approaching deficiency for oats, barley, wheat and soybeans
- less than 15 — likely insufficient for oats, barley, wheat and soybeans.
Iron and copper
Neither iron nor copper has a soil test that correlates well with plant uptake or fertilizer response in Ontario. Copper deficiency has been observed on muck soils in Ontario but is rare on mineral soils. There are no confirmed cases of iron deficiency in Ontario.

Plant analysis is a much more reliable indicator of the availability of these nutrients.

Boron
There is no accredited test for boron in Ontario. To give a rough indication of availability, boron can be determined by extracting with hot water using barium chloride to flocculate the soil. Boron in the extracting solution can be read using a colour-forming reaction or ICP.

Because levels of boron are often less than 1 ppm, it is much more difficult to get an accurate measurement than it is for other soil nutrients. As well, the borate ion is mobile in the soil so that concentrations fluctuate, depending on leaching and mineralization.

Plant tissue analysis is a much more sensitive indicator of boron availability than a soil test.

Organic matter
Soil organic matter content is not used to adjust fertilizer recommendations in Ontario, but it plays an important role in soil fertility.

Organic matter contributes to the soil’s cation exchange capacity and enhances its ability to hold nutrients available for plant uptake. Through microbial action, many nutrients also cycle through organic and mineral forms, so that organic matter is a reservoir of slowly available nutrients. Adequate organic matter is essential for soil tilth and water-holding capacity. The level of organic matter is also important for the activity of several herbicides.

Determining soil organic matter has taken on new importance with the need to understand the dynamics of soil carbon in relation to greenhouse gas emissions or sequestration. Soil management can influence the net movement of carbon into or out of the soil, and this can create opportunities for farmers to participate in carbon credit programs. Evaluation of the effectiveness of these programs will require precise measurements of changes in soil organic matter content.

There are two approaches to measuring soil organic matter:

• The first is to measure the amount of organic carbon in the soil, using either wet chemistry or a combustion analyzer, and to multiply this weight by a factor to convert it to organic matter.
• The second approach is direct measurement of the weight of organic matter lost from the soil when it is burned, called loss on ignition (LOI).

Organic carbon measurements are more precise than LOI, particularly on soils with low organic matter contents, but they require either aggressive chemicals to dissolve the organic compounds or specialized equipment.

The measurements of organic matter and organic carbon are fairly well correlated, but the carbon content of organic matter can vary depending on the source and age of the material. This will lead to slightly different measurements, depending on the method used.

In Ontario, the loss on ignition method has been determined to be sufficiently precise for farm soils. Most scientific research, however, uses the increased precision of organic carbon determinations. Soil organic matter content is about 1.8 to 2.0 times the organic carbon content.

**Determining organic carbon**

**Modified Walkley Black**
The Walkley Black method operates on the principle that potassium dichromate oxidizes soil carbon. The potassium dichromate changes colour depending on the amount it is reduced, and this colour change can be related to the amount of organic carbon present. The final solution is read on a spectrophotometer and compared to a chart or a standard.

**Comments**
• This method measures organic carbon rather than organic matter. The conversion factor itself may be a source of error. Also, some organic compounds are not completely oxidized by the dichromate, resulting in low test values.
• This method cannot be used with soils containing over 7.5% organic matter.
• The reagents used in this analysis are toxic and must be disposed of as hazardous waste.

**Combustion furnace**
This furnace burns the sample at a temperature of more than 900°C and measures the concentration of carbon dioxide released — the total carbon. The results are fast and accurate, but the equipment is expensive.

Then, another sample is ashed overnight in a muffle furnace to remove the organic carbon. The inorganic carbon (carbonate) in the residue is measured. Organic carbon is the difference between the total carbon and the inorganic carbon.
**Soil texture**

**Texture estimation**

Soil texture is not measured in most soil samples but is estimated by hand. Soil texture is recorded on most soil reports as a letter. The four categories are C (coarse), for sand or sandy loam; M (medium), for loam; F (fine), for clay or clay loam; and O (organic).

These are used only to give the client a rough idea of the texture. It is sometimes a useful check that the samples are from the right fields.

