QuikChem® Method 10-107-04-1-A

DETERMINATION OF NITRATE/NITRITE IN SURFACE AND WASTEWATERS BY FLOW INJECTION ANALYSIS

(LOW FLOW METHOD)

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Applications Group

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LACHAT INSTRUMENTS

QuikChem® Method 10-107-04-1-A

Determination of Nitrate/Nitrite in Surface and Wastewaters by Flow Injection Analysis

0.2 to 20 mg N/L as NO_3^- or NO_2^-

– Principle –

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 520 nm. Nitrite alone also can be determined by removing the cadmium column.

– Interferences –

1. Residual chlorine can interfere by oxidizing the reductor column.
2. 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.
3. Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. This interference is eliminated by pre-extracting the sample with an organic solvent.
4. Sample turbidity may interfere. Turbidity can be removed by filtration through a 0.45µm pore diameter membrane filter prior to analysis.

– Special Apparatus –

Please see Parts and Price list for Ordering Information

1. Cadmium-Copper Reduction Column (Lachat Part No. 50237)

Revised by A. Sardina 28 August 2000. **Lachat Instruments, Inc., 6645 West Mill Road, Milwaukee, WI 53218-1239 USA**. Phone: (414) 358-4200 FAX: (414) 358-4206.

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QuikChem® Method 10-107-04-1-A

DETERMINATION OF NITRATE/NITRITE IN SURFACE AND WASTEWATERS BY FLOW INJECTION ANALYSIS

1. SCOPE AND APPLICATION

1.1. This purpose of this method is to determine nitrate/nitrite in surface waters, and industrial wastewaters.

1.2. The applicable range is 0.2 to 20.0 mg N/L as NO_3^- or NO_2^-. The statistically determined detection limit as determined in water is 0.01 mg N/L. The method throughput is 55 injections per hour.

1.3. Each laboratory that uses this method must demonstration the ability to generate acceptable results using the procedure in section 11.

2. SUMMARY OF METHOD

2.1. 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 520 nm. Nitrite alone also can be determined by running without the cadmium column.

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 Residual chlorine can interfere by oxidizing the reductor column.

4.2 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.

4.3 Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. This interference is eliminated by pre-extracting the sample with an organic solvent.

4.4 Sample turbidity may interfere. Turbidity can be removed by filtration through a 0.45µm pore diameter membrane filter prior to analysis.

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 explanations consult the MSDS.

5.3.1. Cadmium granules

5.3.2. Ammonium hydroxide

5.3.3. Sodium hydroxide

5.3.4. Phosphoric acid

5.3.5. Sulfanilamide

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

6.3.6. 10 mm, 80 µL, glass flow cell

6.3.7. 520 nm interference filter

6.4. Special Apparatus

6.4.1. Cadmium-Copper Reduction Column (Lachat Part No. 50237).

7. REAGENTS AND STANDARDS

7.1. PREPARATION OF REAGENTS

Use deionized (10 megohm) 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. 15 N Sodium Hydroxide

By Volume: Add **150 g NaOH** very slowly to **250 mL or g of DI water**. **CAUTION**: The solution will get very hot! Swirl until dissolved. Cool and store in a plastic bottle.

Reagent 2. Ammonium Chloride buffer, pH 8.5

By Volume: In a **1 L** volumetric flask, dissolve **85.0 g ammonium chloride** (NH_4Cl) and **1.0 g disodium ethylenediamine tetraacetic acid dihydrate** ($Na_2EDTA{\cdot}2H_2O$) in about **800 mL DI water.** Dilute to the mark and invert to mix. Adjust the **pH to 8.5** with **15 N sodium hydroxide solution.**

By Weight: To a tared **1 L** container, add **85.0 g ammonium chloride** (NH_4Cl)**, 1.0 g disodium ethylenediamine tetraacetic acid dihydrate** ($Na_2EDTA{\cdot}2H_2O$) and **938 g DI water**. Shake or stir until dissolved. Then adjust the **pH to 8.5** with **15 N sodium hydroxide solution.**

ACS grade ammonium chloride has been found occasionally to contain significant nitrate contamination. An alternative recipe for the ammonium chloride buffer is:

By Volume: CAUTION: Fumes!!! In a hood, to a **1 L** volumetric flask, add **500 mL DI water, 105 mL concentrated hydrochloric acid** (HCl), **95 mL ammonium hydroxide** (NH_4OH), and **1.0 g disodium EDTA.** Dissolve and dilute to the mark. Invert to mix. Adjust the pH to 8.5 with **HCl** or **15 N NaOH solution.**

By Weight: CAUTION: Fumes!!! In a hood, to a tared **1 L** container, add **800 g DI water**, **126 g concentrated hydrochloric acid** (HCl), **85 g ammonium hydroxide** (NH_4OH) and **1.0 g disodium EDTA**. Stir until dissolved. Adjust the pH to 8.5 with **HCl** or **15 N NaOH**.

Reagent 3. Sulfanilamide color reagent

By Volume: In a **1 L** volumetric flask, add approximately **600 mL DI water**. Then add **100 mL 85% phosphoric acid** (H_3PO_4), **40.0 g sulfanilamide**, **and 1.0 g N-(1-naphthyl) ethylenediamine dihydrochloride** (NED). Shake to wet, and stir to dissolve for 30 minutes. Dilute to the mark, and invert to mix. Store in a dark bottle. This solution is stable for one month.

By Weight: To a **1 L** dark, tared container, add **876 g DI water**, **170 g 85% phosphoric acid** (H_3PO_4), **40.0 g sulfanilamide**, **and 1.0 g N-(1-naphthyl) ethylenediamine dihydrochloride** (NED). Shake until wet and stir with stir bar for 30 minutes until dissolved. This solution is stable for one month.

7.2. PREPARATION OF STANDARDS

NOTE: Following are standards preparation instructions for a 1 channel system determining $NO_2^- + NO_3^-$ or NO_2^- alone and instructions for a 2 channel system where one channel is used for $NO_2^- + NO_3^-$ and the other channel is used for determining NO_2^-. For the 1 channel system, either NO_2^- or NO_3^- standards may be used. We recommend the use of NO_3^- standards when running a 1 channel method for $NO_2^- + NO_3^-$. For the 2 channel system, we recommend the use of separate $NO_2^- + NO_3^-$ standard sets.

Standard 1. Stock Nitrate Standard 200 mg N/L as NO_3^-

By Volume: In a **1 L** volumetric flask, dissolve **1.444 g potassium nitrate** (KNO_3) in about **600 mL DI water**. Dilute to the mark and invert to mix. This solution is stable for six months.

Standard 2. Stock Nitrite Standard, 200 mg N/L as NO_2^-

By Volume: In a **1 L** volumetric flask, dissolve **0.986 g sodium nitrite** ($NaNO_2$) or **1.214 g potassium nitrite** (KNO_2) in approximately **800 mL DI water.** Dilute to the mark and invert to mix. Refrigerate. This solution is stable for 3-5 days.

Nitrate Standards

Working Standards (Prepare Daily)	**A**	**B**	**C**	**D**	**E**	**F**
Concentration mg N/L as NO_3	20.0	8.0	4.0	1.00	0.40	0.20
By Volume						
Volume (mL) of **stock standard 1** diluted to 250 mL with **DI water**	25.0	10.0	5.0	---	---	---
Volume (mL) of **standard A** diluted to 250 mL with **DI water**	---	---	---	12.5	5.00	2.50
By Weight						
Weight (g) of **stock standard 1** diluted to final weight (~250 g) divided by factor below with **DI water**	25.0	10.0	5.0	---	---	---
Weight (g) **standard A** diluted to final weight (~250 g) divided by factor below with **DI water**	---	---	---	12.5	5.00	2.50
Division Factor Divide exact weight of the standard by this factor to give the final weight	0.1	0.04	0.02	0.05	0.02	0.01

