

Cornell Soil Health Laboratory 2022	Code: CSH 13
	Date: 1/6/2022

Modified Morgan Extraction and pH Measurement Standard Operating Procedure

Cornell Soil Health Laboratory / Cornell Nutrient Analysis Laboratory

Location: G01 Bradfield



Purpose and Justification:

The Cornell Soil Health Laboratory (CSHL) receives soil samples year-round for a CSHL package and for individual soil analyses. Standard nutrient testing is offered as part of the CSHL packages along with pH measurement. Offering Nutrient Analysis and pH measurement is essential for providing CSHL clients with complete soil health analysis data.

Nutrient Analysis is done using a Modified Morgan (MM) extraction and reported in available nutrients. Samples are sieved past 2mm and placed in labeled paper cups, dried at 50C before extraction. For the MM extraction, soil is extracted at a 1:4 soil/solution ratio with Modified Morgan solution [.62 N NH_4OH + 1.25 N CH_3COOH] buffered at pH 4.80 with the filtrate subsequently analyzed by ICP for Ca, Mg K, Fe Al, Mn, and Zn.

Soil pH is a measure of hydronium ion (H_3O^+ , or more commonly the H^+) activity in the soil solution. Soil pH influences many facets of crop production and soil chemistry, including availabilities of nutrients and toxic substances, activities and nature of microbial populations, and activities of certain pesticides. A suspension of two parts water to one part soil (2:1 ratio), is prepared and allowed to stand at room temperature for one (1) hour .The pH can then be determined with a manual pH meter or robotic system.

Modified Morgan Extraction Materials and Equipment

1. Orbital shaker, VWR brand Model OS-500 w/18"x18" platform. WWR 14005-830
2. Pipetting Machine, SEPCO Model 40A w/30cc glass syringe. VWR 53537-205
3. Fisherbrand 30 ml Bottle-Top Dispenser. Fisher 03-692-180
4. Soil Scoop, 4.25 cm^3 (5g) volumetric, Illinois Soil Testing Association

5. Spatula, stainless steel, 4" Fisher brand. Fisher 14365B
6. Erlenmyer Flasks (50), 50 ml, Nalgene Polypropylene. VWR 29136-842
7. Funnels (50), VWR brand Polypropylene, I.D. 48 mm. VWR 30252-903
8. Test Tubes (50), 23 ml, 16x150 mm, Polypropylene. Lake Charles Mfg.
9. Four (4) pouring racks holding ten (10) 50 ml flasks each. Filtration bench to accommodate funnels and test tubes.
10. Filter paper, SAP-1F, 110 cm, conically folded. Laboratory Filtration Group llc
11. Short stem filling funnel for soil scooping, I.D. 52mm.
12. Analytical Balance, AND GR-200 or equivalent.
13. Magnetic Stirrer VWR brand Model 361. VWR 12365-386
14. Magnetic Stir bars 1.5", 3"
15. Single channel, adjustable pipetter, 0.10 to 1.00 ml. Corning Lambda or equivalent. Fisher 07-200-814.
16. Ten head sprayer/washer to hold pouring racks and funnels. Dairy One custom design Cleaning bath

I. Reagents

a. List

1. Glacial Acetic Acid (CH_3COOH). J.T. Baker 'Baker Analyzed' Reagent Grade. F.W. 60.05 VWR JT9507-33
2. Ammonium Hydroxide (NH_4OH). 28-30%. J.T. Baker 'Baker Analyzed Reagent Grade. F.W. 35.05 VWR JT9721-33
3. Carbon, Activated Powder (Darco G-60). J.T. Baker. F.W. 12.01 VWR JTE343-9
4. Deionized H_2O (DI H_2O)
5. Citranox acid cleaner and detergent. VWR 21835-350

b. Preparation

1. Modified Morgan Extractant (for 20L)
 - a. Add 1437 ml of glacial acetic acid to a 20L carboy containing approximately 10L of DI H_2O water. Add stirbar and mix well.
 - b. Slowly Add 912.5 ml of ammonium hydroxide, mix for approximately ten (10) minutes and then dilute to 20 L with DI H_2O . Mix well.
 - c. pH of the solution should be $4.80 \pm .05$. Adjust pH if needed with glacial acetic acid or ammonium hydroxide.

Safety Note 1: Ammonium Hydroxide: Danger! Poison! Causes irritation or burns to skin, eyes, and respiratory tract. May be fatal if swallowed or inhaled. Irritation or burn effects may be delayed. Harmful if absorbed through skin. Wear all safety gear including lab coat, safety glasses, and proper gloves.