**Texture measurement**

Soil texture can be measured by dispersing the soil in a high-sodium solution such as Calgon or triple sodium phosphate and measuring the amount of soil settling out over time. This method is based on the fact that large particles will settle out faster than finer ones. Between half a minute and 1 minute after agitation, all the sand will have settled. Between 6 hr and 24 hr after, all the silt will have settled out, leaving the clay in suspension. The technique uses a pipette or hydrometer to measure the concentration of soil in suspension at these times.

The technician uses a pipette to sample the solution. The solution from the pipette is dried in an oven and the amount of soil in the pipette is determined by weight.

Alternatively, the technician can use a special hydrometer to measure the density of the suspension. As the soil settles out of suspension, the density decreases and the hydrometer sits lower in the water.

The pipette method is more accurate than the hydrometer method but more expensive and time-consuming.

Ordinarily, organic matter does not significantly affect the texture measurement. An amount for organic matter can be deducted from the silt or clay fraction. Or, before determining texture, the organic matter can be removed by chemical means.

Once the proportions of sand, silt and clay have been determined, the texture class is determined as shown in Figure 5-1.

<table>
<thead>
<tr>
<th>Particle sizes of the soil fractions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sand 0.05–2 mm</td>
</tr>
<tr>
<td>Silt 0.002–0.05 mm</td>
</tr>
<tr>
<td>Clay &lt;0.002 mm</td>
</tr>
</tbody>
</table>

Particles larger than 2 mm (gravel and stones) are not included in determining soil texture.
Figure 5–1. Soil texture triangle.
This figure shows the relationship between the class name of a soil and its particle size distribution. The points corresponding to the percentages of silt and clay in the soil are located on the silt and clay lines respectively. Lines are then projected inward, parallel in the first case to the clay side of the triangle and in the second case to the sand side. The name of the compartment in which the two lines intersect is the class name of the soil.
Cation exchange capacity and per cent base saturation

Cation exchange capacity (CEC) and per cent base saturation are not used for fertilizer recommendations in Ontario. In calibration trials where these factors have been considered, the accuracy of fertilizer recommendations has not been improved and has sometimes been decreased.

Many soil test reports do, however, include these determinations. They are useful as a general indication of soil fertility and can point towards some potential production problems. Understanding how these numbers are derived can help keep them in perspective.

Note:
Cation exchange capacity is measured in units of electrical charge rather than weight, since the weight per unit charge of the cations varies greatly.

CEC is expressed as centimoles of positive charge per kilogram (cmol+/kg). This is preferred to milliequivalents per 100 grams, but the numbers for each are the same. To convert from parts per million to centimoles per kilogram, the ppm is divided by 10 times the atomic weight of the cation divided by its charge. For example, a soil test for magnesium (atomic weight 24, charge 2+) of 480 ppm would give a value of $\frac{480}{(24*10)/2} = 4$ centimoles per kilogram.

Cation exchange capacity
Cation exchange capacity (CEC) is a relative reflection of the total ability of the soil to hold cation nutrients — its potential fertility. For a full discussion, see the beginning of Chapter 2.

Cation exchange sites are the major source of available cations for plant uptake. CEC may be measured directly or estimated by adding the total cations measured in a soil test.

Estimating CEC
The cation exchange capacity is often estimated from the nutrients extracted by ammonium acetate. This estimation assumes that only the nutrients occupying the cation exchange sites are extracted, which is not always the case. The presence of calcium carbonate (lime) in soils with high pH may distort the values for cation exchange capacity because the ammonium acetate will dissolve some of this calcium as well.

Another quick method of estimating CEC is to use the percentage of clay and organic matter. Multiply the percentage of clay by 0.5 and the percentage of organic matter by 2. The sum of these figures estimates the cation exchange capacity of the soil.
Formula for estimating cation exchange capacity

\[
b\text{CEC value} = \left( \text{Ca value} \div 200 \right) + \left( \text{K value} \div 390 \right) + \left( \text{Mg value} \div 120 \right)
\]

(b\text{CEC} = \text{cation exchange capacity occupied by bases})

Where each of the Ca, K and Mg values (mg/kg of soil) is obtained from the ammonium acetate extraction. This equation converts them to the centimole per kilogram value.

A factor is also added for the H\(^+\) content of the soil:

- if the pH is between 6.0 and 7.0, then CEC value = bCEC value + 1.2
- if the pH is greater than 7.0, then CEC value = bCEC value.
- if the pH is less than 6.0, then CEC value = bCEC value + \{1.2 \times [70 – (pH_B \times 10)]\}.

