

Nitrate, Nitrite and Nitrate/Nitrite Nitrogen

Reference: Methods 353.2: Methods for the Determination of Inorganic Substances in Environmental Samples, EPA 600/ R-93/ 100. August, 1993.

Methods 4500NO₃-F, 4500NO₂-B: Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

Method 10-107-04-1, Lachat Instruments, 6645 West Mill Road, Milwaukee, WI 53218, 1992

1. Scope and Application

Matrices: This method is limited to optically clear water samples with a total concentration of nitrite and nitrate below 8mg N/L.

Definitions: Refer to Alpha Analytical Quality Manual.

In waters and wastewaters, the forms of nitrogen of greatest interest are, in order of decreasing oxidation state, nitrate, nitrite, ammonia, and organic nitrogen. All these forms of nitrogen, as well as nitrogen gas (N₂), are biochemically interconvertible and are components of the nitrogen cycle. They are of interest for many reasons.

Organic nitrogen is defined functionally as organically bound nitrogen in the trinegative oxidation state. It does not include all organic nitrogen compounds. Analytically, organic nitrogen and ammonia can be determined together and have been referred to as "kjeldahl nitrogen," a term that reflects the technique used in their determination. Organic nitrogen includes such natural materials as proteins and peptides, nucleic acids and urea. Numerous concentrations vary from a few hundred micrograms per liter in some lakes to more than 20mg/L in raw sewage.

Total oxidized nitrogen is the sum of nitrate and nitrite nitrogen. Nitrate generally occurs in trace quantities in surface water but many attain high levels in some groundwater. In excessive amounts, it contributes to the illness known as methemoglobinemia in infants. A limit of 10mg nitrate as nitrogen/L has been imposed on drinking water to prevent this disorder. Nitrate is found only in small amounts in fresh domestic wastewater but in the effluent of nitrifying biological treatment plants, nitrate may be found in concentrations of up to 30mg nitrate as nitrogen/L. It is an essential nutrient for many photosynthetic autotrophs and has been identified as a growth-limiting nutrient.

Nitrite is an intermediate oxidation state of nitrogen, both in the oxidation of ammonia to nitrate and in the reduction of nitrate. Such oxidation and reduction may occur in wastewater treatment plants, water distribution systems, and natural waters. Nitrite can enter a water supply system through its use as a corrosion inhibitor in industrial process water. Nitrite is the actual etiologic agent of methemoglobinemia. Nitrous acid, which is formed from nitrite in acidic solution, can react with secondary amines (RR'NH) to form nitrosamines (RR'N-NO), many of which are known to be carcinogens. The toxicologic significance of nitrosation reactions in vivo and in the natural environment is the subject of much current concern and research.

Within this SOP, organic nitrogen is referred to as organic N, nitrate nitrogen as NO₃⁻-N, and nitrite nitrogen as NO₂⁻-N.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Lachat Analyzer and in the interpretation of Lachat data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Nitrate is quantitatively reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite (reduced nitrate plus original nitrite) is then determined by diazotizing with sulfanilamide followed by coupling with N-(1-naphthyl)ethylenediamine dihydrochloride. The resulting water-soluble dye has a magenta color, which is read at 520nm. Nitrite alone can be determined by removing the cadmium column. The nitrate is calculated as the difference between the reduced and non-reduced sample

2.1 Method Modifications from Reference

Soils can be analyzed using 1:10 ratio soil to water extraction, following filtration.

3. Reporting Limits

This method has an analytical range of 0.1 to 8.0mg N/L in the form of nitrate, and 0.05 to 8.0mg N/L in the form of nitrite.

