Soil Core NO3/NH4 Extraction
07_06_02.002
Dates Active:
7/10/2018 - present
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Soil Core Processing

1 INTRODUCTION

Soil core processing is a method used to prepare a soil core subsample for instrumental analysis. Prior to chemical analysis of ammonia and nitrate, soil cores must be thawed, divided into smaller subsamples, homogenized and extracted with a with a 1 or 2N potassium chloride solution. Thawed and air-dried samples should be ground and sieved within 24 hours of thawing. After this process is complete, the core subsample will be represented as a liquid, 100 mL potassium chloride solution. Extraction of mineral and exchangeable nitrogen (ammonia and nitrate) requires a KCl salt solution rather than water because the strong activity of the cation in the salt solution will exchange adsorbed $NH4^+$ chloride anion can help displace weakly exchangeable NO_3 on positive adsorption sites.

2 SCOPE AND APPLICATION

2.1 OVERVIEW

This method describes the process of sediment separation, homogenization and extraction for determining moisture content, bulk density, pH, as well as the preparation for ammonia and nitrate analysis.

2.2 METHOD DETECTION LIMIT

A method detection limit is not applicable for this procedure. A method detection limit for nitrate and ammonia on the Lachat can be found on the Lachat SOP (13_02_02).

2.3 ACCEPTABLE RANGES

Ranges are not applicable for this procedure. Acceptable ranges for nitrate and ammonia Lachat analysis can be found in the Lachat SOP.

2.4 TRAINING TIME

1-3 days of training time may be necessary for this procedure.

2.5 SAMPLE PRESERVATION

The final subsample should be preserved with 5 drops of sulfuric acid per 100 mL. If the sample will not be ran on the Lachat the same day the sample must be frozen.

3 REQUIRED TRAINING

Users should receive 3-4 full days of training on the procedure.

4 EQUIPMENT AND MATERIALS

4.1 APPARATUS AND MATERIALS

4.1.1. Aluminum Foil
4.1.2. Knife
4.1.3. Beakers, 250 mL
4.1.4. Top Loader Scale
4.1.5. Drying Oven
4.1.6. 20 Liter Carboy
4.1.7. Graduated Cylinder
4.1.8. Thomas-Wiley Mill
4.1.9. Whirl-Pak Bags
4.1.10. Erlenmeyer Flasks, 250 mL
4.1.11. Wrist-Action Shaker
4.1.12. Buchner Filters
4.1.13. Whatman #42 filter paper, 7cm
4.1.14. Erlenmeyer Vacuum Flasks
4.1.15. Polyethylene Bottles, 150 mL
4.1.16. Pipette
4.1.17. pH Electrode
4.1.18. EC Electrode

4.2 REAGENTS

4.2.1. Reagent Grade 1 N KCl
4.2.2. DDI Water
4.2.3. pH Buffer Solutions of pH= 4.00 and pH=7.00

4.3 CHEMICALS

Chemical	CAS Number	Hazards	Location
Potassium Chloride	7447-40-7		Room 203 Countertop
Sulfuric Acid	7664-93-9		Dropper located in Soil lab, stock solution located in Room 205

5 SAFETY PRECAUTIONS

5.1 SAFETY PRECAUTIONS

5.1.1 Facemasks should be worn to protect against dust particles when using the mill.
5.1.2 Gloves should be worn when handling chemicals.

5.2 WASTE DISPOSAL

5.2.1 KCl can be dumped down the sink.

6 SOLUTIONS AND REAGENTS

6.1 1 M POTASSIUM CHLORIDE

Potassium Chloride	74.55	1491.2 g
Distilled Deionized Water	FW	20 Liters

Protocol: Carefully weigh out 1491.2 g of reagent grade KCl and transfer to a 20-liter carboy. Measure 4 liters of DDI water using a graduated cylinder and add it to the carboy containing the KCl. Shake vigorously to dissolve the KCl. Add an additional 16 liters (again, measured with a graduated cylinder) and swirl to mix thoroughly.
Storage: 1 M KCl may be stored at room temperature.
Disposal: 1 M KCl may be dumped down the sink without any further dilution.

