QuikChem® Method 10-107-06-1-B

DETERMINATION OF AMMONIA (PHENOLATE) BY FLOW INJECTION ANALYSIS COLORIMETRY

Written by Lachat

Applications Group

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QuikChem® Method 10-107-06-1-B

Ammonia (Phenolate) in Potable and Surface Waters

0.05 to 5.00 mg N as NH_3/L

– Principle –

This method is based on the Berthelot reaction. Ammonia reacts with alkaline phenol, then with sodium hypochlorite to form indophenol blue. Sodium nitroprusside (nitroferricyanide) is added to enhance sensitivity. The absorbance of the reaction product is measured at 630 nm, and is directly proportional to the original ammonia concentration in the sample.

– Interferences –

1. Calcium and magnesium ions may precipitate if present in sufficient concentration. EDTA is added to the sample in-line in order to prevent this problems.
2. Color, turbidity and certain organic species may interfere. Turbidity is removed by manual filtration. Sample color may be corrected for by running the samples through the manifold without color formation.
3. Eliminate any marked variation in acidity or alkalinity among samples because intensity of measured color is pH-dependent. Likewise, ensure that the pH of standard ammonia solutions approximates that of sample.

– Special Apparatus –

Please see Parts and Price list for Ordering Information

1. Heating Unit Lachat Part No. A85X00 (X=1 for 110V, X=2 for 220V)
2. PVC PUMP TUBES MUST BE USED FOR THIS METHOD

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QuikChem® Method 10-107-06-1-B

DETERMINATION OF AMMONIA (PHENOLATE) BY FLOW INJECTION ANALYSIS COLORMETRY

1. SCOPE AND APPLICATION

1.1. This method covers the determination of ammonia (phenolate) in potable and surface waters.

1.2. The method is based on reactions that are specific for the ammonium (NH_4) ion.

1.3. The applicable range is 0.05 to 5.00 mg N as NH_3/L. The method detection limit is 0.007 mg N as NH_3/L. The method throughput is 60 injections per hour.

2. SUMMARY OF METHOD

2.1. This method is based on the Berthelot reaction. Ammonia reacts with alkaline phenol, then with sodium hypochlorite to form indophenol blue. Sodium nitroprusside (nitroferricyanide) is added to enhance sensitivity. The absorbance of the reaction product is measured at 630 nm, and is directly proportional to the original ammonia concentration in the sample.

3. DEFINITIONS

The definitions and purposes below are specific to this method, but have been conformed to common usage as much as possible.

3.1. ANALYTICAL BATCH -- The set of samples extracted/distilled/or digested at the same time to a maximum of 10 samples.

3.2. CALIBRATION BLANK (CB) -- A volume of reagent water in the same matrix as the calibration standards, but without the analyte.

3.3. CALIBRATION STANDARD (CAL) -- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

3.4. FIELD BLANK (FMB) -- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, including exposure to a sample bottle holding time, preservatives, and all preanalysis treatments. The purpose is to determine if the field or sample transporting procedures and environments have contaminated the sample.

3.5. FIELD DUPLICATE (FD) -- Two samples taken at the same time and place under identical circumstances which are treated identically throughout field and laboratory procedures. Analysis of field duplicates indicates the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.

3.6. LABORATORY BLANK (LRB) -- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, except that it is not taken to the sampling site. The purpose is to determine if the if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

3.7. LABORATORY CONTROL STANDARD (LCS) -- A solution prepared in the laboratory by dissolving a known amount of one or more pure compounds in a known amount of reagent water. It's purpose is to assure that the results produced by the laboratory remain within the acceptable limits for precision and accuracy. (This should not be confused with a calibrating standard).

3.8. LABORATORY DUPLICATE (LD) -- Two aliquots of the same environmental sample treated identically throughout a laboratory analytical procedure. Analysis of laboratory duplicates indicates precision associated with laboratory procedures but not with sample collection, preservation, or storage procedures.

3.9. QUALITY CONTROL CHECK SAMPLE (QCS) -- A sample containing analytes of interest at known concentrations (true values) of analytes. The QCS is obtained for a source external to the laboratory or is prepared from standards obtained from a different source than the calibration standards. The purpose is to check laboratory performance using test materials that have been prepared independently from the normal preparation process.

3.10. METHOD DETECTION LIMIT (MDL) -- The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero.

4. INTERFERENCES

4.1. Calcium and magnesium ions may precipitate if present in sufficient concentration. Tartrate or EDTA is added to the sample in-line in order to prevent this problems.

4.2. Color, turbidity and certain organic species may interfere. Turbidity is removed by manual filtration. Sample color may be corrected for by running the samples through the manifold without color formation.

4.3. Eliminate any marked variation in acidity or alkalinity among samples because intensity of measured color is pH-dependent. Likewise, ensure that the pH of standard ammonia solutions approximates that of sample.

5. SAFETY

5.1. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

5.2. Each laboratory is responsible for maintaining a current awareness file of the Occupational Health and Safety Act (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

5.3. The following chemicals have the potential to be highly toxic or hazardous, for detailed explanation consult the MSDS.

5.3.1. Phenol

5.3.2. Sodium Nitroferricyanide

5.3.3. Sulfuric Acid

5.3.4. Sodium Hydroxide

6. EQUIPMENT AND SUPPLIES

6.1. Balance -- analytical, capable of accurately weighing to the nearest 0.0001 g.

6.2. Glassware -- Class A volumetric flasks and pipettes or plastic containers as required. Samples may be stored in plastic or glass.

6.3. Flow injection analysis equipment designed to deliver and react sample and reagents in the required order and ratios.

6.3.1. Sampler

6.3.2. Multichannel proportioning pump

6.3.3. Reaction unit or manifold

6.3.4. Colorimetric detector

6.3.5. Data system

6.4. Special Apparatus

6.4.1. Heating unit Lachat Part No. A85X00 (X=1 for 110V, X=2 for 220V)

6.4.2. PVC PUMP TUBES MUST BE USED FOR THIS METHOD

7. REAGENTS AND STANDARDS

7.1. PREPARATION OF REAGENTS

Use ASTM Type II water for all solutions. (See Standard Specification for Reagent Water D1193-77 for more information).

Degassing with helium:

To prevent bubble formation, degas all solutions except the standards with helium. Use He at 140kPa (20 lb/in^2) through a helium degassing tube (Lachat Part No. 50100.) Bubble He through the solution for one minute.

Reagent 1. Sodium Phenolate

CAUTION: Wear gloves. Phenol causes severe burns and is rapidly absorbed into the body through the skin.

By Volume: In a **1 L volumetric flask** dissolve **88 mL** of **88% liquified phenol** or **83 g crystalline phenol** (C_6H_5OH) in approximately **600 mL DI water**. While stirring, slowly add **32 g sodium hydroxide** (NaOH). Cool, dilute to the mark, and invert to mix. Do not degas this reagent. Prepare fresh every 3 to 5 days. Discard when reagent turns dark brown.

By Weight: To a tared **1 L container**, add **888 g DI water.** Add **94.2 g** of **88% liquified phenol** or **83 g crystalline phenol** (C_6H_5OH). While stirring, slowly add **32 g sodium hydroxide** (NaOH). Cool and invert to mix thoroughly. Do not degas this reagent. Prepare fresh every 3 to 5 days. Discard when reagent turns dark brown.