(pH_B = \text{buffer pH})

This formula, developed in Michigan, takes into account the pH of the soil and the electrical charge of each cation. It does not take into account the presence of other cations such as aluminum or the amount of calcium or magnesium dissolved from free carbonates in the soil.

Another quick method of estimating CEC is to use the percentage of clay and organic matter. Multiply the percentage of clay by 0.5 and the percentage of organic matter by 2. the sum of these figures estimates the cation exchange capacity of the soil.

CEC measurement

A more accurate indication of the cation exchange capacity can be obtained by measuring it in the lab. The process involves saturating the soil with a particular marker cation, forcing all other cations off the exchange sites. This marker cation is then itself extracted with the ammonium acetate solution. This solution is then analyzed for the quantity of marker cation, which represents the total cation exchange capacity.

Barium is a good marker ion because it is not a common element in the soil and it has a strong enough affinity for the exchange sites to force the other cations off.

Per cent base saturation

The per cent base saturation is the ratio of basic cations to the cation exchange capacity expressed as a percentage (see per cent base saturation equations following). The term is often used loosely and sometimes refers to each individual cation or to the sum of all the basic cations.

Care must be taken when calculating and interpreting the values for per cent base saturation because the values depend on the how the CEC is obtained. For example, a potassium saturation value derived from a CEC estimate in a calcareous soil will be misleading because of the artificially high values for calcium and magnesium.

As a rule, per cent base saturation should increase with increasing pH and soil fertility.
Per cent base saturation equations

\%
Ca saturation = \(\frac{\text{ppm Ca}}{200} \div \text{CEC value} \times 100\)

\%
K saturation = \(\frac{\text{ppm K}}{390} \div \text{CEC value} \times 100\)

\%
Mg saturation = \(\frac{\text{ppm Mg}}{120} \div \text{CEC value} \times 100\)

Lab equipment

**Auto analyzer**

This machine automates the repetitive tasks of chemical analysis. The concentration of most elements in a soil or plant extract can be measured by reacting them with specific compounds to produce a coloured reaction product. The intensity of the colour is related to the concentration of the nutrient element.

In the auto analyzer, small samples of extracts, separated from each other by air bubbles, are drawn into fine plastic tubing. Other chemicals are introduced into the tube in proper proportions and mixed. The mixture might be heated or cooled or passed over a catalyst. The end product is passed through a photocell to measure the intensity of colour produced. A specific analysis track is necessary for each nutrient being tested, although they can often be set up in parallel, so that one set of samples can undergo two or more analyses.

These machines are commonly used in the analysis of nitrate, ammonium and phosphorus.

The auto analyzer is much faster than manual analysis but must be carefully calibrated with a range of stock solutions for accurate correlation to actual concentrations. Constant quality control is necessary.

**Atomic absorption**

This equipment uses a flame to break the extract down into its elements and then passes a beam of light through the flame to measure the absorption of light by those atoms. Each element absorbs light of a specific wavelength, so a light source is used with a wavelength specific for the element being tested. The concentration of the element is proportional to the amount of light absorbed. The flame temperature is important to ensure the compounds are broken down into atoms.

Because the atoms that make up the air also absorb light, this method cannot be used for elements with absorption wavelengths in the range of the elements found in air. This means that atomic absorption spectrometry cannot be used to measure nitrogen, phosphorus, sulphur or boron. This method can be used for several micronutrients (Fe, Mn, Zn, Cu, etc.) and alkaline earth elements (K, Ca, Mg).

**Emission spectrometry**

At very high temperatures and in strong electrical fields, atoms can become excited and emit light.
Each element emits light at specific frequencies, which can be measured by a photocell. The intensity of light emission indicates the amount of each element present.

An inductively coupled plasma spectrometer (ICP) or a direct coupled plasma spectrometer (DCP) can rapidly measure the concentration of elements in a solution. A tiny sample of soil or plant extract is simultaneously passed through a torch that produces high temperatures and through a strong magnetic field to excite the atoms. When the excited atoms return to their stable state, they emit light waves at specific wavelengths. The intensity of the emission indicates the amount of each element present.

This instrument produces accurate measurements of total elements present in the extracting solution over a relatively wide range of concentrations, but it must be carefully calibrated with stock solutions for each element.