The Reporting Limit is 0.1mg/L for Nitrate and 0.05 mg/L for Nitrite. Reporting limit is 1.0 mg/kg for soils

4. Interferences

- 4.1 Suspended matter in the column will restrict sample flow.
- 4.2 For turbid samples, filter through 0.45µm membrane filter prior to analysis.
- 4.3 Low results would be obtained for samples that contain high concentrations of iron, copper or other metals. In this method, EDTA is added to the buffer to reduce this interference.
- 4.4 Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. In this case, only the water phase of the sample is used for analysis and a narrative is submitted with the data. Dilutions are performed as necessary.
- 4.5 Residual chlorine can interfere by oxidizing the Cd column, reducing its efficiency. Prior to analysis, check wastewater and drinking water samples for residual chlorine and record results in the Laboratory Notebook. If residual chlorine is present, and the samples are preserved with H₂SO₄, the sample may be analyzed for NO₃/NO₂ determination. However, NO₂ must be performed by a manual method. If it is not possible to analyze NO₂ by a manual method, the result is reported as NA and a narrative is submitted.
- 4.6 Sample color interferes if it is absorbed at about 540nm.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in glass or plastic bottles; 250mL minimum volume. Soils can be collected in plastic or glass containers.

6.2 Sample Preservation

Refrigerate samples at 4 ± 2 °C.

For Nitrate/Nitrite analysis, the samples are preserved with 1:1 H₂SO₄.

6.3 Sample Shipping

No specific requirement.

6.4 Sample Handling

Begin NO₃⁻ and/or NO₂⁻ determinations promptly after sampling. If storage is necessary, store for up to 48 hours at 4 ± 2 °C.

NOTE: If the 48-hour hold time cannot be met, the sample is to be handled as follows, only in an emergency situation. These instructions are not to be used on a regular basis.

Prior to the expiration of the 48-hour hold time, the following three steps are executed:

1. A manually colored Nitrite test is performed by Method 354.2. Results are recorded in the Laboratory Notebook.
2. A 50mL aliquot of the sample is preserved to a pH of <2 with concentrated H₂SO₄. Preservation is recorded in the Laboratory Notebook.

Prior to analysis, within 14 days of preservation, the preserved sample is neutralized using 6N NaOH. The sample is analyzed using only the Lachat Instrument.

CAUTION! Samples must NOT be preserved with mercuric chloride or thiosulfate because this will degrade the cadmium column

7. Equipment and Supplies

7.1 Lachat 8000 Automated Ion Analyzer or Lachat QuickChem 8500 Automated Ion Analyzer

7.2 Nitrate+Nitrite Lachat Board

7.3 Nitrite Lachat Board

7.4 Pre-packed Cadmium Columns: Available from Lachat.

7.5 Ottawa sand.

7.6 Disposable Culture Tubes 13x100 ml

7.7 Disposable pipettes.

8. Reagents and Standards

8.1 Stock Nitrate Standard, 1000mg N/L as NO_3^- : Purchased commercially prepared with certificate of analysis. Expires upon manufacturer's expiration date. There must be different manufacturers for calibration stock and ICV/LCS stock.

8.1.1 Stock Nitrate Standard, 200.0mg N/L as NO_3^- : Pipet 50mL of 1000ppm standard (Section 8.1) into 250mL volumetric flask and bring to volume with DI.

Alternately, in a 1L volumetric flask, dissolve 1.444g potassium nitrate (KNO_3) in about 600mL DI. Add 2mL chloroform. Dilute to the mark with DI and invert to mix. Refrigerate at $4\pm 2^\circ\text{C}$. This solution is stable for six months.

8.2 Stock Nitrite Standard, 1000mg N/L as NO_2^- : Purchased commercially prepared with certificate of analysis. Expires upon manufacturer's expiration date. There must be different manufacturers for calibration stock and ICV/LCS stock.

8.2.1 Stock Nitrite Standard, 200.0mg N/L as NO_2^- : Pipet 50mL of 1000ppm standard (Section 8.2) into 250mL volumetric flask and bring to volume with DI.

Alternately, in a 1L volumetric flask, dissolve 0.986g sodium nitrite (NaNO_2) or 1.214g potassium nitrite (KNO_2) in approximately 800mL DI. Add 2mL chloroform. Dilute to the mark with DI and invert to mix. Refrigerate at $4\pm 2^\circ\text{C}$. This solution is stable for six months.

8.3 Intermediate Nitrate Working Standard, 20 mg N/L as Nitrate: To a 250mL volumetric flask, add 25.0mL of the 200mg N/L NO_3^- stock standard. Dilute to the mark with DI and invert to mix. These solutions are stable for two weeks. Refrigerate at $4\pm 2^\circ\text{C}$.