Reagent 2. Sodium Hypochlorite

By Volume: In a **500 mL volumetric flask** dilute **250 mL 5.25% sodium hypochlorite** (NaOCl), to the mark with **DI water**. Invert to mix. Prepare fresh daily

By Weight: To a tared **500 mL container** add **250 g 5.25% sodium hypochlorite** (NaOCl) and **250 g DI water**. Stir or shake to mix. Prepare fresh daily

Reagent 3. Buffer

By Volume: In a **1 L volumetric flask**, dissolve **50.0 g disodium ethylenediamine tetraacetate dihydrate** ($Na_2EDTA{\cdot}2H_20$) and **9.0 g sodium hydroxide** (NaOH) in approximately **900 mL DI water.** Dilute to the mark and mix with a magnetic stirrer until dissolved. Prepare fresh monthly

By Weight: To a **1 L tared container** add **50.0 g disodium ethylenediamine tetraacetate dihydrate** ($Na_2EDTA{\cdot}2H_20$) and **9.0 g sodium hydroxide** (NaOH). Add **965 g DI water**. Mix with a magnetic stirrer until dissolved. Prepare fresh monthly

Reagent 4. Sodium Nitroprusside

By Volume: To a **1 L volumetric flask** dissolve **3.50 g sodium nitroprusside** (Sodium Nitroferricyanide [$Na_2Fe(CN)_5NO{\cdot}2H_2O$]). Dilute to the mark with **DI water** and invert to mix. Prepare fresh every 1 to 2 weeks

By Weight: To a **tared 1 L container** add **3.50 g sodium nitroprusside** (Sodium Nitroferricyanide [$Na_2Fe(CN)_5NO{\cdot}2H_2O$]) and **1000 g DI water**. Invert to mix. Prepare fresh every 1 to 2 weeks.

Reagent 5. Carrier and Diluent (0.20 % Sulfuric Acid)

By Volume: To a **1 L volumetric flask**, add approximately **900 ml DI water**. Then add **2 mL concentrated sulfuric acid** (H_2SO_4). Dilute to the mark with **DI water** and invert to mix.

By Weight: To a **tared 1 L container**, add **998 g DI water**. Then add **3.68 g concentrated sulfuric acid** (H_2SO_4). Invert to mix.

7.2. PREPARATION OF STANDARDS

To prepare the stock and working standards, the following containers will be requires:

By Volume: Two 1 L and six 250 mL volumetric flasks.

By Weight: Two 1 L, and six 250 mL containers.

Standard 1. Stock Standard 1000 mg N as NH_3/L

In a **1 L volumetric flask** dissolve **3.819 g ammonium chloride** (NH_4Cl) that has been dried for two hours at 110°C in about **800 mL DI water.** Dilute to the mark and invert to mix.

Standard 2. Intermediate Stock Standard 12.5 mg N as NH_3/L in 0.20 % H_2SO_4

By Volume: To a **1 L volumetric flask**, add **12.50 mL Stock Standard** (Standard 1) to approximately **900 ml DI water**. Then add **2 mL concentrated sulfuric acid** (H_2SO_4.) Dilute to the mark with **DI water**. Invert to mix.

By Weight: To a **tared 1 L container** add about **12.5 g Stock Standard** (Standard 1). Measure the exact weight of the solution added and divide this weight by **0.0125**. This

will give you the total weight of the diluted solution to be made. Make up the solution to this weight with **0.20 % sulfuric acid** (**Reagent 5**.) Shake before using.

Working Standards (Prepare weekly)	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Concentration mg N as NH_3/L	5.00	2.50	1.25	0.50	0.10	0.05	0.00
Concentration mg NH_3/L	6.05	2.07	1.51	0.61	0.12	0.61	0.00
By Volume							
Volume (mL) of **stock standard 2** diluted to 500 mL with Reagent 5	200	--	--	--	--	--	---
Volume (mL) of **standard A** diluted to 250 mL with Reagent 5	--	125	62.5	25	5	2.5	--
By Weight							
Weight (g) of **stock standard 2** diluted to final weight (~500 g) divided by **factor** below with Reagent 5.	200	--	--	--	--	--	--
Weight (g) of **standard A** diluted to final weight (~250 g) divided by **factor** below with Reagent 5.		125	62.5	25	5	2.5	
Division Factor Divide exact weight of the standard by this **factor** to give final weight	0.4	0.5	0.25	0.1	0.02	0.01	---

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples should be preserved to pH <2 with sulfuric acid upon collection. The maximum holding time is 28 days.

8.2 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.

8.3. The Federal Register entry which defines standard EPA NPDES and NPDWR methods states that "Manual Distillation is NOT required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies". (see SOURCES). This suggests that the user perform recovery studies on representative sample types, studies which show that the undistilled samples give the same recoveries as the manually distilled samples. Follow EPA Method 350.2 for the complete distillation procedure.

9. QUALITY CONTROL

9.1. Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing check on performance. The

laboratory is required to maintain performance records that define the quality of the data that are generated.

9.1.1. Analyses of matrix spike and matrix spike duplicate samples are required to demonstrate method accuracy and precision and to monitor matrix interferences (interferences caused by the sample matrix). The procedure and QC criteria for spiking are described in section 9.3.

9.1.2. Analyses of laboratory blanks are required to demonstrate freedom from contamination.

9.1.3. The laboratory shall, on an ongoing basis, demonstrate through calibration verification and analysis of the ongoing precision and recovery sample that the analysis system is in control.

9.1.4. The laboratory should maintain records to define the quality of data that is generated.

9.2. INITIAL DEMONSTRATION OF PERFORMANCE

9.2.1. Method Detection Limit (MDL) --To establish the ability to detect the analyte, the analyst shall determine the MDL per the procedure in 40 CFR 136, Appendix B using the apparatus, reagents, and standards, that will be used in the practice of this method. An MDL less than or equal to the MDL in section 1.2 must be achieved prior to the practice of this method.

9.2.2. Initial Precision and Recovery -- To establish the ability to generate acceptable precision results, the operator shall perform 10 replicates of a mid-range standard, according to the procedure beginning in Section 11.

9.2.2.1. Using the results of the replicates compute the average percent recovery (X) and the standard deviation (s) for the analyte. Use the following equation for the calculation of the standard deviation.

$$s = \sqrt{\frac{\sum x^2 - \frac{\left(\sum x\right)^2}{n}}{n-1}}$$

Where, n = Number of samples, x = concentration in each sample

9.2.2.2. Compare s and x results with the corresponding data in Section 17. If the results meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If however, s and x do not match the data in Section 17, system performance is unacceptable. In this event correct the problem, and repeat the test.

9.3. Matrix spikes- The laboratory must spike, in duplicate, a minimum of 10 percent of all samples (one sample in each batch of ten samples) from a given sampling site or if for compliance monitoring, from a given discharge. The two sample aliquots shall be spiked with the stock standard (section 7.2).

9.3.1. The concentration of the spike in the sample shall be determined as follows:

9.3.1.1. If, as in compliance monitoring, the concentration of the analyte in the sample is being checked against a regulatory concentration limit, the

spiking level shall be at that limit or at 1 to 5 times higher than the background concentration of the sample (determined in Section 9.3.2), which ever is higher.

9.3.1.2. If the concentration of the analyte in a sample is not being checked against a limit, the spike shall be at the concentration of the precision and recovery standard used in Section 9.2.5 or at 1 to 5 times higher than the background concentration, whichever concentration is higher.

9.3.2. Analyze one sample aliquot out of each set of ten samples from each site or discharge according to the procedure beginning in Section 11 to determine the background concentration of (B) of the analyte.

9.3.2.1. If necessary, prepare a standard solution appropriate to produce a level in the sample at the regulatory compliance limit or at 1 to 5 times the background concentration (per Section 9.3.1).

9.3.2.2. Spike two additional sample aliquots with the spiking solution and analyze these aliquots to determine the concentration after spiking (A)

9.3.3. Calculate the percent recovery (P) of the analyte in each aliquot using the following equation.

$$P = \frac{(A - B)100}{T}$$

Where, A = Measured concentration of analyte after spiking, B = measured background concentration of analyte, T = True concentration of the spike

9.3.4. The percent recovery of the analyte should meet current laboratory acceptance criteria.

9.3.4.1. If the results of the spike fail the acceptance criteria, and the recovery of the QC standard in the ongoing precision and recovery test of the analytical batch is within the current laboratory acceptance criteria, an interference is present. In this case, the results may not be reported for regulatory compliance purposes and the analyst must assess the potential cause for the interference. If the interference is attributable to sampling, the site or discharge should be resampled. If the interference is attributable to a method deficiency, the analyst must modify the method, repeat the test required in Section 9.1.2 and repeat the analysis of the sample and the matrix spike.

