

Chapter 5

Recommended Soil Tests for Macro and Micronutrients

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Macro and micronutrients in soils are commonly measured in the Northeast with the multi-element test extracts Mehlich 3, Morgan and Modified Morgan. These tests are the focus of the discussion that follows along with the water extraction test for boron.

Macronutrient Extraction

Soil test extractants for phosphorus (P) and the cations calcium (Ca), magnesium (Mg), and potassium (K) were designed to rapidly assess the available nutrient status of soils. They serve as the basis for making recommendations for the addition of plant nutrients needed to achieve optimum yields.

Most extractants for P were developed to estimate the capacity of the soil to supply P to plants. They were designed to dissolve and/or desorb some fraction of the labile P and thus provide an index of the availability of P to crops over the growing season. Since the P chemistry of northeastern soils primarily involves factors affecting the availability of aluminum phosphates, soil tests in the Northeast use a dilute acid solution to dissolve these minerals and extract P. The Morgan and Modified Morgan extracts are well-buffered weak acid extractants. In contrast, Mehlich 3 contains HNO_3 , a strong acid, and generally removes about 5-10 times more P from soils than the Morgan extractants. The ability of the Mehlich 3 to extract more P from soils is also due to the presence of the F^- ion (as NH_4F), similar to the Bray P extractant used in the Midwest.

Soil tests for cations (Ca, Mg, K) typically estimate the quantity of water-soluble and exchangeable forms by replacing the cations on the soil's exchange sites with a counter ion such as Na^+ (Morgan) or NH_4^+ (Modified Morgan and Mehlich 3). Since all of these extractants are acidic, they will also extract some non-exchangeable K. Additionally, ammonium-based extractants like the Modified Morgan and Mehlich 3 solutions will extract more K than the Na-based Morgan extractant from some soils. All of these soil tests will extract similar amounts of Ca and Mg. However, because all three are buffered acid extracts they will also extract non-exchangeable Ca and Mg from soils with carbonate content, either naturally occurring from parent material or from recent lime application.

Micronutrient Extraction

Copper (Cu), manganese (Mn), zinc (Zn) and boron (B) are the micronutrients most commonly tested for in Northeastern laboratories. Crop responses to micronutrient fertilization are uncommon in the Northeastern United States except under certain well-defined situations.

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As a result, routine analyses of soils for micronutrients are conducted by only a few laboratories, and recommendations are generally made for only selected crops known to be sensitive to micronutrient deficiencies. Interpretation of micronutrient soil test results is often modified, either quantitatively or qualitatively, by other soil properties, such as pH, texture, organic matter, or extractable phosphorus.

Most laboratories in the Northeast use the same extractant and analytical procedures when testing for Cu, Mn and Zn as they do for the macronutrients (Table A-2). The majority of laboratories that test soils for B use some modification of the hot-water extraction procedure originally developed by Berger and Truog (1939) [see Chapter 6 “Recommended Soil Tests For Boron”] although the Mehlich 3 extractant is receiving more interest (Shuman, et al., 1992). The modified Morgan extract is also used to routinely test for B in some states (Table A-2).

Measurement of Macro and Micronutrients in Soil Extracts

There are several approaches that can be used to measure the macro and micronutrients in the Mehlich 3, Morgan, and modified Morgan soil extracts. Phosphorus may be measured by inductively coupled plasma (ICP) or by the ammonium molybdate or ammonium vanadate colorimetric methods; K and Ca may be measured by atomic absorption, atomic emission, or by ICP; Mg, Cu, Mn and Zn may be measured by atomic absorption or by ICP; and B may be measured by colorimetric methods or by ICP. The results from the various techniques used to measure the macro and micronutrients in the soil extracts should, in general, be comparable although P measured by ICP may be higher than when measured colorimetrically for many soils. Because of this difference, soil test calibrations should be based on the specific analytical method routinely used. Because of the greater efficiency of ICPs, the majority of Northeast laboratories utilize this approach when measuring the macro and micronutrients in soil extracts although several utilize a colorimetric method for phosphorus.

This bulletin provides recommended procedures for extracting macro and micronutrients from soils by the Mehlich 3, Morgan and Modified Morgan extracts with general reference to analysis of extracted elements by ICP. Details on ICP calibration and measurement will be dictated by the specific instrument used and, thus, are not described here. A procedure for measuring P colorimetrically by the ammonium molybdate method is provided.

