Standard Operating Procedure

Ammonia operation

Soil Environmental Chemistry Program, The Ohio State University

Version 2

1.0 Scope

Lachat may be used to determine the following ions in solution: Ammonium (NH_4)

1.1 The application ranges:

- 0.10- 20 mg N/L as NH_4

1.2 Throughput:

- 30 injections/hour

2.0 Definitions

2.1 Check standard: Standard that is independently made, that is used for Quality control.

2.2 Matrix: also known as the carrier, it matches the substance the samples are in.

3.0 Equipment and Supplies

3.1 Balance

3.2 500 ml beaker

3.3 Volumetric flask (v.f.)

3.4 Hamilton Autodiluter

3.5 Various graduated cylinders

3.6 Stir bars

3.7 ACS grade Potassium Chloride (KCl)

3.8 Ethylenediamne tetraacetic acid disodium salt dihydrate (EDTA)

3.9 ACS grade Sodium hydroxide (NaOH)

3.10 Sodium phosphate dibasic Heptahydrate (SPDH)

3.11 Sodium salicylate

3.12 Sodium nitroprusside

3.13 Sodium hypochlorite

3.14 ACS grade Ammonium Chloride

4.0 Reagents and Standards
***Note strong bases and acids will be used: caution is advised. All concentrated acids and bases should be handled in the fume hood in 413.**

4.1 Reagent 1: EDTA Solution

4.1a Dissolve 66g EDTA in about 500 mL of DI water
4.1b Adjust pH to 7.0 with 5M NaOH
4.1c Dilute to 1000 mL volume in a 1000 mL v.f.
4.1d Store in HDPE screw top bottle, good for 2 months.

4.2 Reagent 2: Buffer

4.2a Dissolve 28.0g NaOH and 50.0 g SPDH in 800 mL DI water using a 1000 mL v.f. Bring to volume
4.2b Store in HDPE screw top bottle, good for 2 months

4.3 Reagent 3: Salicylate- Nitroprusside Color reagent

4.3a Dissolve 150g Sodium Salicylate and 1.0g Sodium Nitroprusside in 800 mL DI water in a 1000 mL v.f. Bring to volume
4.3b Store in light-proof screw top bottle, good for 1 month

4.4 Reagent 4:Hypochlorite Reagent

4.4a Dilute 250ml 5.25% sodium hypochlorite with DI water in a 500 mL v.f.
4.4b Store in HDPE screw top bottle, good for 1 month

4.5 Ammonia Standard: 500.0 mg N/L as NH_3

4.11a Stock Solution: Dissolve 1.9095g of ammonium chloride in 800 mL matrix in a 1000 mL v.f. Bring to volume

4.6 Ammonia Check: 500.0 mg N/L as NH_3

4.11a Stock Solution: Dissolve 1.9095g of ammonium chloride in 800 mL matrix in a 1000 mL v.f. Bring to volume

4.7 Blank Standard: Matrix

Standard Operating Procedure

Ammonia operation

Soil Environmental Chemistry Program, The Ohio State University

Version 2

5.0 Sample Collection, Preservation and Storage

5.1 Samples to be analyzed for NH4 should be stored in the refrigerator and run within 48 hours. For storing samples longer than 48 hours, freeze to -22C in the dark for up to 12 weeks.

5.2 Samples should be filtered with a .45um Nylon filter that have been checked for contaminants to make sure all particles are free from the sample and to avoid contamination.

5.3 Sample Runlists

5.3.1 Sample runlists (ie. Lachat 19-1.xls) should be created and stored to Wdrive>SEC Lab>Lachat> >year (i.e 2019)>Runlist. A runlist template is in Wdrive>SEC Lab>Lachat.

5.4.2. Each time the method is started or stopped and restarted, a new file is created. Files are named with a timestamp. Record the file name on the runlist alongside the samples in that run. It is helpful to record the time or file name on paper during a run.

6.0 Lachat Setup

6.1 Turn on all instrument components and then start the OMNION software.

6.2 Some of the reactions are temperature-dependent, so the reagents and samples should be brought to room temperature before analysis. This can be expedited by placing items in a water bath.

6.3 Clamp down tubing on the pump, Turn on pump by hitting manual start/stop, and ensure the speed is 35.

6.4 Pump DI water through all the reagent lines and check for leaks and smooth flow. The ammonia chloride line should be kept in a separate DI container than the reagents for the NH4 line to avoid contamination. Select the preview icon to observe the baseline. Check that the baseline is smooth and near zero. A choppy or wavy baseline indicates poor flow.

