Standard Operating Procedure for Ammonia (Lachat Method)

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1.0 Scope and Application

1.1 This method covers the determination of ammonia in lake water.

1.2 The approximate working range is 0.02 to 2.00 mg-N(as NH_3)/L. The method detection limit is 0.02 mg-N/L.

2.0 Summary

When ammonia is heated with salicylate and hypochlorite in an alkaline phosphate buffer, an emerald green color is produced which is proportional to the ammonia concentration. The color is intensified by the addition of sodium nitroprusside.

3.0 Sample Handling and Preservation

3.1 Samples are collected in clean glass or plastic containers.

3.2 Samples are preserved by the addition of 1 mL of concentrated sulfuric acid per liter of sample.

4.0 Interferences

4.1 In alkaline solutions, calcium and magnesium will interfere by forming a precipitate which scatters light. EDTA is added to the buffer to prevent this interference.

4.2 Sample turbidity may interfere. Turbid samples may be decanted or filtered prior to analysis.

5.0 Apparatus

5.1 13 X 100 mm Test Tubes

5.2 Lachat QuikChem AE

5.2.1 Ammonia manifold (Lachat method number 10-107-06-2-C)

5.2.2 XYZ Sampler

5.2.3 Printer

6.0 Reagents and Standards

6.1 All reagents should be stored in the appropriate bottles and labeled with the following information:

Identity:	(Buffer)
Date:	(mm/dd/yy)
Initials of Preparer:	(M.S.)

All standards should be stored in appropriate bottles and labeled as above with the following also included:

Concentration:	(1000 mg-N/L)

6.2 Use deionized water for all solutions.

6.3 Buffer: In a 1 L volumetric flask dissolve 30.0 g sodium hydroxide (NaOH), 25.0 g ethylenediaminetetraacetic acid, disodium salt dihydrate, and 67 g sodium phosphate dibasic heptahydrate ($Na_2HPO_4 \cdot 7H_2O$) in about 900 mL of water. Dilute to the mark and invert to mix. De-gas with helium.

6.4 Salicylate-Nitroprusside Color Reagent: In a 500 mL volumetric flask, dissolve 72 g sodium salicylate (salicylic acid sodium salt, $[C_6H_4(OH)(COO)Na]$) and 1.75 g sodium nitroprusside (sodium nitroferricyanide dihydrate, $[Na_2Fe(CN)_5NO \cdot 2H_2O)$, in about 400 mL water. Dilute to the mark. Stir or shake to dissolve. Refrigerate. Prepare fresh weekly. De-gas with helium.

6.5 Hypochlorite Reagent: In a 1 L volumetric flask, dilute 60 mL Regular Clorox Bleach [5.25% sodium hypochlorite (NaClO), The Clorox Company, Oakland, CA] to the mark with water. Invert three times to mix. De-gas with helium. Refrigerate.

6.6 Preparation of Standards

6.6.1 Stock 1000 mg-N(as NH_3)/L Calibration Standard: In a 1 L volumetric flask, dissolve 3.819 g of ammonium chloride (NH_4Cl), dried for one hour at 105°C, in about 500 mL water. Add 1 mL concentrated H_2SO_4 and dilute to the mark.

6.6.2 Intermediate 100 mg-N(as NH_3)/L Calibration Standard: In a 1 L volumetric flask, dilute 100.0 mL of stock calibration standard (6.6.1) in about 500 mL water. Add 1 mL H_2SO_4 and dilute to the mark with water. This solution is also the spike solution.

6.6.3 Working Calibration Standards: Prepare standards over the range of analysis. For the working range of 0-2.00 mg-N(as NH_3)/L, the following standards may be used:

mL Intermediate Standard (6.6.2) diluted to 1L	Concentration mg/L
0.0	0.00
0.2	0.02
2.5	0.25
5.0	0.50
7.5	0.75
10.0	1.00
20.0	2.00

Note: Use volumetric flasks and preserve the working standards by addition of 1 mL of concentrated H_2SO_4.

6.6.4 Stock 100 mg/L Ammonia Control Standard: (Any ammonia compound may be used for the control standards. They should be prepared by someone other than the analyst.) In a 1 L volumetric flask, dissolve 0.4716848 g of ammonia sulfate [$(NH_4)_2SO_4$], dried at 105°C for one hour, in about 500 mL water. Add 1 mL of concentrated H_2SO_4 and dilute to the mark.

6.6.5 Working Control Standards: The following concentrations are typical:

	mL Stock Control Standard (6.6.4) diluted to 1 L	Concentration mg/L
CS-1	2.0	0.20
CS-2	6.0	0.60

Note: Use volumetric flasks. Preserve the control standards by addition of 1 mL of concentrated H_2SO_4.

7.0 Procedure

7.1 Allow at least 15 minutes for the heating block to warm up to 60°C before beginning the analysis.

7.2 Follow the Lachat Procedural SOP (Typical Daily Operation Section) for the remainder of the analysis.

7.3 This method can be run simultaneously with the Nitrate/Nitrite method. Combined standards should then be prepared.

8.0 Calculations

The computer yields results directly in mg-N(as NH_3)/L.