Extraction Methods

Mehlich 3 Extraction (Mehlich, 1984)

Equipment:

1. Standard soil scoops (2.5 cm³, 2.5g, and 1.0 cm³)
2. Reciprocating or rotary shaker, capable of 180 oscillations per minute (opm).
3. 100 mL plastic (PVC), wide-mouthed, extraction bottles (using glass containers can contaminate the extract with boron).
4. PVC Filter funnels.
5. ICP

Reagents:

1. **Mehlich 3 Stock Solution (3.75 M NH₄F + 0.25 M EDTA):** Mix 277.8 g ammonium fluoride (NH₄F) with ~1200 mL distilled water in a 2 L volumetric flask. Add 146.1 g of EDTA (ethylenediaminetetraacetic acid: [(HOOCCH₂)₂NCH₂N(CH₂COOH)₂]). Dilute to volume with distilled water and mix well. This is enough stock solution for ~10,000 samples.
2. **Mehlich 3 Extracting Solution: (0.2 N CH₃COOH + 0.25 N NH₄NO₃ + 0.015 N NH₄F + 0.013 N HNO₃ + 0.001 M EDTA).** Dissolve 1000 g ammonium nitrate (NH₄NO₃) in ~ 40 L of distilled water in a 50 L calibrated plastic carboy. Add 200 mL of the Mehlich 3 stock solution and mix well. Add 575 mL of glacial acetic acid (CH₃COOH) and 41 mL of concentrated nitric acid (HNO₃). Dilute to 50 L final volume with distilled water and mix thoroughly. The solution pH should be 2.5 +/- 0.1.
3. **Standard calibration solutions:** Prepare a series of mixed calibration standards for the elements of interest that encompass the expected range in the soil extracts and are within the linear range of each element on the ICP. Standards should be matrix-matched to the Mehlich 3 extract.

Procedure:

1. Scoop 2.5 cm³ or 2.5 g of air-dried, sieved soil into a 100 mL extraction bottle. See Chapter 2 for details on soil preparation and scooping technique.
2. If a colorless filtrate is needed, add 1 cm³ of activated carbon (Norit™ SG Extra or equivalent) to each flask.

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3. Add 25 mL of the Mehlich 3 extracting solution to each bottle.
4. Shake at 200 oscillations per minute for 5 minutes on a reciprocating shaker.
5. Filter through a medium-porosity filter paper (Whatman No. 2 or equivalent).
6. Analyze filtrate for P, K, Ca, Mg, Cu, Mn, Zn and/or B on the ICP. Alternatively, P may be measured colorimetrically (see method later in chapter).

Note:

The original procedure of Mehlich (Mehlich, 1984) calls for maintaining a 10:1 solution:soil ratio on a volumetric basis (i.e. 25 mL Mehlich 3 solution to 2.5 cm³ soil). However, many laboratories that switched to the Mehlich 3 from other test procedures developed their calibration database on soils extracted with a 10:1 solution: soil ratio on a weight instead of a volume basis (i.e. 25 mL Mehlich 3 solution to 2.5 g of soil or 20 mL of Mehlich 3 solution to 2.0 g of soil). The current recommended procedure for Mehlich 3 for laboratories in the North Central region is to maintain a 10:1 solution:soil ratio on a weight basis (Knudsen and Beegle, 1996). Performing the procedure by either the weight or volume method is appropriate so long as the calibration database upon which the recommendations are based was developed using the method selected.

Morgan Extraction
(Morgan, 1941)

Equipment:

1. 10 cm³ and 1 cm³ standard soil scoops.
2. Reciprocating or rotary shaker, capable of 180 oscillations per minute (opm).
3. 125 mL extraction flasks

Reagents:

1. **Morgan Extractant (0.72 N NaOAc + 0.52 N CH₃COOH)**: Add 5000 g of sodium acetate trihydrate (CH₃COONa·3H₂O) to a 50 L carboy containing approximately 20 L of distilled water. Add 1450 mL glacial acetic acid and mix until the sodium acetate is dissolved. Dilute to 50 L with distilled water and mix well. The pH of the solution should be 4.8 +/- 0.05. If necessary, adjust to 4.8 with sodium acetate or acetic acid.
2. **Activated carbon** (Norit™ SG Extra or equivalent)
3. **Standard calibration solutions**: Prepare a series of mixed calibration standards for the elements of interest that encompass the expected range in the soil extracts and are within the linear range of each element on the ICP. Standards should be matrix-matched to the Morgan extract.

Procedure:

1. Scoop 10 cm³ of air-dried, sieved soil into 125-mL Erlenmeyer flasks. If weight-basis results are needed, weigh the scooped soil before transferring to the container. See Chapter 2 for details on sample preparation and scooping techniques.
2. If a colorless filtrate is needed, add 1 cm³ of activated carbon to each flask.
3. Add 50 mL of the Morgan extractant to each flask.
4. Shake at 180 oscillations per minute for 15 minutes on a reciprocating shaker.
5. Filter through a medium porosity filter paper (Whatman No. 2 or equivalent).
6. Analyze filtrate for P, K, Ca, Mg, Cu, Mn, and/or Zn on the ICP. Alternatively, P may be measured colorimetrically (see method later in chapter).