8.4 Intermediate Nitrite Working Standard, 20 mg N/L as Nitrite: To a 250mL volumetric flask, add 25.0mL of the 200mg N/L NO_2^- stock standard. Dilute to the mark with DI and invert to mix. These solutions are stable for two weeks. Refrigerate at $4\pm 2^\circ\text{C}$.

8.5 Set of Six Calibration NO_3^- Standards, 8.0, 4.0, 1.00, 0.40, 0.20 and 0.1mg N/L as Nitrate: These standards are stable for 2 weeks. Refrigerate at $4\pm 2^\circ\text{C}$.

To four 200mL volumetric flasks, add respectively: 8.0, 4.0, 1.0 and 0.4mL of the 200mg N/L NO_3^- stock standard. Bring to volume with DI water.

To two 200mL volumetric flasks, add respectively: 2.0 and 1.0mL of the 20mg N/L NO_3^- intermediate standard. Bring to volume with DI water.

Alternatively, an autodiluter can be used to make the standards during calibration, in which case only 8.0ppm and 1.0 ppm need to be manually prepared. If an autodiluter is used then it must be checked in an analytical tray by autodiluting 8.0mg N/L as Nitrite. The recovery for NO_2 must be within 10% of the true value.

8.6 Set of Six Calibration NO_2^- Standards, 8.0, 4.0, 1.00, 0.40, 0.10 and 0.05mg N/L as Nitrite: These standards are stable for 2 weeks. Refrigerate at $4\pm 2^\circ\text{C}$.

To three 200mL volumetric flasks, add respectively: 8.0, 4.0 and 1.0 of the 200mg N/L NO_2^- stock standard. Bring to volume with DI water.

To three 200mL volumetric flasks, add respectively: 4.0, 1.0mL and 0.5mL of the 20mg N/L NO_2^- intermediate standard. Bring to volume with DI water.

Alternatively, an autodiluter can be used to make the standards during calibration, in which case only 8.0ppm and 1.0 ppm need to be manually prepared.

- 8.7 Ammonium Chloride Buffer, pH 8.5:** In a 2L volumetric flask, dissolve 170g ammonium chloride (NH_4Cl) and 2.0g disodium ethylenediamine tetraacetic acid dihydrate ($\text{Na}_2\text{EDTA}\cdot 2\text{H}_2\text{O}$) in about 800mL water. Dilute to the mark with DI water and invert to mix. Adjust the pH to 8.5 with concentrated ammonium hydroxide. This solution is prepared monthly and stored at room temperature.
- 8.8 Sulfanilamide Color Reagent:** To a 2L volumetric flask add about 1200mL water. Then add 200mL of 85% phosphoric acid (H_3PO_4), 80.0g sulfanilamide, and 2.0g $\text{N}^-(1\text{-naphthyl})$ ethylenediamine dihydrochloride (NED). Shake to wet, and stir to dissolve for 30 minutes. Dilute to the mark with DI water and invert to mix. Store in a dark bottle. This solution is stable for one month. Store at room temperature.
- 8.9 200ppm Nitrate Stock Standard, (for ICV/LCS):** Pipet 50mL of 1000ppm standard (Section 8.1) into 250mL volumetric flask and bring to volume with DI. Store refrigerated at $4\pm 2^\circ\text{C}$. Expires six months from preparation or upon manufacturer's expiration date.
- 8.10 200ppm Nitrite Stock Standard:** Pipet 50mL of 1000ppm standard (Section 8.2) into 250mL volumetric flask and bring to volume with DI. Store refrigerated at $4\pm 2^\circ\text{C}$. Expires six months from preparation or upon manufacturer's expiration date.
- 8.11 Initial Calibration Verification Standard (ICV)/Laboratory Control Sample (LCS):** Store refrigerated at $4\pm 2^\circ\text{C}$. Expiration is 2 weeks from date of preparation.
- 8.11.1 Nitrate LCS, 5.0ppm:** Pipet 5.0mL of 200ppm stock (Section 8.9) into a 200mL volumetric flask and bring to volume with DI.
- 8.11.2 Nitrate ICV, 0.5ppm:** Pipet 10.0mL of 5.0ppm standard (Section 8.11.1) into a 100mL volumetric flask and bring to volume with DI.
- 8.11.3 Nitrite LCS, 5.0ppm:** Pipet 5.0mL of 200ppm stock (Section 8.9) into a 200mL volumetric flask and bring to volume with DI.
- 8.11.4 Nitrite ICV, 0.5ppm:** Pipet 10.0mL of 5.0ppm standard (Section 8.11.3) into a 100mL volumetric flask and bring to volume with DI.
- 8.12 DPD Free Chlorine Reagent Powder Pillows:** HACH brand, for 25mL sample. Store at room temperature. Expires upon manufacturer's expiration date.
- 8.13 1N Hydrochloric acid (HCL):** To a 1L volumetric flask add about 600mL DI. Then add 83mL of concentrated hydrochloric acid (HCL) Stir to dissolve. Dilute to the mark with DI water and invert to mix. This solution is stable for six month. Store at room temperature.
- 8.14 1N Sodium Hydroxide (NaOH):** To a 1L volumetric flask add about 600mL DI. Then add 40 g of sodium Hydroxide. Stir to dissolve. Dilute to the mark with DI water and invert to mix. This solution is stable for six month. Store at room temperature
- 8.15 6N Sulfuric Acid (H_2SO_4):** To a 1L volumetric flask add about 600mL DI. Then add 140 ml of concentrated Sulfuric Acid (H_2SO_4). Stir to dissolve. Dilute to the mark with DI water and invert to mix. This solution is stable for six month. Store at room temperature