9.3.4.2. If the results of both the spike and ongoing precision and recovery test fail the acceptance criteria, the analytical system is judged to be out of control, and the problem shall be identified and corrected, and the sample reanalyzed.

9.3.5. Compute the relative percent difference (RPD) between two sample results using the following equation:

$$RPD = \frac{(D_1 - D_2)}{(D_1 + D_2)/2} \text{ x } 100$$

Where, D1 = Concentration of analyte in the sample, D2 = Concentration of analyte in the second (duplicate) sample.

9.3.6. The RPD for duplicates shall meet the current laboratory acceptance criteria. If the criteria are not met, the analytical system is judged to be out of control, and the problem must be immediately identified and corrected and the analytical batch reanalyzed.

9.4 Laboratory blanks - Laboratory reagent water blanks are analyzed to demonstrate freedom from contamination.

9.4.1. Analyze a laboratory reagent water blank initially (with the test in Section 9.2) and with each analytical batch. The blank must be subjected to the same procedural steps as a sample.

9.4.2. If analyte is detected in the blank at a concentration greater than the Minimum Level (Section 1.6), analysis of the samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination. All samples must be associated with an uncontaminated method blank before the results may be reported for regulatory compliance purposes.

9.5. Calibration Verification Verify calibration using the procedure described in Section 10

9.6. On-going Precision and Recovery (OPR) - With every analytical batch, a midrange standard must be prepared using the procedure described in Section 11.

9.6.1. Compare the results with the current laboratory acceptance criteria. If the criteria are not met, the analytical system is judged to be out of control, and the problem must be immediately identified and corrected and the analytical batch reanalyzed.

9.7. Quality Control Samples (QCS) It is suggested that the laboratory obtain and/or prepare a quality control sample using a source different from the source routinely used in section 7. The QCS is used to verify the concentrations of the calibration standards.

9.8. Depending on the specific program requirements, field replicates and field spikes of the analytes of interest into samples may be required to assess the precision and accuracy of the sampling and sample transporting techniques.

10. CALIBRATION AND STANDARDIZATION

10.1. Prepare reagents and standards as described in Section 7.

10.2. Set up manifold as shown in Section 17.

10.3. Input data system parameters as shown in Section 17.

10.4. Pump DI water through all reagent lines and check for leaks and smooth flow. Switch to reagents and allow the system to equilibrate until a stable baseline is achieved.

10.5. Place standards in the sampler. Input the information required by the data system.

10.6. Calibrate the instrument by injecting the standards. The data system will then associate the concentrations with the peak area for each standard to determine the calibration curve.

10.7. Verify calibration using a midrange calibration standard every ten samples or every analytical batch. Compute the percent recovery using the following equation:

$$\%recovery = \frac{D}{K} \text{ x } 100$$

Where, D = Determined concentration of analyte in the calibration standard, K = Actual concentration of the analyte in the calibration standard

10.8. If % recovery exceeds +/-10%, the analytical system is judged to be out of control, and the problem must be immediately identified and corrected and the analytical batch reanalyzed

11. PROCEDURE

11.1. CALIBRATION PROCEDURE

11.1.1. Prepare reagent and standards as described in Section 7.

11.1.2. Set up manifold as shown in Section 17.

11.1.3. Input data system parameters as in Section 17.

11.1.4. Pump DI water through all reagent lines and check for leaks and smooth flow. Switch to reagents and allow the system to equilibrate until a stable baseline is achieved.

11.1.5. Place samples and/or standards in the autosampler. Input the information required by the data system, such as concentration, replicates and QC scheme. (See Section 17)

11.1.6. Calibrate the instrument by injecting the standards. The data system will then associate the concentrations with the instrument responses for each standard.

11.2. SYSTEM NOTES

11.2.1. If samples are preserved and determined without distillation the level of preservation acid is critical. See section 17, page 20.

11.2.2. For information on system maintenance, and troubleshooting refer to the Troubleshooting Guide in the System Operation Manual. This guide is also available on request from Lachat.

11.2.3. Allow 15 min for heating unit to warm up to 60°C.

11.2.4. If baseline drifts, peaks are too wide, or other problems with precision arise, clean the manifold by the following procedure.

11.2.4.1. Place all reagent lines in deionized water and pump to clear reagents (2 to 5 min).

11.2.4.2. Place all reagent lines in 1 M hydrochloric acid (1 volume concentrated HCl added to 11 volumes of deionized water) and pump for several minutes.

11.2.4.3. Place all reagent lines in deionized water and pump until the HCl is thoroughly washed out.

11.2.4.4. Resume pumping reagents.

11.2.4.5. Use consumer bleaches with caution. Proprietary additives may contribute to staining of tubing and data quality.

11.2.4.6. Add reagents in the order that they appear on the manifold to reduce staining.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Calibration is done by injecting standards. The data system will then prepare a calibration curve by plotting response versus standard concentration. Sample concentration is calculated from the regression equation.

12.2. Report only those values that fall between the lowest and highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

12.3. Report results in mg N/L or mg NH_3–N /L.

13. METHOD PERFORMANCE

13.1. The method support data are presented in section 17. This data was generated according to a Lachat Work Instruction during development of the method.

13.2. Although Lachat Instrument publishes method performance data, including MDL, precision, accuracy and carryover studies, we cannot guarantee that each laboratory will be capable of meeting such performance. Individual laboratory and instrument conditions, as well as laboratory technique, play a major role in determining method performance. The support data serves as a guide of the potential method performance. Some labs may not be able to reach this level of performance for various reasons, while other labs may exceed it.

14. POLLUTION PREVENTION

14.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the United States Environmental Agency (USEPA) recommends recycling as the next best option.

14.2. The quantity of chemicals purchased should be based on expected usage during their shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

14.3. For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N. W., Washington D. C. 20036, (202) 872-4477.

15. WASTE MANAGEMENT

15.1. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods an bench operation. Compliance with all sewage discharge permits and regulations is also required.

15.2. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N. W., Washington D. C. 20036, (202) 872-4477.

16. REFERENCES

16.1. U.S. Environmental Protection Agency, **Methods for Chemical Analysis of Water and Wastes**, EPA-600/4-79-020, Revised March 1983, Method 350.1

16.2 U.S. Environmental Protection Agency, 40 CFR, Part 36 Table 1B, footnote 6, 1994.

17. TABLE, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

17.1. DATA SYSTEM PARAMETERS FOR QUIKCHEM 8000

The timing values listed below are approximate and will need to be optimized using graphical events programming.