9.0 Quality Control

9.1 The minimum acceptable correlation coefficient (r) = 0.995.

9.2 The following items are required with the minimum frequency indicated:

Audit	Type	Frequency	Limits
CS-1	Method	Beg, End, 1/40 Samp.	0.60 ± 0.04
CS-2	Method	Beg, End, 1/40 Samp.	0.20 ± 0.03
Reagent Blank	Method	Beg, End, 1/40 Samp.	0.00 ± MDL
Lab Blank*	Method	Beg, End, 1/40 Samp.	0.00 ± MDL
Duplicate*	Method	1/40 Sample	$\triangle \leq 0.02$
Spike*	Method	1/40 Sample	100 ± 12

*These audits are not included in Lake Water Analysis.

10.0 Waste Disposal

Effluent from this channel is basic. It should be disposed of in a blue labeled waste container.

11.0 Preventive Maintenance

Required maintenance is described in the Lachat Procedural SOP.

12.0 Troubleshooting

It is very important to thoroughly purge all reagents of air before they are used. Insufficient purging will result in a noisy baseline and air spikes in the peaks.

13.0 References

13.1 Lachat Instruments, Method Number 10-107-06-2-C, Ammonia in surface water, wastewater. Revision Date, August 1992.

13.2 Lachat QuikChem Operating Manual.

13.3 GLAS Standard Operating Procedure, Ammonia Nitrogen, February 1993.

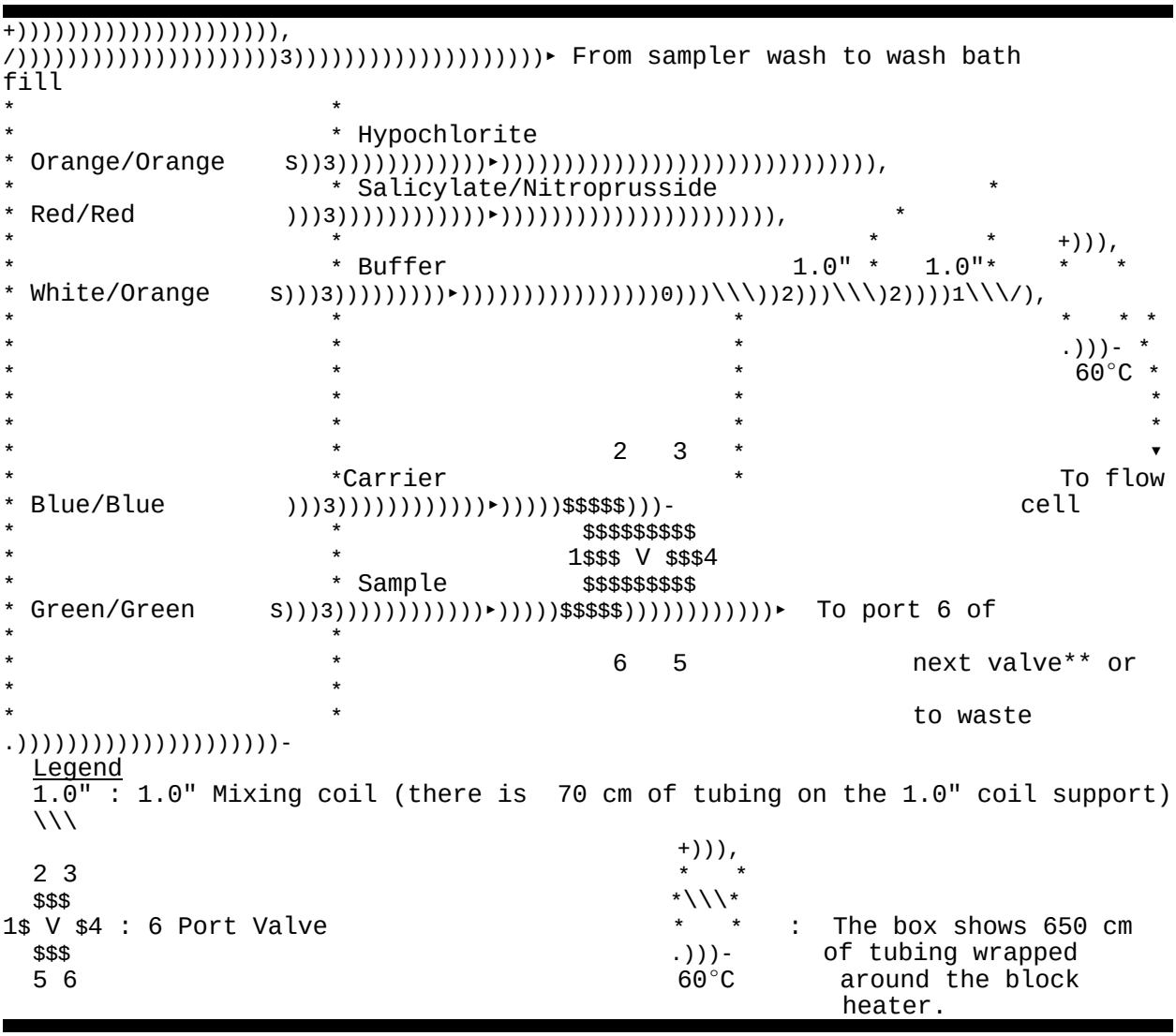

Figure 1. Ammonia Analytical Manifold

Comments:

1. Filter used is 660 nm.
2. Sample loop length is 25 cm.
3. All manifold tubing is 0.8 mm (0.032") ID. This relates to a flow of 5.2 µL/cm.
4. The Carrier is helium degassed DI Water.
5. Timing: Cycle period is 40 seconds. Inject to start of peak is 26 seconds.

** If more than one channel is being used.