Nitrite Standards

Working Standards (Prepare Daily)	**A**	**B**	**C**	**D**	**E**	**F**
Concentration mg N/L as NO_2	20.0	8.0	4.0	1.00	0.40	0.20
By Volume						
Volume (mL) of **stock standard 2** diluted to 250 mL with **DI water**	25.0	10.0	5.0	---	---	---
Volume (mL) of **standard A** diluted to 250 mL with **DI water**	---	---	---	12.5	5.00	2.50
By Weight						
Weight (g) of **stock standard 2** diluted to final weight (~250 g) divided by factor below with **DI water**	25.0	10.0	5.0	---	---	---
Weight (g) **standard A** diluted to final weight (~250 g) divided by factor below with **DI water**	---	---	---	12.5	5.00	2.50
Division Factor Divide exact weight of the standard by this factor to give the final weight	0.1	0.04	0.02	0.05	0.02	0.01

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. 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.

8.2. Samples must be preserved with H_2SO_4 to a pH< 2 and cooled to 4°C at the time of collection.

8.3. Samples should be analyzed as soon as possible after collection. If storage is required, preserved samples are maintained at 4°C and may be held for up to 28 days.

8.4. Samples to be analyzed for nitrate or nitrite only should be cooled to 4°C and analyzed within 48 hours.

8.5. CAUTION: Samples must not be preserved with mercuric chloride or thiosulfate because this will degrade the cadmium column.

8.6. If build-up of suspended matter in the reduction column restricts sample flow, the samples may be prefiltered.

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. Prepare reagents and standards as described in Section 7.

11.2. Set up manifold as shown in Section 17.

11.3. Input data system parameters as in Section 17.

11.4. 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.

11.5. Adjust samples to pH between 5 and 9 before analysis with either concentrated HCl or NaOH for preserved samples.

11.6. Place samples in the autosampler. Input the sample identification required by the data system.

11.7. Troubleshooting Guide in the System Operation Manual. This guide is also available on request from Lachat.

11.8. Instructions for repacking a cadmium column are available at customer request. Please request a copy from the Technical Services Department.

12. DATA ANALYSIS AND CALCULATIONS

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

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

12.3 Report sample results for nitrate/nitrite in mg N/L as NO_3^- or NO_2^- to two significant figures for samples above the MDL. Report results below the MDL as less than the detection limit.

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 and 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 Method 353.2

16.2. Methods for Determination of Inorganic Substances in Water and Fluvial Sediments. Book 5. Chapter A1. U.S. Department of the Interior, U.S. Geological Survey.

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: 55samples/h, 65 s/sample
Pump Speed: 35
Cycle Period: 65

Analyte Data:

Concentration Units: mg N/L
Peak Base Width: 25 s
% Width Tolerance: 100
Threshold: 7083
Inject to Peak Start: 21 s
Chemistry: Direct

Calibration Data:

Level	1	2	3	4	5	6	7
Concentration mg N/L	20.0	8.0	4.0	1.0	0.40	0.20	0.00

Calibration Rep Handling: Average
Calibration Fit Type: 2nd Order Polynomial
Weighting Method: 1/X
Force through zero: No

Sampler Timing:

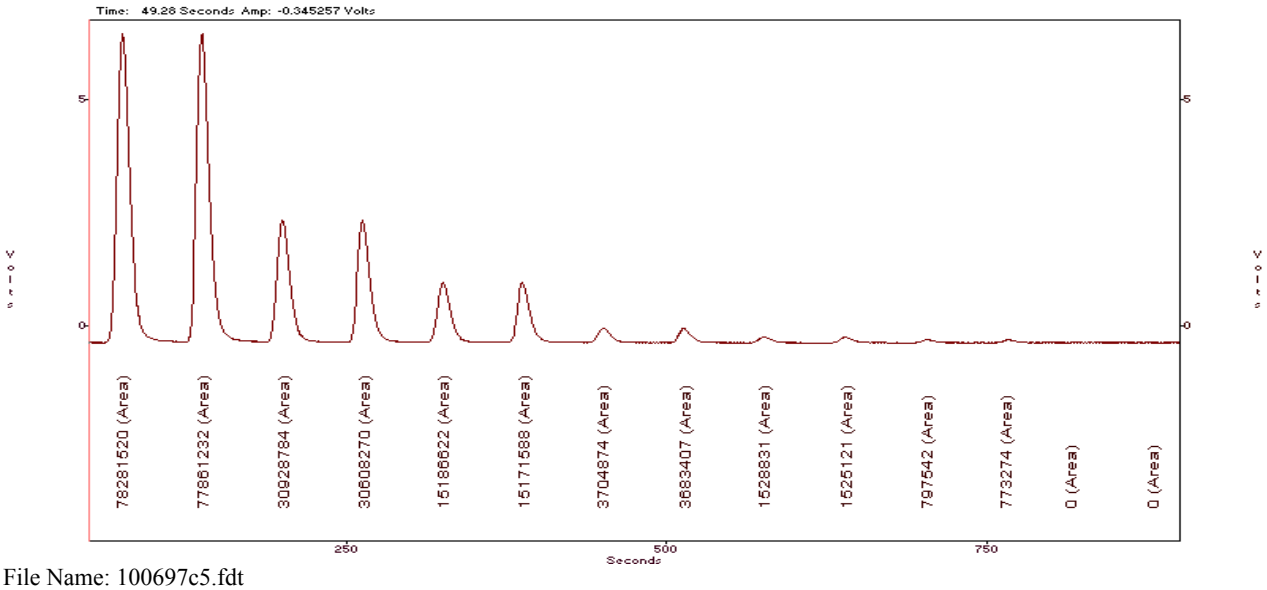
Min. Probe in Wash Period: 5 s
Probe in Sample Period: 12 s

Valve Timing:

Load Time: 0 s
Load Period: 10 s
Inject Period: 55 s

17.2. SUPPORT DATA FOR QUIKCHEM 8000

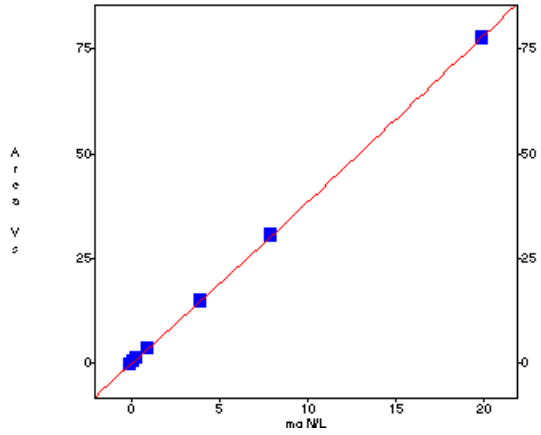
Calibration Data for Nitrate/Nitrite



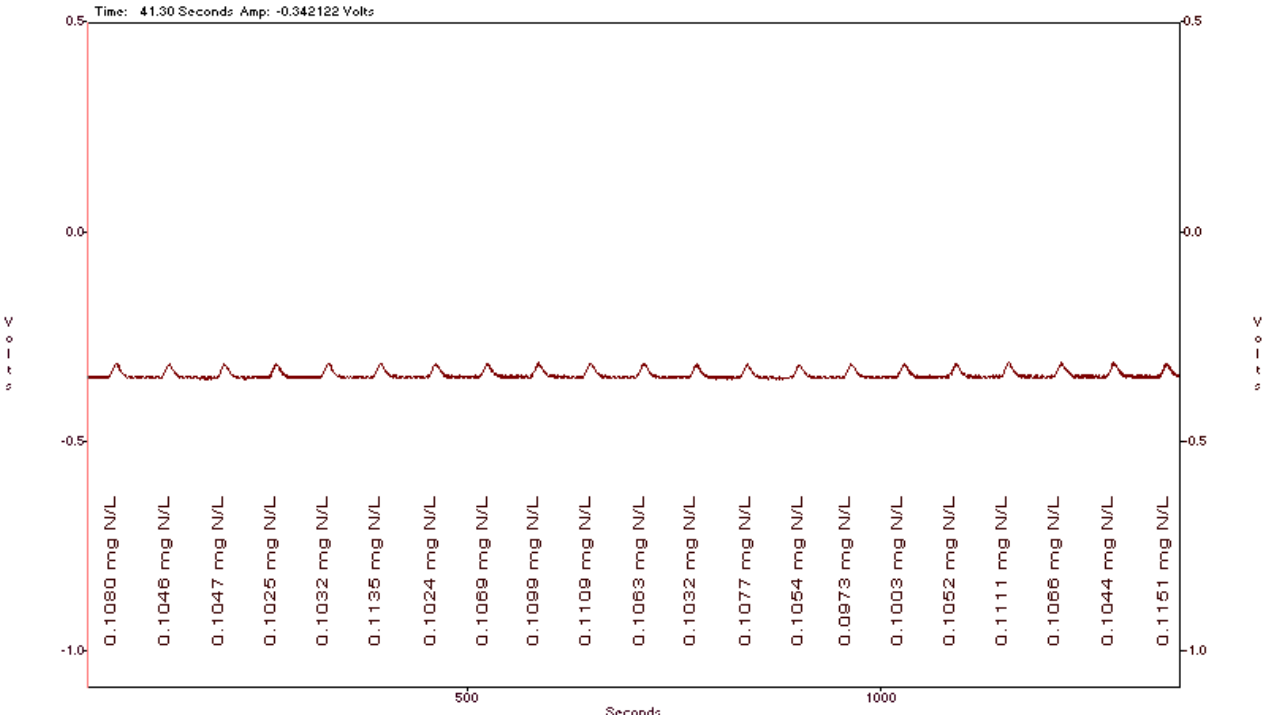
File Name: 100697c5.fdt
Acq. Date: 10June 1997