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

One Method Blank is analyzed per batch of 20 samples or less. The Method Blank consists of DI.

For soils: 5g of Ottawa sand extracted with 50 ml of DI. Results must be $< 0.1\text{mg/L}$. If this criterion is not met, the blank is re-analyzed. If there is still failure, the problem must be found and corrected prior to any sample analysis.

9.2 Laboratory Control Sample (LCS)

The 5ppm ICV is reported as the LCS for the batch. See Section 9.3.

For soil LCS: 5g of Ottawa sand extracted with 0.25 ml of 1000 mg/l nitrate (8.1) (or 1000 mg/l Nitrite standard (8.2)) and 50 ml DI. The nitrate standard is used for spikes for Nitrate-N as well as Nitrate/Nitrite-N. LCS recoveries must be recovered within $\pm 10\%$ of the true value. If these criteria are not met, LCS's must be re-analyzed. If failure continues, the batch has to be re-extracted and re-analyzed.

9.3 Initial Calibration Verification (ICV)

Two ICVs are analyzed at the beginning of the analytical sequence. One is at a concentration of 0.5ppm, and the other is at a concentration of 5.0ppm.

Both must be recovered within $\pm 10\%$ of the true value. If these criteria are not met, the ICVs must be re-analyzed. If failure continues, the ICVs are to be re-made and/or a new calibration curve is to be generated.

The 5ppm ICV is reported as the LCS for the batch.

9.4 Continuing Calibration Verification (CCV)

At the beginning of the first tray, after every ten samples and at the end of every analytical sequence, a CCV and a CCB pair must be analyzed to verify both calibration curves.

1.0ppm Nitrate CCV (Section 8.5)

1.0ppm Nitrite ICV (Section 8.6)

Calibration Blank (DI)

The results of the CCVs must be within $\pm 10\%$ of the true value, otherwise re-calibration is required.

The results of the CCBs must be less than our standard limit of detection, otherwise the analysis is stopped and the problem corrected.

9.5 Matrix Spike

One Matrix Spike is analyzed per batch of 20 samples or less. Separate spikes are performed for Nitrate and Nitrite. In a 25mL volumetric flask, 0.5mL of 200ppm stock calibration standard (Section 8.1 or 8.2) is added to the sample. The final concentration of the matrix spike is

4.0ppm. The nitrate standard is used for spikes for Nitrate-N as well as Nitrate/Nitrite-N. The nitrite standard is used for spikes for Nitrite-N.