6.5 Switch to reagents one by one in the order they appear on the pump, from front to back, to prevent staining of the lines. Wait for the system to equilibrate until a stable baseline is achieved for each one. When all reagents are flowing, baseline should be below 1.0 V. Higher voltage indicates contamination in the system (more likely) or a bad reagent (less likely as long as reagents are not expired). If contamination is suspected, replace 

the DI water and return all lines, aside from the color lines, to the DI. Add reagents one by one to isolate the source. Remake contaminated reagent.

6.6 Pressing the "MIN" button on the pump reduces the speed to 4. This is useful for preserving reagents while samples are not being run (for example, when time is needed to troubleshoot or prep samples). Return the pump to 35 by pressing manual start/stop twice and allow to equilibrate before running (baseline will shift at different pump speeds).

6.7 Templates are in Documents> OmnionData> OSU>Templates> NH4

6.7.1 Open template

6.7.2 Modify the template if necessary and save the file in Document>Omnion Data>OSU> Data>(Current year)>(runlist #)

When switching between runs, the system will prompt you to change the heater settings to the newly selected run. Maintaining the heater settings of your run will hasten the run, as it takes several minutes for the heater to change temperature.

6.8 Templates are created by inputting the following data system parameters. Ensure that these are the values included in the template:

In the timing Tab:

Run:

Cycle period: 90 s

Concentration units: mg N/L

Sampler timing:

Min. probe in wash period: 40 s

Probe in sample period: 30 s

Ammonia Channel:

Peak Base Width: 30 s

% Width Tolerance 100

Threshold: 5000

Inject to peak start: 46 s

Chemistry: Direct

Heater: 60^{o}C

Calibration Data:

Calibration Rep: Average

Calibration fit type: 2nd order polynomial

Weighting method: none

Force through zero: no

Valve timing:

Load Period: 18 s

Inject period: 42 s

Time to Valve: 27 s

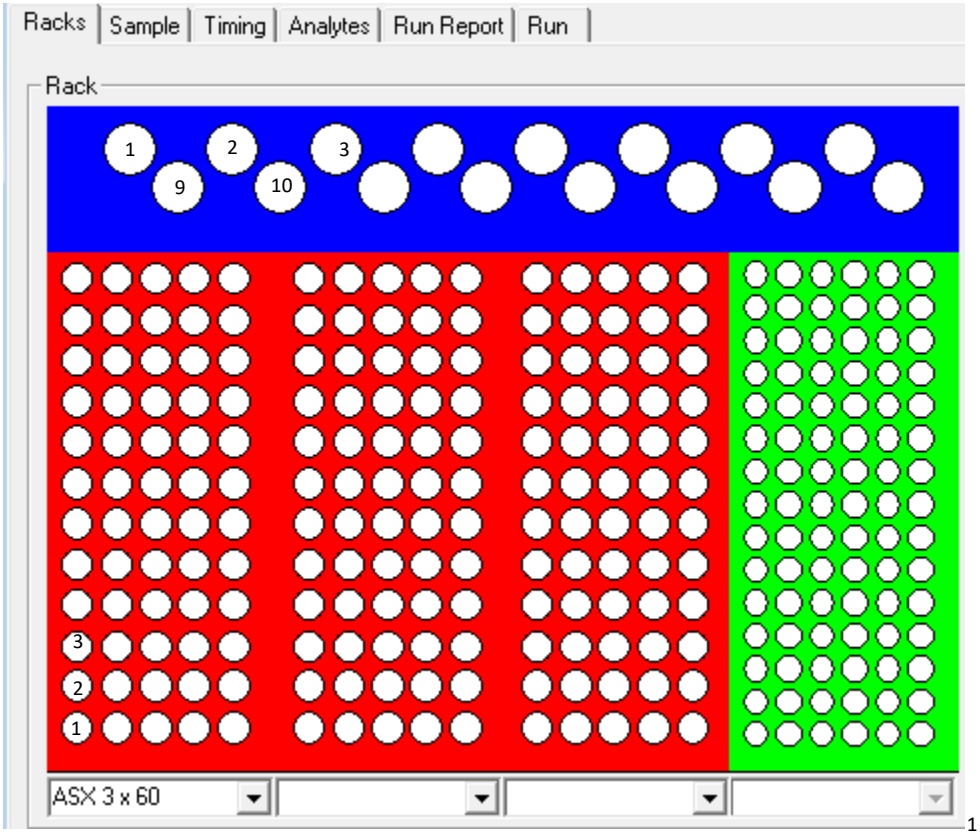
6.9 Prepare 50 mL of working solutions from each of the standard and check solutions using the sample matrix as dilutent. The concentration of the standard working solution should be 20mg N/L and the check working solution 10mg N/L.