In Ontario with the bicarbonate extractant, ICP analysis is not used due to mechanical difficulties with the solution itself.

**Laser analysis**
Laser-induced breakdown spectroscopy (LIBS) uses an instrument that requires no special extractants or chemicals, creates no waste, takes 3,000 readings per sample and converts total values into calibrated extractable values.

**Organic materials (plant tissue and manure)**

**Handling and preparation**

**Plant tissue**
Plant tissue samples may be sent to the lab in fresh condition or air dried if they cannot be shipped immediately. Samples should never be dried in an oven, since high temperatures can affect the analysis.

It is critical to avoid contamination from soil, dust or fertilizer. Ship the samples in paper bags, never plastic, to avoid condensation and mould.

At the lab, the samples are identified, logged and dried. The dry samples are ground to a particle size of 1 mm or less and stored in airtight containers until analysis.

**Manure**
At the lab, liquid manure samples are analyzed as they are received. Containers are mixed by inverting them several times before sampling.

In the case of solid manure, part of the sample is tested for nitrogen. The balance is dried in an oven at 100°C overnight, and then ground to pass through a 1 mm screen and stored in an airtight container until analysis. Moisture content of the manure is determined in the drying process.
Nitrogen

**Kjeldahl method**
Until the 1990s, nitrogen in manure was most often measured using a lab analysis test called total Kjeldahl nitrogen (TKN). TKN is an environmentally “unfriendly” method, using sulphuric acid to digest the organic material, with the help of a catalyst (usually mercury oxide, selenium or copper). Currently, Ontario labs use the Dumas combustion method and report that result as total nitrogen. Before the combustion method (Dumas) was economically available, TKN was the standard method. As a result, total N and TKN are often synonymous; however, NO₃-N is not measured in the TKN, which does make a difference on certain products such as leachates.

**Combustion (Dumas) method**
This method determines total nitrogen (ammoniacal, protein and nitrate sources) in organic materials. Samples are ignited in a furnace and the gases are collected. Oxygen, carbon dioxide and moisture are removed, and the nitrogen gases are determined by thermal conductivity.

In general, nitrogen determination by combustion results in slightly higher values than the conventional Kjeldahl method because the Kjeldahl method accounts only for the protein and ammoniacal sources of nitrogen.

Comments
- Uniformity of particle size and fineness is essential. A particle size of 1 mm diameter or less is recommended.
- Frequent calibration and maintenance of reagents in the instrument are crucial.

**Ammonium nitrogen**
Ammonium nitrogen in liquid manure can be measured using an ammonium-specific electrode. In either solid or liquid organic materials, the ammonium nitrogen can be measured by steam distillation or by extracting the ammonium with a KCl solution and measuring the concentration in the extract. Ammonium nitrogen can be lost during sample drying, so either the determination should be made on fresh samples or the sample should be acidified before drying to retain the ammonium.

Plant available nitrogen from manure or biosolids can be more accurately determined if both the ammonium and organic nitrogen are known, rather than just total nitrogen. Organic nitrogen is assumed to be the total nitrogen content minus the ammonium nitrogen. Nitrate content in raw manure samples is generally insignificant and not measured.
Calcium, phosphorus, potassium, magnesium, manganese, copper, iron, boron

The concentration of these elements is determined after oxidizing (ashing) the plant tissue and then dissolving the ash in acid. The samples are burned at 500°C for 2 hr. The acid digests are then analyzed for their nutrient contents. Some elements, such as phosphorus, potassium, boron and copper, tend to volatilize at elevated temperatures.

Regulated metals in biosolids

There are currently 11 metals that cannot exceed specified limits in a non-agricultural source material if it is going to be applied to land. These are arsenic, cadmium, cobalt, chromium, copper, mercury, molybdenum, nickel, lead, selenium and zinc. Levels of these metals are determined by dissolving the organic material in a strong acid and then analyzing the concentration of these elements in the digest. Mercury is determined using a slightly different procedure to prevent the release of toxic mercury vapour.

Seven of the regulated metals are also essential nutrients for either plants or animals. The concentrations determined in this procedure are useful indicators of the potential for buildup of these elements to harmful levels in the soil, but they are not always good indicators of availability for uptake by plants.

Other resources

Basic references


For more detail