For soils: weigh 5.0 g of sample, add 2.0 ml of 200 mg/l Nitrate or Nitrite standard and 48 ml of DI. The final concentration of the matrix spike is 80.0 mg/kg. The nitrate standard is used for spikes for Nitrate-N as well as Nitrate/Nitrite-N. The nitrite standard is used for spikes for Nitrite-N.

% Recovery for the Matrix Spike must be within in-house control limits. If acceptance criteria are not met, the Matrix Spike is reanalyzed. If failure continues, a narrative is included with the data for inclusion on the Client report.

Note: For samples, analyzed by method 353.2 (NO₂-353 and NO₃-353) maximum batch size is 10 samples; every 10 samples required separate matrix spike (MS) to be analyzed. % Recovery for the Matrix Spike must be within +/- 10% of true value. If acceptance criteria are not met, the Matrix Spike is reanalyzed. If failure continues, a narrative is included with the data for inclusion on the Client report.

9.6 Laboratory Duplicate

One Duplicate sample is analyzed per batch of 20 samples or less. A separate aliquot of the sample is analyzed for this purpose.

% RPD for the Duplicate must be within in-house control limits. If acceptance criteria are not met, the Duplicate is reanalyzed. If failure continues, a narrative is included with the data for inclusion on the Client report

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- Calibration
- ICV/LCS – both levels
- Sample analysis
- CCV – every ten samples and at the end of the analytical sequence

10. Procedure

10.1 Equipment Set-up

10.1.1 Preparation

- 10.1.1.1** Place the Nitrate+Nitrite board (containing the cadmium column) in Channel 1. Place the Nitrite board in Channel 2. Make sure the valve to the cadmium column is closed prior to starting to pump the reagents.
- 10.1.1.2** Commence pumping of reagents.
- 10.1.1.3** Once the lines are full of reagent and free of gas bubbles, open the valve to allow reagent to flow through the cadmium column.

NOTE: Be sure to switch the valve back before rinsing the manifold with DI water at the completion of the run.

NOTE: DO NOT LET AIR ENTER THE CADMIUM COLUMN.

10.1.2 Column Efficiency Procedure

10.1.2.1 Visually inspect the column. Check for air bubbles in the column or lines, gaps in the column or any change in the cadmium surface characteristics, (cadmium granules should be dark gray). If air bubbles are present in column, connect the column into the manifold, turn the pump on maximum and tap firmly with a screwdriver handle, being careful not to break the column, working up the column until all air is removed. If air cannot be removed, the column should be repacked. Cadmium columns should be stored filled with buffer. If air enters the column, efficiency will decrease. Check the flow efficiency by disconnecting the cadmium column from the manifold and reconnecting to a green pump tube. Pump buffer through the packed column and collect in a graduated cylinder. The flow rate with the column connected should be greater than 4.0 mL/minute.

10.1.2.2 Column Efficiency – Slope Ratio Method: Calibrate with the mid-range NO₃-N standards. Calibrate with a matching concentration range of NO₂-N standards. The column efficiency is determined by the equation:

$$E = \frac{S_{\text{NO}_3\text{-N}}}{S_{\text{NO}_2\text{-N}}} \times 100$$

where:

$S_{\text{NO}_3\text{-N}}$ = slope of NO₃ calibration
 $S_{\text{NO}_2\text{-N}}$ = slope of NO₂ calibration
 E = % efficiency

10.1.2.3 Column Efficiency – Concentration Ratio Method: Calibrate with the mid-range NO₂-N and NO₃-N standards. Run a known concentration NO₂-N standard. Run a matching concentration NO₃-N standard. The column efficiency is determined by the following equation:

$$E = \frac{C_{\text{NO}_3\text{-N}}}{C_{\text{NO}_2\text{-N}}} \times 100$$

where:

$C_{\text{NO}_3\text{-N}}$ = concentration of NO₃ standard
 $C_{\text{NO}_2\text{-N}}$ = concentration of NO₂ standard
 E = % efficiency

10.1.2.4 Column Efficiency Result: If the efficiency is <75%, the column is repacked. All results are recorded and maintained on file in the QC department.

10.1.3 Residual Chlorine Screening

Check all wastewater and drinking water samples for residual chlorine prior to analysis.