6.10 Prep the autodiluter (The lachat will create the standard curve from the working standard solution using the autodiluter, as well as dilute samples as needed).

6.10a Using the Autodiluter
6.10a1 Put standard in its position
6.10a2 In Omnion select where the standard is going to be taken from.
6.10a3 If it need diluted click the ADF check mark and impute the auto dilution factor in the ADF box
6.10a4 Then under sample in run properties, impute the known concentration of the standard you are achieving by dilution.

6.11 Double check that the test tube racks are the test tube racks being used. That is under the racks tab. There should be a 16 rack for standards (blue), 3 racks of 60 for samples (red), followed by a 90 rack (green) for dilution tubes. The sample and dilution tube racks are numbered from front to back, left to right, while the standard tubes are numbered from left to right, back to front (see image below).

Standard Operating Procedure
Ammonia operation
Soil Environmental Chemistry Program, The Ohio State University
Version 2



6.12 Use the standard working solution to create a calibration curve.

6.12a Calibration curve should have a straight line and r= 1±.0010

6.13 Run the Check standard and blank after calibration is completed

6.14 Before running samples create a Composite (Comp), comp spk, and comp x5 solution.

6.14.1 **Comp**- make a solution with a mixture that is a composite of all the samples going to be run. Make about 15 ml of solution.

6.14.2 **Comp spk**- Take 5 ml of comp solution and add 0.125 mL10 mg N/L Ammonia standard working solution.

6.14.3 **Comp x5-** dilute Comp solution x5

6.14.4 Calculate the % recovery for each. Should be approx. ±10%

6.15 Run samples as unknowns and check standard and blank after every 10 samples.

6.15a. Add sample runlist by right clicking the last sample in the run and selecting append many. Insert the desired number of samples.

7.0 Quality Control

7.1 Run the Check standards, 1.0 mg N/L as prepared in 6.7 and blank on the Lachat.

7.1.1 Results: should fall with in ±10%

7.2 Check standards as prepared in 6.7 and blank should be run after ever 10 samples.

7.2.1 If check standards fail then recalibrate instrument and re-run the previous 10 samples and check standards and blank

7.2.2 If it continues to fail then remake standards and recalibrate and test using check standards

7.2.3 If check standards fail then remake check standards and re-run check standard

7.3 After the instrument has equilibrated and all reagents are flowing during setup, baseline should be < 1.0 V. If this is not the case, then check the lines one by one to isolate the source of the elevated baseline and remake reagents as necessary.

8.0 Data Summary

8.1 Data automatically goes to excel sheet in the Document>Omnion Data>OSU> Data>(Current year).

8.1.1 This can be found and/or modified in the Configuration tab>Options>Data export> Data Items.

8.2 See Lachat Summary SOP for Data Summary.

9.0 Lachat Power off procedure

9.1 Move lines to the DI containers (ensuring the ammonium chloride line is separate from the NH4 lines) for 10 minutes

9.2 Allow air to pump through lines until no liquid remains.

9.3 Restart Auto-sampler so that the sampling wand ends out of the rinse solution.

9.4 Turn off all devices and computer

10.0 Waste

10.1 All Waste lines from the Lachat should be positioned into a carboy, not down the sink

12.0 References

12.1 Hofer, S. Determination of Ammonia (Salicylate) in 2 M KCl Soil Extracts by Flow Injection Analysis. QuickChem Method 12-107-06-2-A. 2003

12.2 Lachat Applications Group. Determination of Nitrate in 2 M KCl Soil Extracts by Flow Injection Analysis Calorimetry. QuickChem Method 12-107-04-1-F. 2015

12.3 Lachat Applications Group. Determination of Ortho Phosphate in Waters by Flow Injection Analysis Calorimetry. QuickChem Method 10-115-01-1-A. 2007

12.4 O'Dell, J.W. Determination of Ammonia Nitrogen by Semi-Automated Colorimetry. Environmental Monitoring Systems Laboratory. United States Environmental Protection Agency. Method 350.1. 1993

12.5 O'Dell, J.W. Determination of Nitrate-Nitrite by Automated Colorimetry. Environmental Monitoring Systems Laboratory. United States Environmental Protection Agency. Method 353.2. 1993

12.6 United States Environmental Protection Agency. Method 365.3: Phosphorous, All Forms (Colorimetric, Ascorbic Acid, Two Reagent). 1978