Calibration Graph and Statistics

Level	Area	mg N/L	Determined	Replicate %RSD	% residual
1	78071376	20.0	20.0	0.4	0.1
2	30768528	8.0	8.0	0.7	-0.4
3	15179105	4.0	4.0	0.1	0.3
4	3694141	1.00	1.0	0.4	2.5
5	1526976	0.40	0.40	0.2	-0.9
6	785408	0.20	0.21	2.2	-3.8
7	0	0.00	0.0	0.0	---



Scaling: None
Weighting: 1/X
2nd Order Poly
Conc = -1.077e-016 Area² + 2.644e-007 Area - 5.678e-009
r = 1.0000



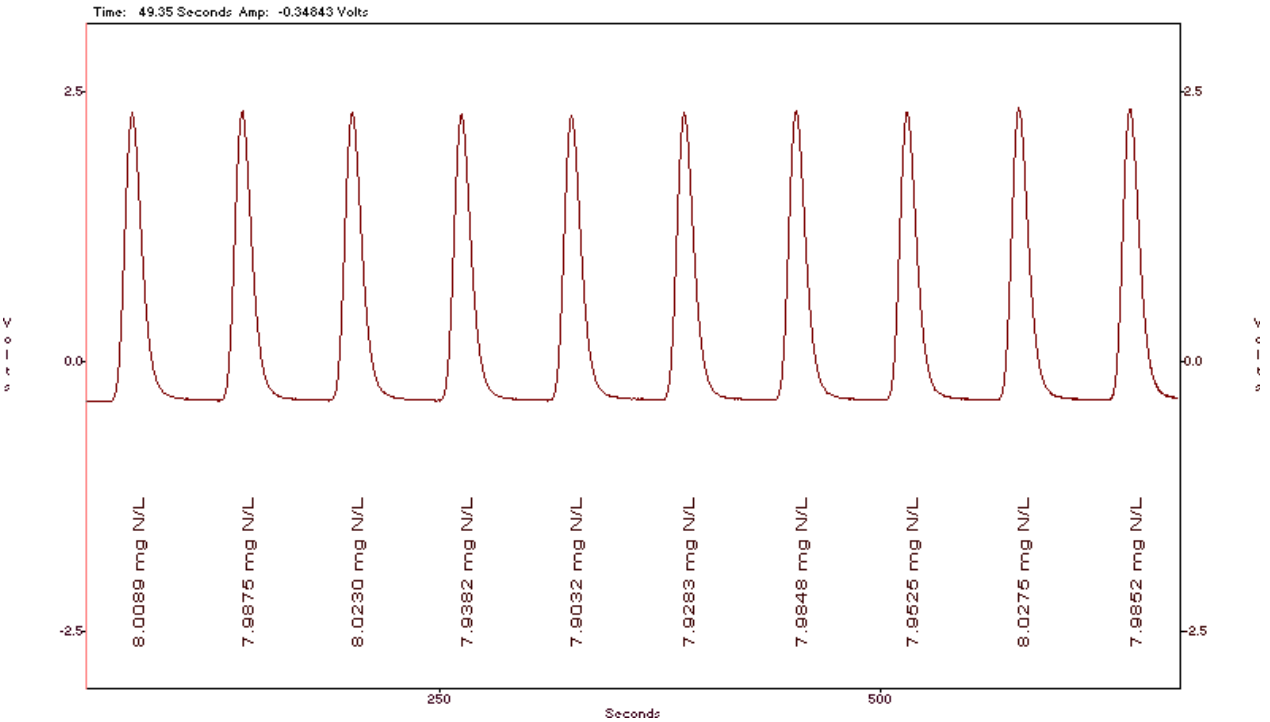
Method Detection Limit for nitrate + nitrite using 0.10 N mg/L standard