Sample throughput:	60 samples/h, 60 s/sample
Pump Speed:	35
Cycle Period:	60

Analyte Data:

Concentration Units:	mg N as NH_3 / L
Peak Base Width:	29 s
% Width Tolerance:	100
Threshold:	10000
Inject to Peak Start:	30 s
Chemistry:	Direct

Calibration Data:

Level	1	2	3	4	5	6	7
Concentration mg N/L as NH_3	5.00	2.50	1.25	0.50	0.10	0.05	0.00

Calibration Rep Handling:	Average
Calibration Fit Type:	1st Order Polynomial
Weighting Method:	None
Force through zero:	No

Sampler Timing:

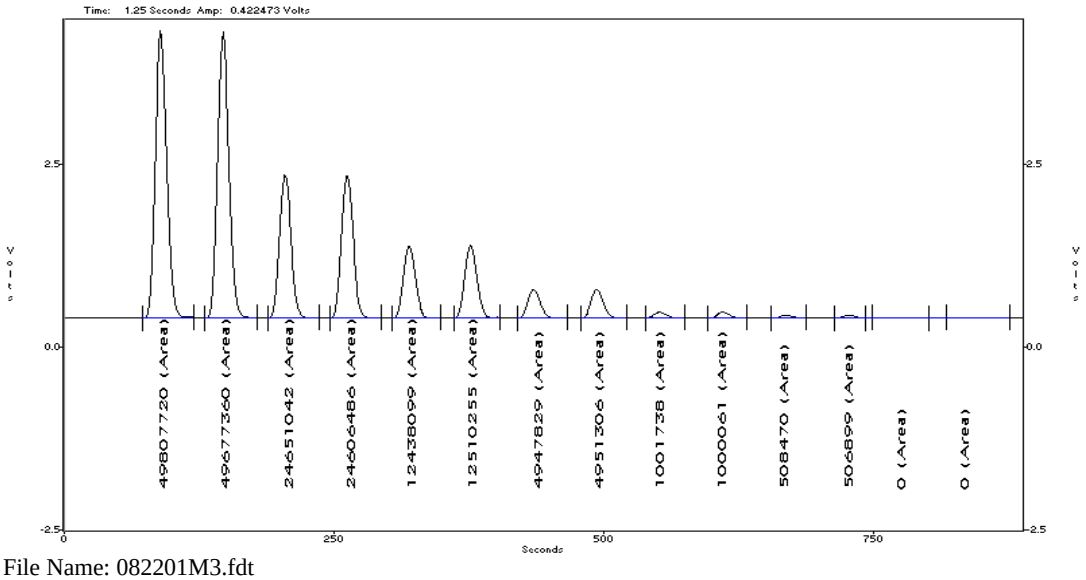
Min. Probe in Wash Period:	5.0 s
Probe in Sample Period:	24 s

Valve Timing:

Load Period:	15 s
Inject Period:	45 s

17.2. SUPPORT DATA FOR QUIKCHEM 8000

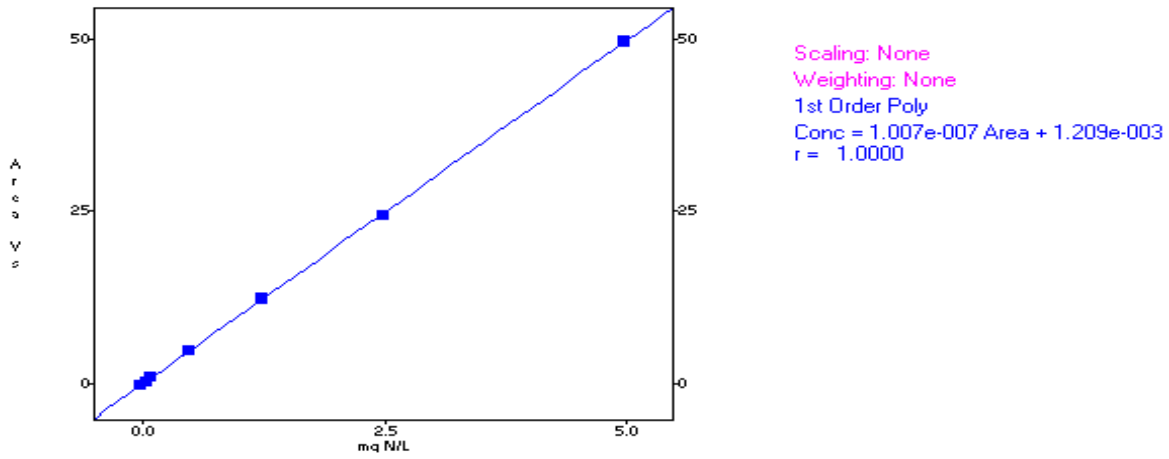
Calibration Data for Ammonia

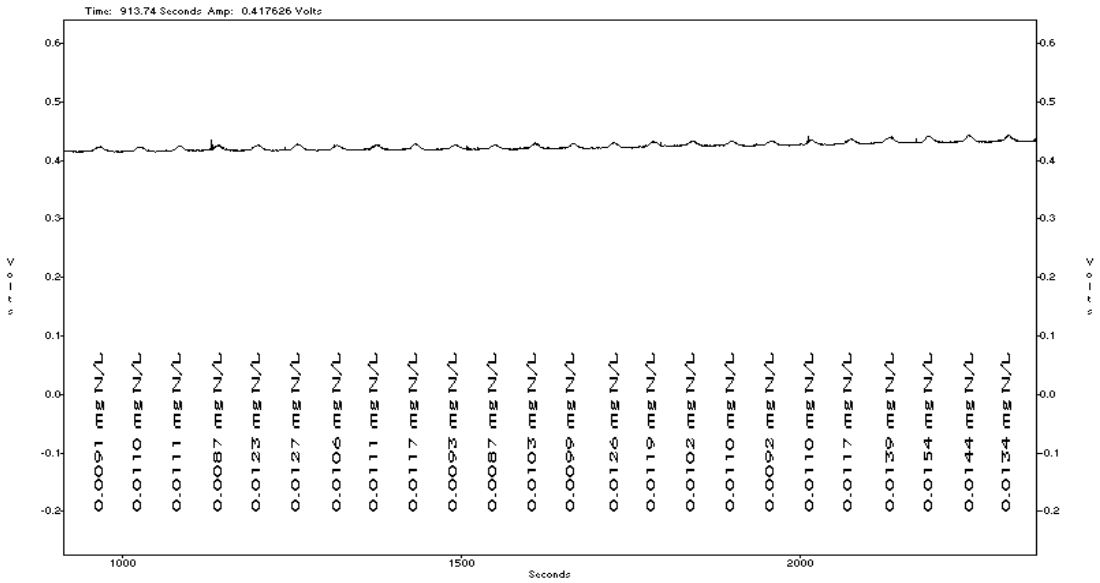


File Name: 082201M3.fdt
Acq. Date: 22 August 2001

Calibration Graph and Statistics

Level	Area	mg N/L	Determined	%RSD	%residual
1	49742540	5.00	5.01	0.2	-0.2
2	24628764	2.50	2.48	0.1	0.8
3	12474177	1.25	1.25	0.4	-0.5
4	4949568	0.50	0.49	0.0	0.1
5	1000900	0.10	0.10	0.1	-2.0
6	507685	0.05	0.05	0.2	-4.6
7	0	0.00	0.00	0.0	--



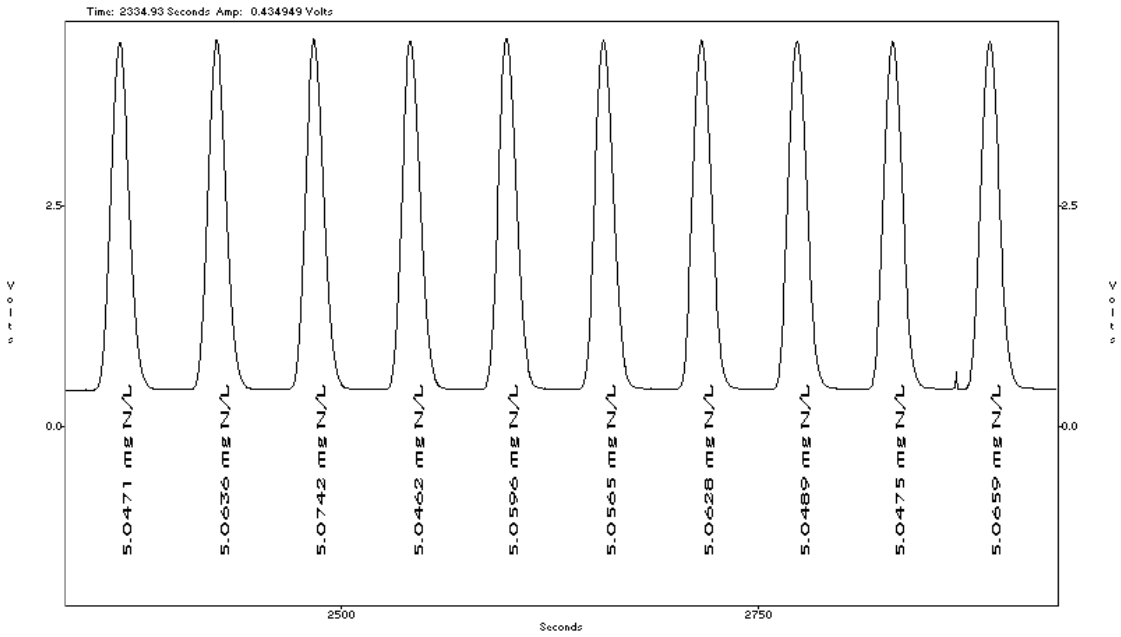


Method Detection Limit for Ammonia using a 0.01 mg N/L Standard

MDL = 0.007 mg N/L

Standard Deviation (s) = 0.003 mg N/L, Mean (X) = 0.011 mg N/L, Known Value = 0.01 mg N/L