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Modified Morgan Extraction (McIntosh, 1969)

Equipment:

1. 4 cm³ and 1 cm³ standard soil scoop.
2. Reciprocating or rotary shaker, capable of 180 oscillations per minute (opm)
3. 50 mL extraction flasks.
4. Filter funnels.

Reagents:

1. **Modified Morgan Extractant (0.62 N NH₄OH + 1.25 N CH₃COOH)**: Add 2874 mL glacial acetic acid to a 40 L carboy containing approximately 20 L of distilled water. Add 1825 mL concentrated NH₄OH. Dilute to 40 L with distilled water and mix well. The pH of the solution should be 4.8 +/- 0.05. If necessary, adjust to 4.8 with concentrated NH₄OH or acetic acid.
2. **Activated carbon** (NoritTM SG Extra or equivalent)
3. **Standard calibration solutions**: Prepare a series of mixed calibration standards for the elements of interest that encompass the anticipated range in the soil extracts and are within the linear range of each element on the ICP. Standards should be matrix-matched to the Modified Morgan extract.

Procedure:

1. Scoop 4 cm³ of air-dried, sieved soil into 50 mL extraction flasks. If weight-basis results are needed, weigh the scooped soil before transferring to the container. See Chapter 2 for details on sample preparation and scooping techniques.
2. If a colorless filtrate is needed, add 1 cm³ of activated carbon to each flask.
3. Add 20 mL of the Modified Morgan extractant to each flask.
4. Shake at 180 oscillations per minute for 15 minutes on a reciprocating shaker.
5. Filter through a medium porosity filter paper (Whatman No. 2 or equivalent)
6. Analyze filtrate for P, K, Ca, Mg, Cu, Mn, and/or Zn on the ICP. Alternatively, P may be measured colorimetrically (see next method).

Colorimetric Analysis of Phosphorus

Ammonium Molybdate-Ascorbic Acid Method (Knudsen and Beegle, 1988)

Equipment:

1. Colorimeter or autoanalyzer for measuring absorbance at 882 nm.

Reagents:

1. **Concentrated ammonium paramolybdate solution:** Add 60 g ammonium paramolybdate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$) to approximately 200 mL of distilled water in a 1 L volumetric flask. Heat to 60°C if necessary to dissolve. Dissolve 1.455 g antimony potassium tartrate ($\text{KSbO}\cdot\text{C}_4\text{H}_4\text{O}_6$) in the ammonium paramolybdate solution. Add 700 mL of concentrated sulfuric acid and allow the solution to cool to room temperature. Dilute to volume with distilled water. Store in a dark glass bottle in the refrigerator.
2. **Ascorbic acid solution:** Dissolve 132 g of ascorbic acid in distilled water in a 1 L volumetric flask. Dilute to volume with distilled water.
3. **Working colorimetric solution (prepare daily):** Add 25 mL of concentrated ammonium paramolybdate solution to approximately 800 mL distilled water in a 1 L volumetric flask. Add 10 mL of the ascorbic acid solution. Dilute to volume with distilled water.
4. **High phosphorus standard (50 mg P/L):** Dissolve 0.2197 g of reagent grade monobasic potassium phosphate (KH_2PO_4) in distilled water in a 100 mL volumetric flask. Make to volume with distilled water.
5. **Working phosphorus standards:** Add the specified volume of the 50 mg P/L stock solution to volumetric flasks in the sizes specified in Table 5-2. Bring each flask to volume with the soil extracting solution (Mehlich 1, Mehlich 3, Morgan, or Modified Morgan).

Procedure:

1. Transfer a 2 mL aliquot of the soil extract to test tubes.
2. Similarly, transfer 2 mL aliquots of each of the working standards to test tubes.
3. Add 8 mL of the colorimetric working solution to each tube and mix thoroughly.

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4. Allow 20 minutes for color development.
5. Read percent transmittance of all standards and samples on a colorimeter with wavelength set at 882 nm.
6. For automated flow-injection or segmented-flow systems, follow the manufacturers suggested setup, configuration, and operation. Reagent specification and makeup are typically the same or very similar to the manual procedure specified here.

Table 5-2. Working Standard P Solutions for the Mehlich 3, Morgan, or Modified Morgan Extractants.

Volume of 50 mg P/L Stock Solution	Final Solution Volume of Working Standard	P Concentration of Working Standard
----- mls -----	----- mls -----	----- mg P / L -----
1	250	0.2
1	100	0.5
2	100	1.0
4	100	2.0
6	100	3.0
8	100	4.0
10	100	5.0
12	100	6.0
16	100	8.0

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