MDL= 0.01 mg N/L

Standard Deviation (s) = 0.004 mg N/L, Mean (x) = 0.106 mg N/L, Known value = 0.10 mg N/L

File Name: 100697m2.fdt

Acq. Date: 10 June 1997



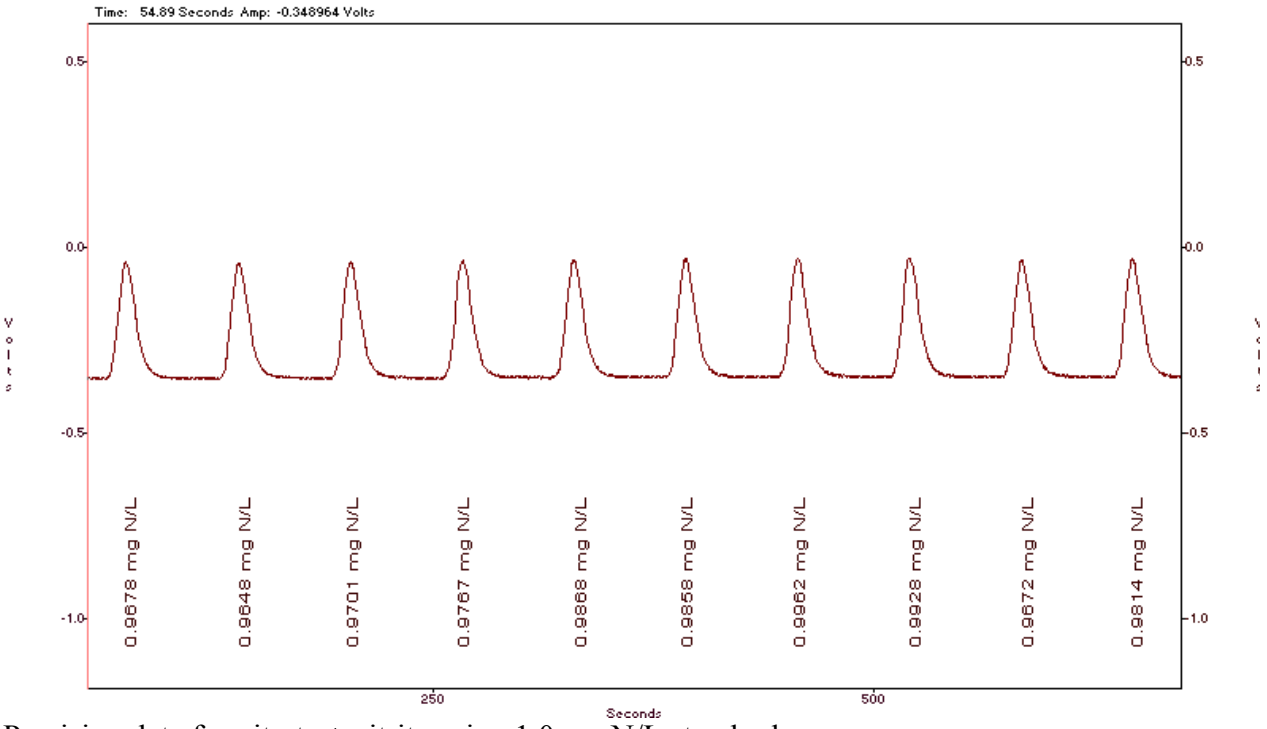
Precision data for nitrate + nitrite using 8.0 mg N/L standard