10.1.3.1 Add 1 DPD Free Chlorine powder pillow (Section 8.12) to 25mL of sample in a centrifuge tube. An immediate color change to pink indicates residual chlorine is present. If residual chlorine is present, add a small amount of ascorbic acid to a sample aliquot (record this in logbook) and check for residual chlorine presence again. If residual chlorine remains, notify the Department Manager and/or the Laboratory Director. Results will be reported as Not Applicable (N/A).

If residual chlorine is not present, continue with sample analysis.

10.2 Initial Calibration

Calibrate the Lachat ion analyzer according to manufacturer's instructions.

10.2.1 Calibration

Two boards are used to calibrate the Lachat instrument. Each curve has seven calibration points. The correlation coefficient of each curve must be ≥ 0.995 , otherwise recalibration is necessary. Prepare standard curves by plotting the peak areas of standards processed through the manifold against $\text{NO}_3 + \text{NO}_2$ as N and NO_2 as N concentrations in standards.

10.2.1.1 Channel 1 is used to generate a calibration curve for Nitrate/Nitrite ranging from 0 to 8.0ppm.

10.2.1.2 Channel 2 is used to generate a calibration curve for Nitrite ranging from 0 to 8.0ppm.

Note: Instrument is calibrated daily, fixed calibration range is used; linearity is verified daily; three standards are used for linear calibration verification (low ICV (0.5 mg/l), High ICV (5.0 mg/l) and CCV (1.0 mg/l)). All standards must be within 10% of true value

10.2.2 Initial Calibration Verification (ICV)

10.2.2.1 Prior to sample analysis, the following ICVs must be analyzed to verify both calibration curves.

10.2.2.1.1 Nitrate ICV, 0.5ppm (Section 8.12.2)

10.2.2.1.2 Nitrate ICV, 5.0ppm (Section 8.12.1)

10.2.2.1.3 Nitrite ICV, 0.5ppm (Section 8.12.4)

10.2.2.1.4 Nitrite ICV, 5.0ppm (Section 8.12.3)

10.2.2.2 The results must be within $\pm 10\%$ of the true value, otherwise re-calibration is required.

10.3 Equipment Operation and Sample Processing

Follow the manufacturer's directions for the operation of the Lachat 8000.

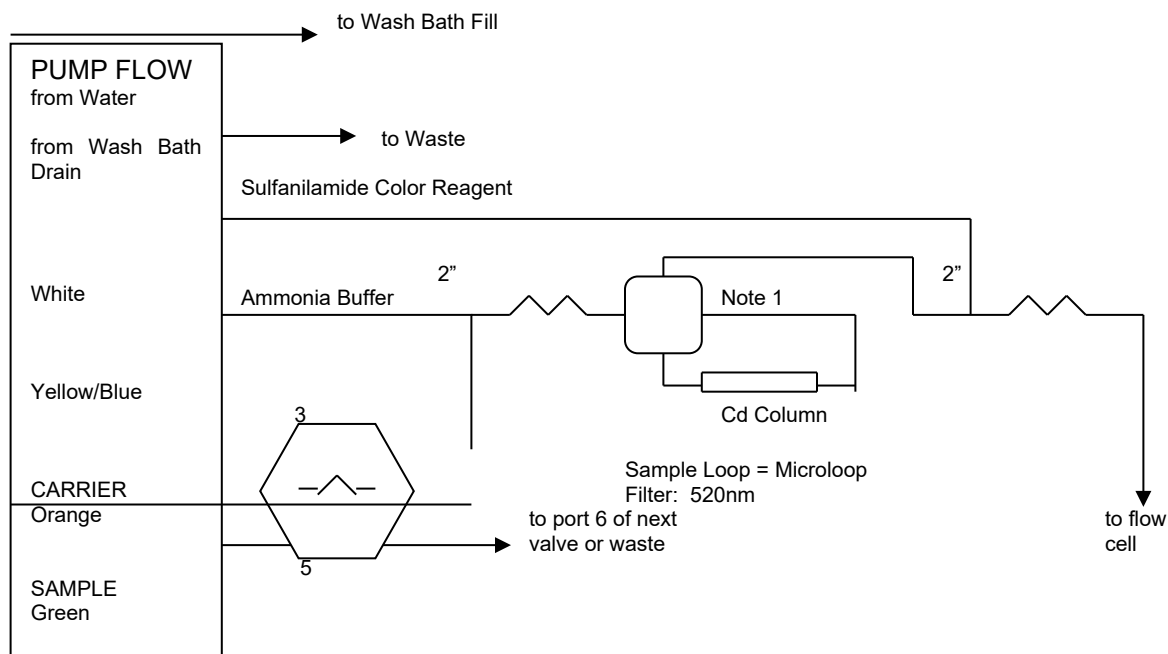
All samples have to be inspected prior to analysis. Samples that are turbid or have sediment have to be filtered prior to analysis.