Safety Note 2: Glacial Acetic Acid: Danger! Corrosive! Poison! Liquid and mist cause severe burns to all body tissue. Inhalation may cause lung and tooth damage. Flammable liquid and vapor. Wear all safety gear including lab coat, safety glasses, face shield, and proper gloves. Use only in proper ventilation hood

Prepping Samples for Nutrient Analyses and pH Measurement

1. Sieve air-dried soil sample to 2mm and place $\frac{3}{4}$ cup in 1 cup paper cans with sample ID recorded on the paper can.
2. Samples are oven dried at 50C in racks of up to fifty samples and arranged sequentially left to right and front to back by ID number on a wooden sample tray.
3. A complete rack contains 48 labelled soil samples and two QC samples placed as samples 49 and 50.
4. When samples have reached a constant weight, usually ~24 hours later they can be removed from the oven and brought to the Ground floor of Bradfield for analysis. Samples should be stored with paper on top of the samples within the wooden rack.
Label Quarantined sample racks with a large “Q” and store these racks in specific Quarantine storage area in lab.

Quarantined Soil Additional Steps

Quarantine soil handling procedures:

- ***All excess soil must be autoclaved.***
- ***All water that comes into contact with soil must be bleached with a 10% bleach solution for $\frac{1}{2}$ hour before disposing of bleach water down drain.***
-

Any area or material that has come into contact with quarantined soil must be disinfected using sterilization solutions approved for use with Quarantined soil:

1. ***Bleach- 10% bleach solution within a labeled spray bottle must be left on contaminated equipment for $\frac{1}{2}$ hour before rinsing. Note, this is the only approved method for handling water contaminated with quarantined materials.***
2. ***Ethanol solution (70%) (stored in a labeled spray bottle) must be left on contaminated equipment for 30 minutes before rinsing***

Modified Morgan Extraction for ICP Analysis

1. Obtain rack of labelled paper cans with soil samples oven dried at 50C and sieved past 2mm.
2. Starting with the first sample, place one calibrated scoop (5g) sample per sample into a labeled 125ml glass erlenmeyer flask. Repeat for each set until all samples are scooped.
3. Obtain sub-sample for pH measurement simultaneously (See methods at end of this document).
4. Dispense 20ml of Modified Morgan solution using pipetting machine or bottle-top dispenser into each flask. (See Preparation of Modified Morgan Extractant later in this document)

To dispense from pipetting machine:

- a. Place tube tip on the carboy cap tube fitting.

- b. Turn on with toggle or foot switch.
- c. Allow system to purge back into carboy for at least twelve cycles.
- d. During the syringe fill part of the cycle quickly remove the tube from the carboy cap fitting and place the tip in first flask to dispense solution.
- e. Continue moving to subsequent flasks during refill until all flasks contain extraction solution.
- f. Turn off pipetter during refill. Place tube on carboy fitting.

Note: The speed of the dispense-refill cycle should not be adjusted as this will change the delivery volume. It should always be set at the indicated mark on the speed adjustment scale. Periodically (once every week) check delivery volume with graduated cylinder and adjust if necessary.

To dispense from bottle:

- a. Set dispenser for 20 ml and purge into bottle or beaker.
 - b. Dispense 20 ml into graduated cylinder to insure accurate delivery.
 - c. Adjust to correct volume with dial on top of dispenser if needed.
 - d. Place flask rack on lab bench and dispense 20 ml into each flask.
 - e. Place each rack on shaker in order when full and lock in place.
5. Close lids tightly.
 6. Place racks of flasks onto shaker (40 oscillations/ minute) for 15 minutes. Ensure oscillating speed is set at 40 oscillations per minute before starting apparatus.
 7. Prepare funnel racks with folder filter paper while shaking. (Depending on experience this may need to be done prior to extraction/shaking)
 8. When the timer indicates stop the shaker and unlock the pouring racks. Pick up the front rack, swirl it a few times, and immediately pour it into the bottom row of funnels on the filtration bench.
 9. Repeat for the remaining racks with the last (back) rack poured into the top row of funnels.

Note: Proper pouring of all flasks can be accomplished by centering the spout of one of the flasks on the corresponding funnel and then inverting the rack.

9. Remove the filter papers after the solution is completely drained and discard in waste barrel.
10. Remove the funnel racks from the filtration bench and place them on a cart or on the counter near the washing station.
11. Place the test tubes with the filtrate in a test tube rack and cap or cover with plastic wrap. Label the tube rack and place at the end of the laboratory bench for ICP analysis.
12. Analyze by ICP for Ca, Mg, K, P, Al, Zn, Mn, and Fe on a Thermo Elemental IRIS Intrepid ICP-AES spectrophotometer

IV. Cleanup

1. Place funnel racks on the sink and rinse with warm tap water, then submerge in 3% Citranox bath. Remove from bath after a minimum of fifteen (15) minutes and rinse with warm tap water until no foaming is apparent. Rinse 3 times with DI H₂O. Place on the drying rack.
2. Invert flask racks, place on the sprayer and rinse until all of the soil is washed into the soil trap in the sink. Turn them right side up and submerge in 3% Citranox bath. Soak for at least fifteen (15) minutes, drain solution into bath, rinse on the sprayer until no foaming is apparent, rinse 3 times with DI H₂O, invert and place on the drying rack.
3. After analysis on the ICP, empty remaining filtrate into sink and place tubes in a polypropylene washing basket. Pack the basket tightly enough so that tubes remain in the basket when it is inverted. Fill the tubes with warm tap water, invert to drain them, and submerge them in the Citranox bath. Soak for at least fifteen (15) minutes, drain solution back into the bath and rinse the tubes by filling them with tap water and draining them repeatedly until no foam from the Citranox is apparent. Rinse 3 times with DI H₂O. Invert the basket on the counter to dry.