ACQ. Date: 22 August 2001

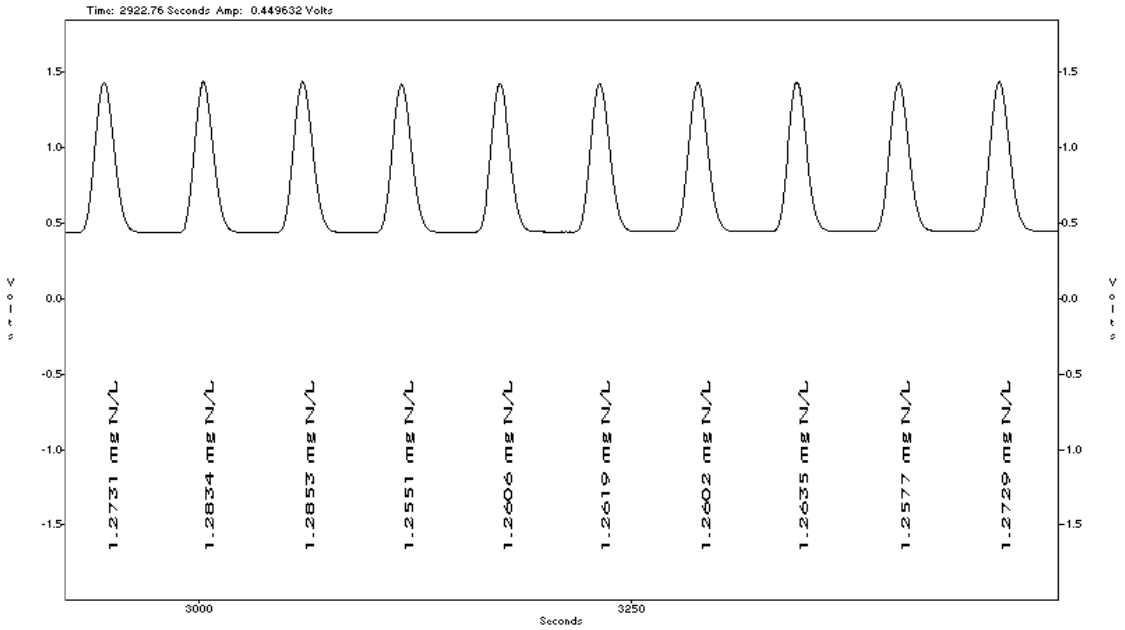


Precision data for Ammonia, using a 5.00 mg N/L Standard

%RSD = 0.26%

Standard Deviation (s) = 0.01 mg N/L, Mean (X) = 5.05 mg N/L, Known Value = 5.00 mg N/L, % residual = -1.13

ACQ. Date:22 August 2001

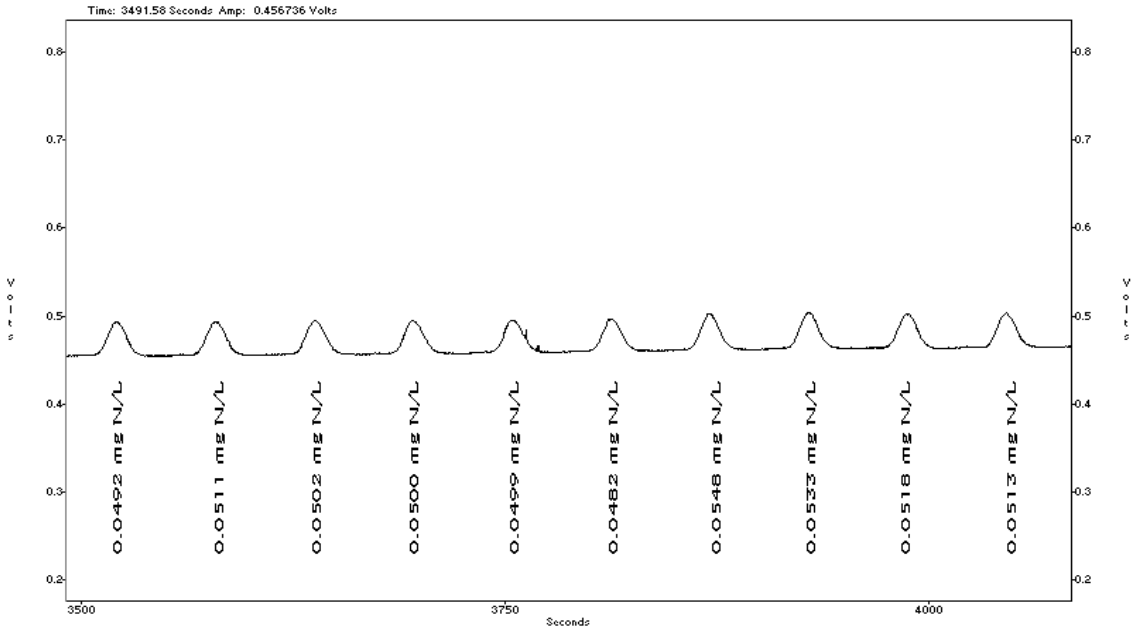


Precision data for Ammonia, using a 1.25 mg N/L Standard.

%RSD = 0.84%

Standard Deviation (s) = 0.0107 mg N/L, Mean (X) = 1.27 mg N/L, Known Value = 1.25 mg N/L, % residual = -1.84

ACQ. Date:22 August 2001

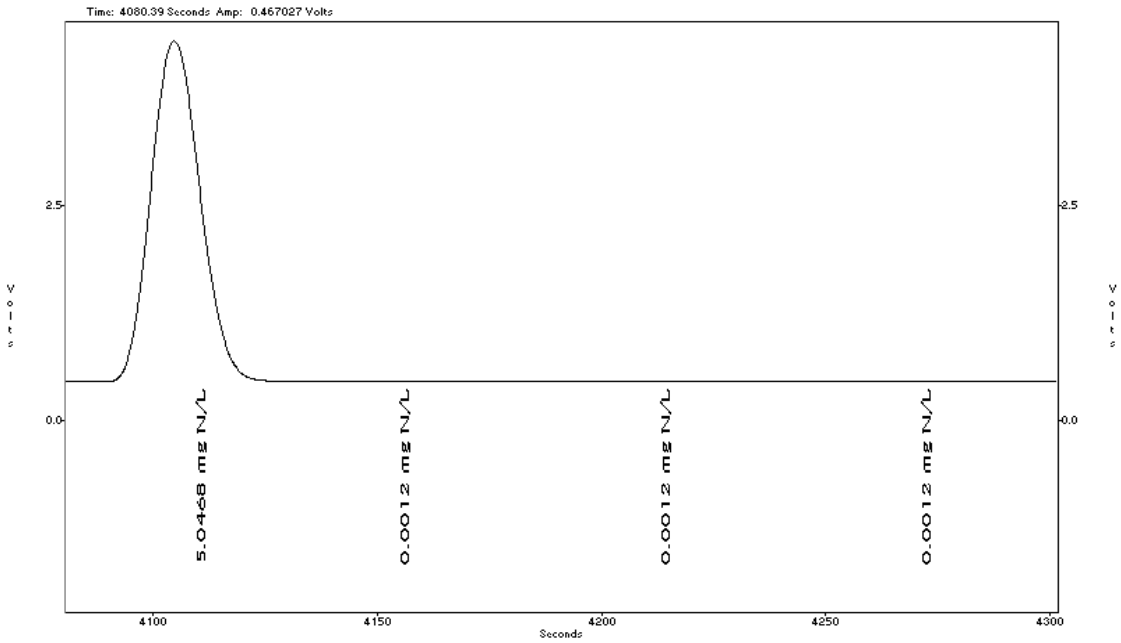


Precision data for Ammonia, using a 0.05 mg N/L Standard.