Check pH of the samples. If pH is less than 5 or greater than 9, then adjust pH using 1N Hydrochloric Acid (HCl) (8.13), 6N Sulfuric Acid (H_2SO_4) (8.15) or 1N Sodium Hydroxide (NaOH) (8.14). Record pH adjustment in the log book.

For soils: extract soils samples prior to analysis: take 5g of sample, add 50 ml of DI, extract for 30 min, then filter thorough 0.45 μm filter. Record all weights for calculations.

Note: if samples are filtered, then Method blank also have to be filtered

The Manifold Diagram follows:



CARRIER is water.

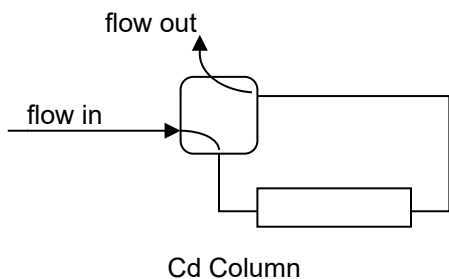
2" is 135cm of tubing on a 2-inch coil support.

APPARATUS: Standard valve, flow cell, and detector head modules are used.

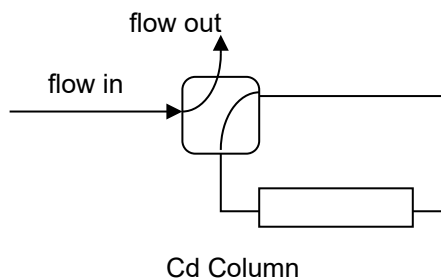
All manifold tubing is 0.8mm (0.032") i.d. This is 5.2 μ L/cm.

NOTE 1: This is a two-state switching valve used to place the cadmium column in line with the manifold.

State 1: Nitrate + Nitrite



State 2: Nitrite Only



10.4 Continuing Calibration

See Section 9.4

10.5 Preventive Maintenance

Tubing is changed monthly or as needed.

At the end of each analytical sequence, the valve to the column is closed. DI is rinsed through the Lachat for five minutes followed by five minutes of air.

All maintenance is documented in the Instrument Maintenance Logbook.

11. Data Evaluation, Calculations and Reporting

11.1 Nitrate/Nitrite: When the software is set up according to the manufacturer's recommendations, the concentration of nitrate plus nitrite in mg NO₃/NO₂-N/L is reported directly when the Cd column is included in the sample train in Channel 1.

11.2 Nitrite: When the software is set up according to the manufacturer's recommendations, the concentration nitrite in mg NO₂-N/L is reported directly when the Cd is not included in the sample train in Channel 2.

11.3 Nitrate: The concentration of nitrate is determined by the subtraction of the nitrite concentration, (Section 11.2 above), from the nitrate-nitrite concentration, (Section 11.1 above).

11.3.1 If the sample was preserved initially as described in Section 6.3, subtract the Nitrite value generated manually from the Nitrate/Nitrite value generated by the Lachat Instrument. This value is reported as the Nitrate result.

When the sample is preserved initially as described in Section 6.3, the value generated by the Lachat instrument for Nitrite is invalid and therefore disregarded.

11.4 If any sample exceeds a concentration of 8.0 mg/L, the sample must be diluted and re-analyzed. All sample concentrations must fall within the calibration curve.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

16. Attachments

None.