7 STANDARD SOLUTIONS

7.1.1 Standard 1: Stock Nitrate Standard 100.0 mg N/L as NO_3 in 1 M KCl
In a 1 L volumetric flask, dissolve 1.444 g potassium nitrate (KNO_3) in about 600 mL 1 M potassium chloride (KCl). Dilute to the mark with 1 M potassium chloride and invert to mix.

7.1.2 Standard 2: Stock Nitrite Standard 100.0 mg N/L as NO_2 in 1.0 M KCl
In a 1 L volumetric flask, dissolve 0.986 g sodium nitrite ($NaNO_2$) in approximately 800 mL 1 M potassium chloride. Dilute to the mark and invert to mix. Prepare this standard and all nitrite standards fresh daily.

7.1.3 Working Stock Standard 3.0 mg N/L as NO_3

In a 1 L volumetric flask add 100.0 mL Stock Standard 1. Dilute to the mark with 1 M KCl and invert to mix. Prepare fresh weekly.

7.1.3 **Working Stock Standard 3.0 mg N/L as NO_2**

In a 1L volumetric flask, add 100.0 mL Stock Standard 2. Dilute to the mark with 1.0 M KCl and invert to mix.

8 PROTOCOL

8.1 PREPARATION OF SOIL CORE EXTRUSION

8.1.1 Line the workbench with aluminum foil and tape in place.

8.1.2 From the freezer, remove 10-12 soil cores (2.5 feet each) and arrange them in order of depth, marking the aluminum foil with the depths.

8.1.3 Allow to thaw for 2-24 hours.

8.1.4 Cores should not be left out unprocessed for longer than 24 hours.

8.2 CORE EXTRUSION

8.2.1 After the cores have thawed (usually the next day), carefully slide the sediment out of the barrel onto the foil. If the soil does not easily slide out of the barrel, a hammer may be used to carefully break the plastic barrel. Be careful not to break the soil core any more than necessary.

8.2.2 Lay successive cores end to end, matching the depths.

8.2.3 Cut the cores into subsamples where a lithological change is identified. Each 2.5' core should be broken into at least 2 subsamples. Use a new knife to cut each interval in order to minimize cross contamination.

8.2.4 Record the new depths of subsample ranges on the foil with a sharpie. Depths can be determined by using a ruler and measuring the length of the subsample and adding it to the previous depth.

8.2.5 On the soil lab worksheet, record observations of color, texture, and evidence of organic matter as well as chemical or physical iron. In addition to these observations, use the Soil Texture Pyramid to classify sediment texture.

8.2.6 Use a ruler and a knife to cut a 1" segment (lengthwise) from each sample. This 1" segment will be processed for gravimetric water content and bulk density. Record the weight of the 1" processed sample by first recording the weight of a 250 mL beaker and then adding it to the beaker and recording the total weight of the beaker and the processed sample.

8.2.7 Ensure the beaker is labeled to properly identify which sample it contains and place the beaker into the oven for 24 hours at 105ºC. After 24 hours, record the weight of the dry sample and the beaker. This data will be used to measure bulk density and gravimetric water content. See section 9.1 for calculations.

8.3 NO_3/NH_4 & pH Processing

8.3.1 Cut each subsample in half, lengthwise. Save half in a plastic Ziploc baggie and freeze it, maintaining the field moisture content. With the other half, homogenize with the soil separator tool and allow it to sit overnight with a fan blowing air over the samples to air dry.

8.3.2 Once the samples have been given ample time to air dry, collect each sample in a large aluminum weighing dish. Grind each processed interval in the Thomas-Wiley Mill with a 2 mm screen. To minimize cross contamination, clean out the mill between samples by removing all soil particles using a flat-head screwdriver, brushes and the shop vacuum located in the soil lab.

8.3.3 Using a scale, weigh out 10.0 g (±0.1 g) of the ground soil and place into a 125mL Erlenmeyer flask. Weigh out an additional 5 g (±0.1 g) of ground soil for pH testing in a dram vial. Weigh out 15g (±0.1 g) for particle size analysis. For this method, see the particle size analysis SOP (07_05_02). Put the remaining ground soil in a Ziploc bag, labeled with the date and soil sample I.D. and store in the freezer.