Quarantined Soil Additional Steps

Quarantine soil handling procedures:

- *All excess soil must be autoclaved.*
- *All water that comes into contact with soil must be bleached with a 10% bleach solution for ½ hour before disposing of bleach water down drain.*

Any area or material that has come into contact with quarantined soil must be disinfected using sterilization solutions approved for use with Quarantined soil:

3. *Bleach- 10% bleach solution within a labeled spray bottle must be left on contaminated equipment for ½ hour before rinsing. Note, this is the only approved method for handling water contaminated with quarantined materials.*
4. *Ethanol solution (70%) (stored in a labeled spray bottle) must be left on contaminated equipment for 30 minutes before rinsing*

References

Adapted for gravimetric determination from Wolf, A. and D. Beegle. 1995. Recommended Soil Tests for Macronutrients: Phosphorous, Potassium, Calcium and Magnesium. p. 30-38. In Sims, J.T. and A.M. Wolf (ed.). Recommended Soil Testing Procedures for the Northeastern United States. Northeastern Regional Pub. No. 493 (2nd edition). Agricultural Experiment Station University of Delaware, Newark, Del 19717-1303

Determination of Soil pH

Materials and Equipment:

1. pH Meter (LIGIN Robotic pH system or other suitable meter).
2. Electrodes: Ross combination pH electrodes from Thermo Scientific (Orion 815600) for manual determination
3. 3 oz. plastic cups
4. Rack to hold fifty (50) cups.
5. Pipetting machine: Oxford automatic variable speed dispenser, Catalog No. 470, Oxford Laboratories Inc., Foster City, CA 94404 capable of delivering 20 ml (optional).
6. Soil Scoop, 20 cm³ (cc).
7. Stirring rod.

Reagents

1. Buffer, pH 7.00: Available from laboratory supply houses. Calibrate a pH meter with a certified pH 7.00 buffer. Read the pH of the prepared solution, and adjust the pH to 7.00.
Buffer, pH 4.00: Available from laboratory supply houses. Calibrate a pH meter with a certified pH 4.00 buffer. Read the pH of the prepared solution; it should read pH 4.00 without adjustment.
2. Deionized water.

Procedure:

1. Place cups in the rack. Label cups with sample IDs.
2. Using the 20 cm³ scoop, scoop a portion of soil from each sample. Also, scoop 5 portions of soil for the master soil. (It is necessary to determine the pH of the masters at the beginning of the run.)
3. The pipetting machine is set to deliver 20 ml, add 40 ml (2X) of distilled water to each cup with sample.
4. Stir each sample several times, until a homogeneous slurry is achieved. Rinse the stirring rod with distilled water.
5. Allow to stand for one (1) hour.

Procedure for LIGNIN system:

1. Turn on the computer. Open ports on electrodes. Double click pH robot icon on the desktop. This will open to the **ANALYSIS SCREEN**. Click to “find home” replace old buffers with fresh.
2. On the **Tray Configuration** screen enter columns and row number.
3. On the **ANALYSIS SCREEN** enter the sample file and run details.
4. Click “**Calibrate**” and robot will automatically calibrate with buffers 4 & 7 rinsing with DDI water in between each buffer.
5. When calibration is checked and complete; select “**Read pH**” and the instrument will begin to analyze samples. When finished click **Save** to an external USB file.

Clean-up

1. Pour off the supernatant liquid, then dispose of the cups and soil in the waste soil container.
2. **Notes :**
 - Electrode maintenance:
 - a. Maintenance: Check level of KCL in electrode, fill with fresh “Saturated KCL”.
 - b. LIGIN System: If calibration data or MV reading is out of range replace electrode and/ or amplifier.

Quarantined Soil Additional Steps

Quarantine soil handling procedures:

- *All excess soil must be autoclaved.*
- *All water that comes into contact with soil must be bleached with a 10% bleach solution for ½ hour before disposing of bleach water down drain.*

Any area or material that has come into contact with quarantined soil must be disinfected using sterilization solutions approved for use with Quarantined soil:

5. *Bleach- 10% bleach solution within a labeled spray bottle must be left on contaminated equipment for ½ hour before rinsing. Note, this is the only approved method for handling water contaminated with quarantined materials.*
6. *Ethanol solution (70%) (stored in a labeled spray bottle) must be left on contaminated equipment for 30 minutes before rinsing*

References

1. EPA Method 150.1 (4-79-020). Downloaded from :
http://www.umass.edu/tei/mwwp/acrobat/epa150_1pH.pdf
2. Hendershot, W.H., H. Lalonde, and M. Duquette. 1993. Soil reaction and exchangeable acidity. In: M.R. Carter Soil Sampling and Methods of Analysis. Lewis Publishers, pp 141-145.