%RSD = 2.97%

Standard Deviation (s) = 0.001 mg N/L, Mean (X) = 0.050 mg N/L, Known Value = 0.05 mg N/L, % residual = -0.46

ACQ. Date:23 April 1997



Carryover Study: 5.00 mg/L Standard followed by 3 blanks

Carryover passed

ACQ. Date: 22 August 2001

APPENDIX A – OMNION 3.0 TIMING PARAMETERS AND SUPPORT DATA

DATA SYSTEM PARAMETERS FOR QUIKCHEM 8000/8500

The timing values listed below are approximate and will need to be optimized using graphical events programming.

Sample throughput:	72 samples/h, 50 s/sample
Pump Speed:	35
Cycle Period:	50

Analyte Data:

Concentration Units:	mg N as NH_3 / L
Inject to Peak Start:	20.5 s
Peak Base Width:	54.5 s
Chemistry:	Direct/Bipolar

Calibration Data:

Level	1	2	3	4	5	6	7
Concentration mg N/L as NH_3	5.00	2.50	1.25	0.50	0.10	0.05	0.00

Calibration Fit Type:	2^{nd} Order Polynomial
Weighting Method:	1/X
Force through zero:	No

Sampler Timing:

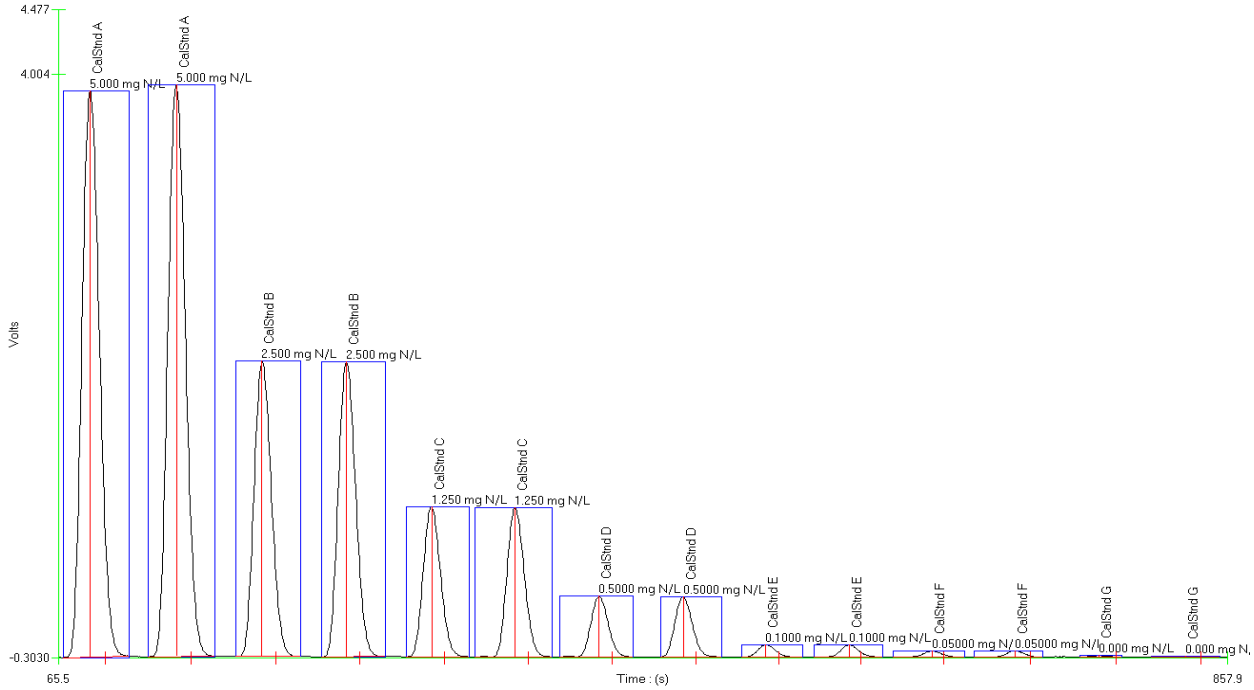
Min. Probe in Wash Period:	5.0 s
Sample Period:	20 s

Valve Timing:

Load Period:	15 s
Inject Period:	35 s

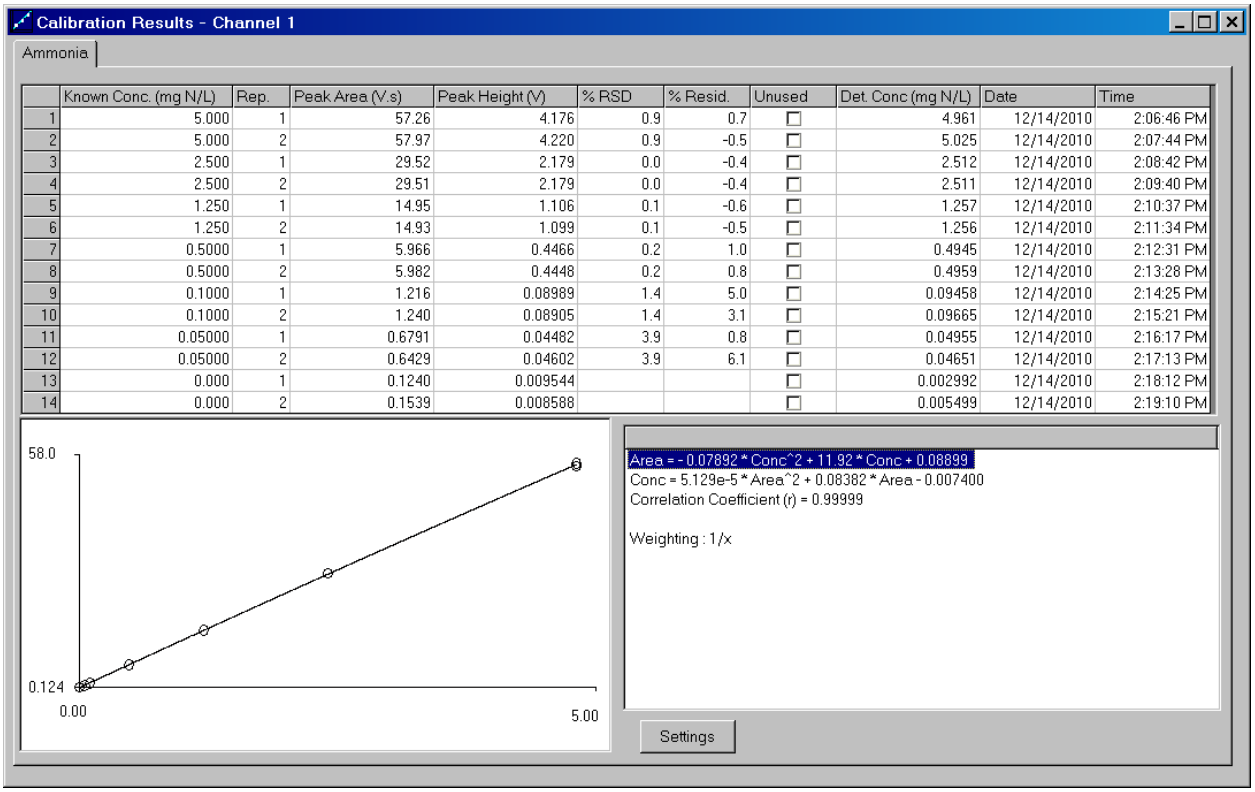
SUPPORT DATA FOR QUIKCHEM 8000/8500

Calibration Data for Ammonia

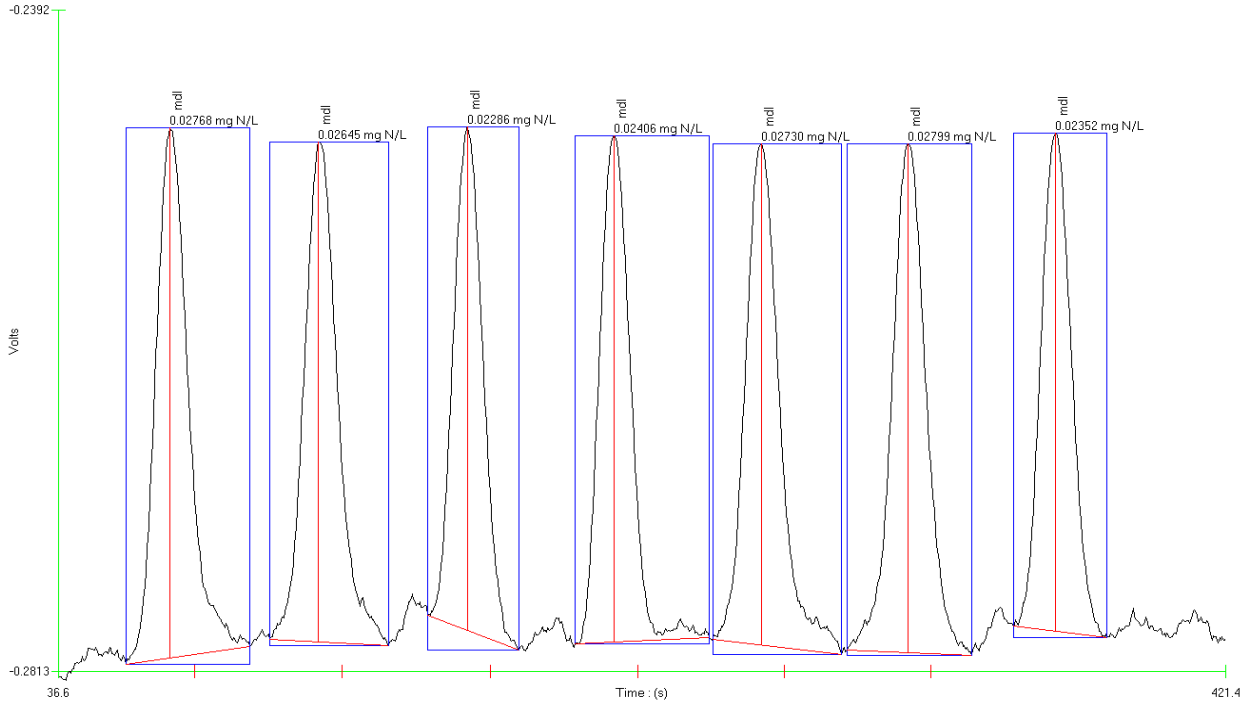


File Name: 12-14 cal prec
Acq. Date: 14 December 2010

Calibration Graph and Statistics



	Known Conc. (mg N/L)	Rep.	Peak Area (V.s)	Peak Height (V)	% RSD	% Resid.	Unused	Det. Conc (mg N/L)	Date	Time
1	5.000	1	57.26	4.176	0.9	0.7	☐	4.961	12/14/2010	2:06:46 PM
2	5.000	2	57.97	4.220	0.9	-0.5	☐	5.025	12/14/2010	2:07:44 PM
3	2.500	1	29.52	2.179	0.0	-0.4	☐	2.512	12/14/2010	2:08:42 PM
4	2.500	2	29.51	2.179	0.0	-0.4	☐	2.511	12/14/2010	2:09:40 PM
5	1.250	1	14.95	1.106	0.1	-0.6	☐	1.257	12/14/2010	2:10:37 PM
6	1.250	2	14.93	1.099	0.1	-0.5	☐	1.256	12/14/2010	2:11:34 PM
7	0.5000	1	5.966	0.4466	0.2	1.0	☐	0.4945	12/14/2010	2:12:31 PM
8	0.5000	2	5.982	0.4448	0.2	0.8	☐	0.4959	12/14/2010	2:13:28 PM
9	0.1000	1	1.216	0.08989	1.4	5.0	☐	0.09458	12/14/2010	2:14:25 PM
10	0.1000	2	1.240	0.08905	1.4	3.1	☐	0.09665	12/14/2010	2:15:21 PM
11	0.05000	1	0.6791	0.04482	3.9	0.8	☐	0.04955	12/14/2010	2:16:17 PM
12	0.05000	2	0.6429	0.04602	3.9	6.1	☐	0.04651	12/14/2010	2:17:13 PM
13	0.000	1	0.1240	0.009544			☐	0.002992	12/14/2010	2:18:12 PM
14	0.000	2	0.1539	0.008588			☐	0.005499	12/14/2010	2:19:10 PM



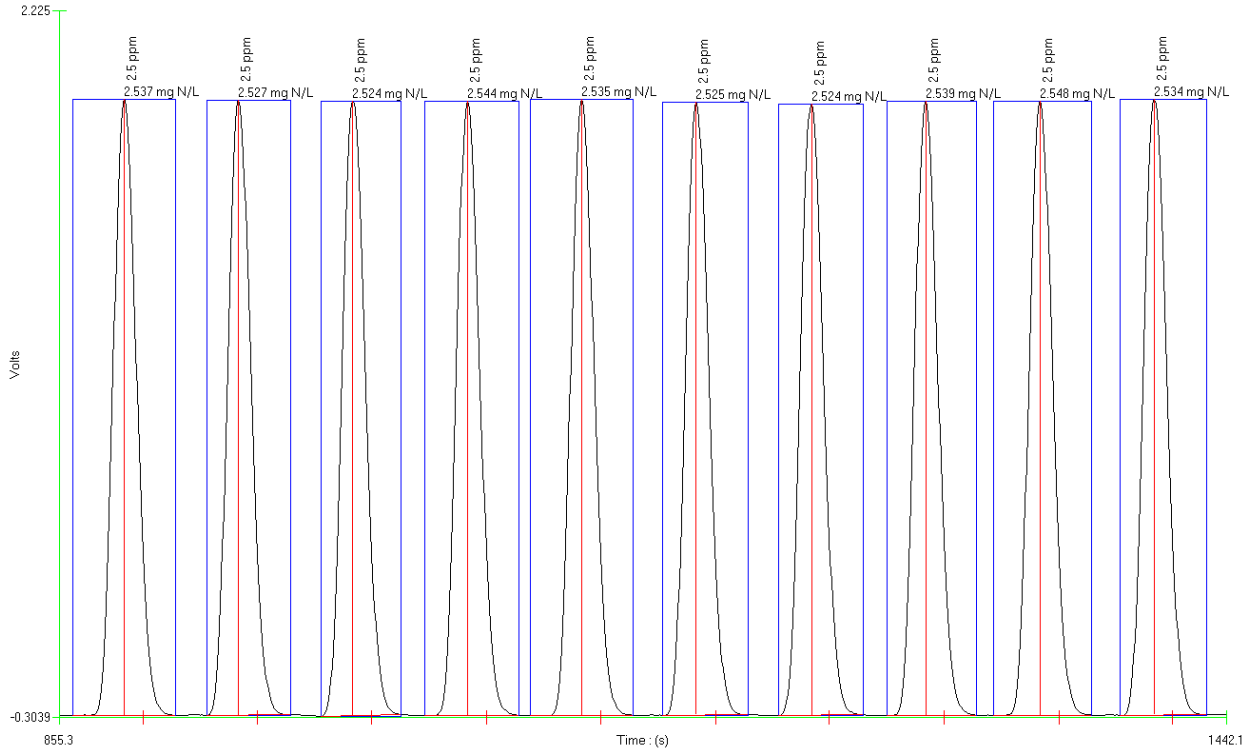
Method Detection Limit for Ammonia using a 0.025 mg N/L Standard