8.3.4 For QA/QC purposes, for every 20 samples processed there should be a duplicate, a known soil standard, an LFB and an LRB.

8.3.5 To each 10 g of processed soil in an Erlenmeyer flask, add 100mL of 1 M KCl. The Dispensette® dispenses 50 mL 1 M KCl per pump. The first time you use the Dispensette® each day, dispense the potassium chloride into an empty beaker in order to remove all of the air and ensure that a full 50 mL is being dispensed each time.

8.3.5.1 The Dispensette® should be cleaned at the end of each day it is used as the KCl dries and degrades the pump over time. To clean the pump, remove it from the container of KCl and pump at least 200 mL of DDI water through it. Make sure that all of the water is out of the pump before placing it back onto the container of KCl.

8.3.6 After 100 mL of KCl has been added to the Erlenmeyer flasks containing samples, put the stoppers onto the flasks and make sure they are tight. Shake by hand for one minute to ensure sediments in the bottom are dislodged.

8.3.7 Fasten the samples onto the wrist action shaker and start the machine at a low speed before increasing the speed to 2 or 3. Leave the samples to shake for one hour.

8.3.8 Set up the Buchner filtering assembly using 7cm Whatman #42 filter paper. Rinse the filters before use by wetting them with 1 N KCl into a separate flask and discard the filtrate.

8.3.9 After an hour on the wrist action shaker has passed, remove the samples and filter into clean Erlenmeyer vacuum flasks. If the filtrate appears cloudy, murky, or discolored, filter again until sample is clear.

8.3.10 Transfer the filtrate into a clean, labeled, 125 mL polyethylene bottle, acidify with 5 drops of sulfuric acid, and store in the freezer. Samples may be stored for 2-3 months. Labels on the bottle should include project name, core number, foot interval and date.

8.4 pH

8.4.1 Using buffers pH= 4.00 and pH 7.00, calibrate the pH meter before use. Check the calibration buffer every 20 samples.

8.4.2 Carefully pipette 5mL of deionized distilled water into the dram bottle containing the 5 g of sample. Shake vigorously to mix and then allow it to sit for 10 minutes before inserting the pH electrode.

8.4.3 Insert the pH electrode and gently swirl the sample until the reading is stable. It is important to consistently record the pH value within a few seconds of stabilization.

8.4.4 Thoroughly rinse the pH electrode with deionized distilled water before measuring the pH of the next sample. When finished, clean the electrode thoroughly, using care to remove any sediment from the reference junction, put the cap back on and soak in electrode soaking solution.

9 Data Reduction and Statistics

9.1 Calculations

9.1.1 Bulk density measurements use a sample volume from the 1”
- For large auger cores = 65.22 cm^3
- For smaller GeoProbe cores = 34.41 cm^3

9.1.2 Bulk Density (g/cm^3) = Oven Dried soil weight (g) ÷ sample volume (cm^3)

9.1.3 Sample water content (g) = Field moisture content soil (g) – oven dried soil (g)

9.1.4 Gravimetric Water Content = Oven dried soil weight (g) ÷ Sample water content (g)

10 Additional Information

10.1 References

- Black, C. A., Evans, D. D., Ensminger, L. E., White, J. L., & Clark, F. E. (1965). *Methods of Soil Analysis*. (C. A. Black & R. Dinauer, Eds.). Madison, Wisconsin: American Society of Agronomy, Inc.
- Bobier, M. W., Frank, K. D., & Spalding, R. F. (1993). Nitrate-N movement in a fine textured vadose zone. Journal of Soil and Water Conservation, 48(4), 350–354.
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- Spalding, Roy F., and Lisa A. Kitchen. "Nitrate in the intermediate vadose zone beneath irrigated cropland." *Groundwater Monitoring & Remediation* 8.2 (1988): 89-95.

11 PREVIOUS ISSUES AND CHANGES

Document File Name	Issue	Issue Effective Dates	Author

11.1 ISSUE CHANGES