MDL = 0.007 mg N/L

Standard Deviation (s) = 0.002 mg N/L, Mean (X) = 0.0257 mg N/L, Known Value = 0.025 mg N/L

File Name: 12-14 mdl

Acq. Date: 14 December 2010



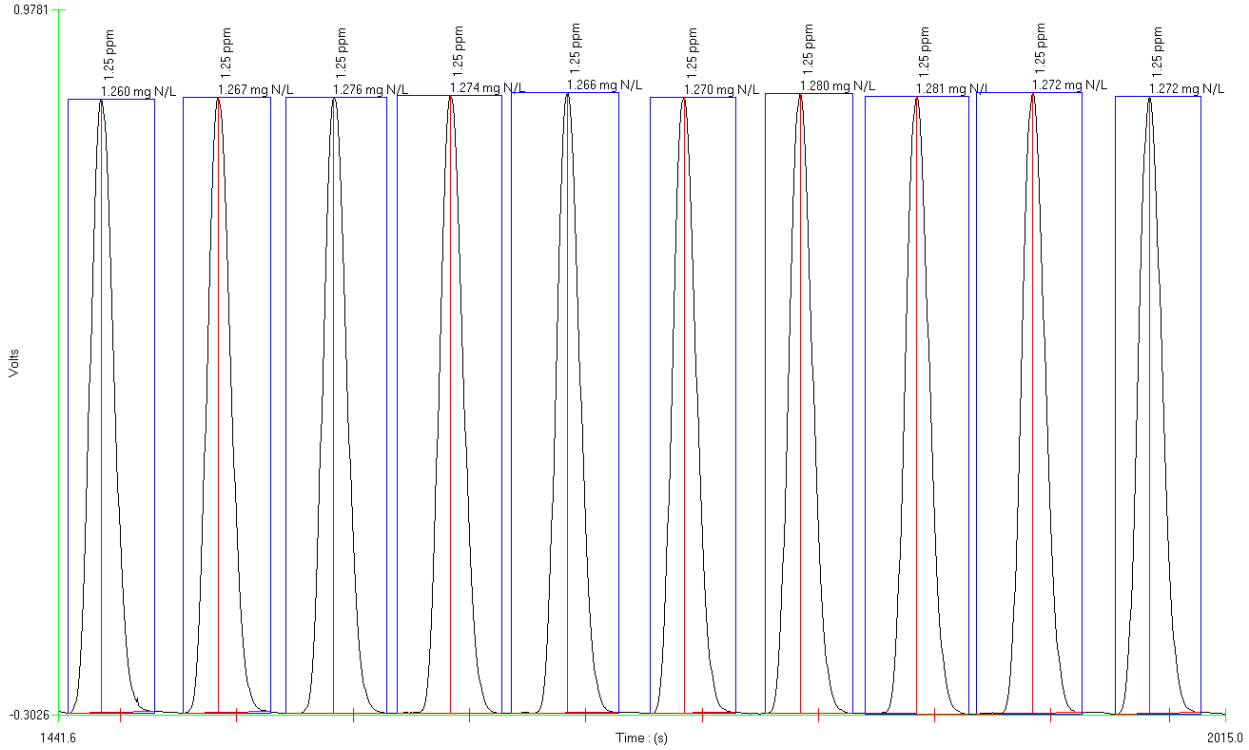
Precision data for Ammonia, using a 2.5 mg N/L Standard

%RSD = 0.34%

Standard Deviation (s) = 0.0086 mg N/L, Mean (X) = 2.53 mg N/L, Known Value = 2.5 mg N/L

File Name: 12-14 cal prec

Acq. Date: 14 December 2010



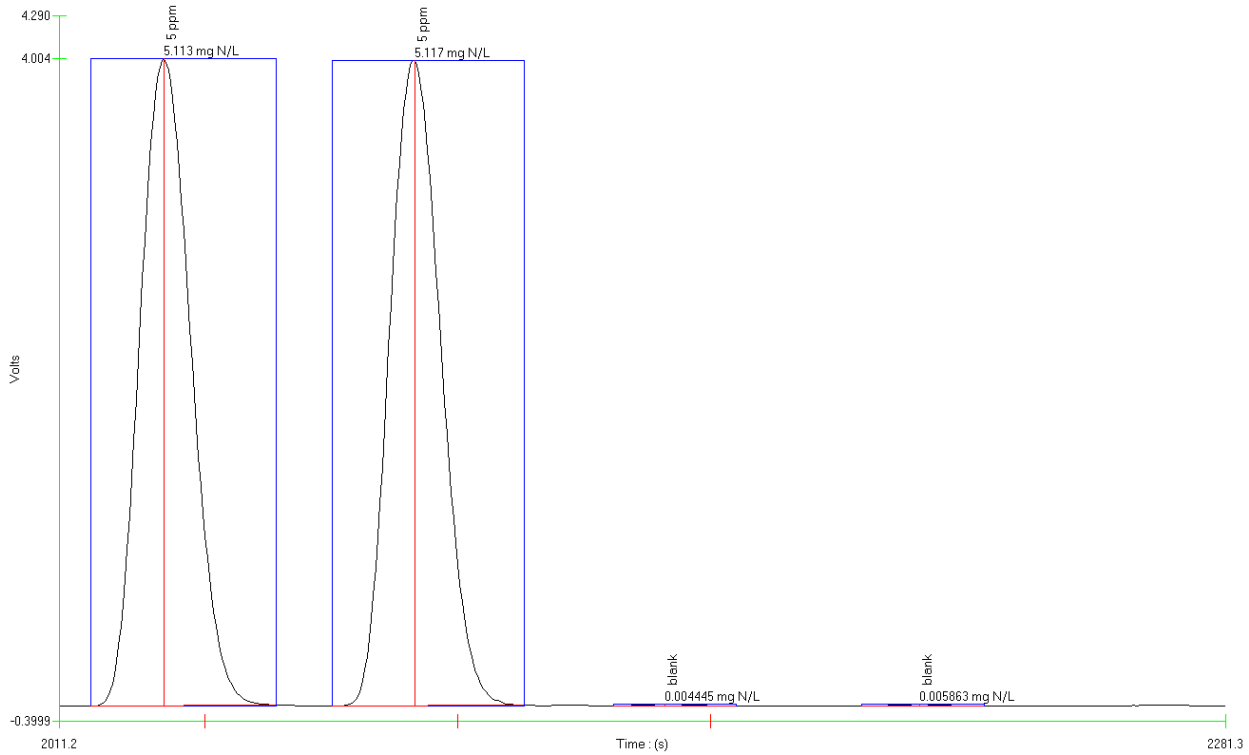
Precision data for Ammonia, using a 1.25 mg N/L Standard

%RSD = 0.51%

Standard Deviation (s) = 0.0064 mg N/L, Mean (X) = 1.27 mg N/L, Known Value = 1.25 mg N/L

File Name: 12-14 cal prec

Acq. Date: 14 December 2010



Carryover Study: 5.00 mg/L Standard followed by 3 blanks

Carryover passed

File Name: 12-14 cal prec

Acq. Date: 14 December 2010