% RSD = 0.52

Standard Deviation (s) = 0.04 mg N/L, Mean (x) = 7.97 mg N/L, Known value = 8.0 mg N/L

File Name: 100697m2.fdt

Acq. Date: 10 June 1997



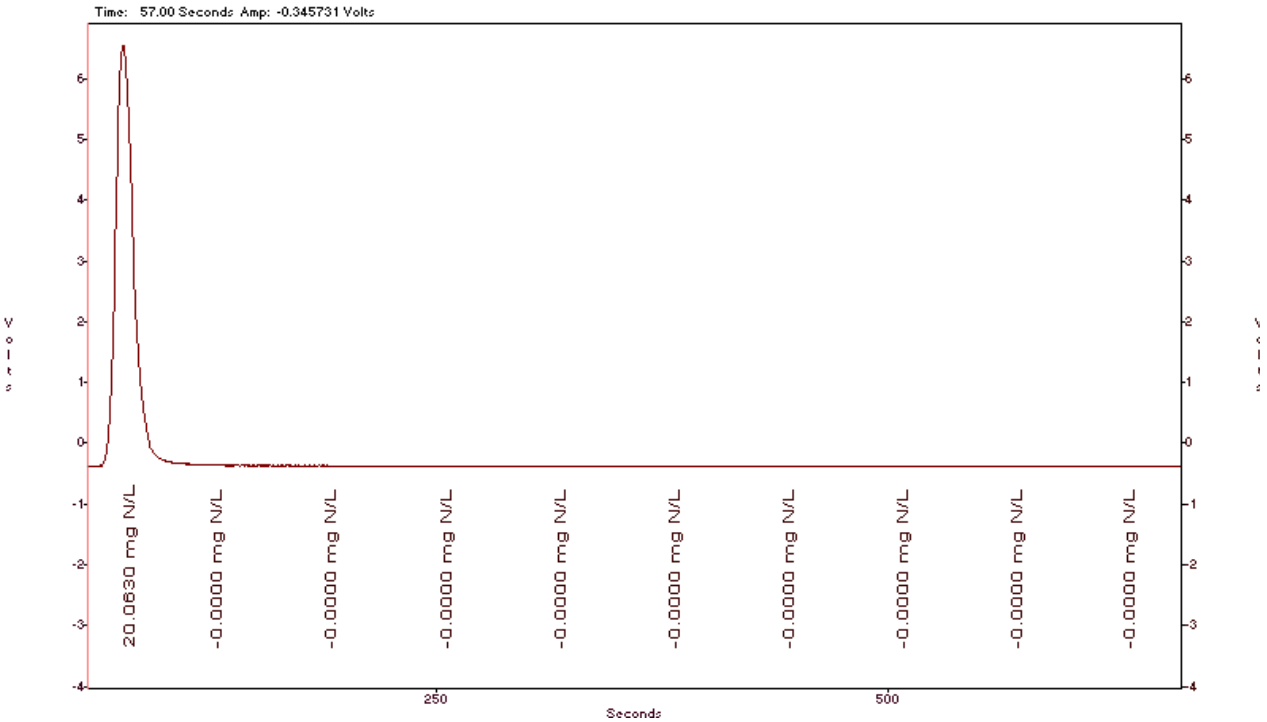
Precision data for nitrate + nitrite using 1.0 mg N/L standard

% RSD = 1.15

Standard Deviation (s) = 0.011 mg N/L, Mean (x) = 0.98 mg N/L, Known value = 1.0 mg N/L

File Name: 100697m2.fdt

Acq. Date: 10 June 1997

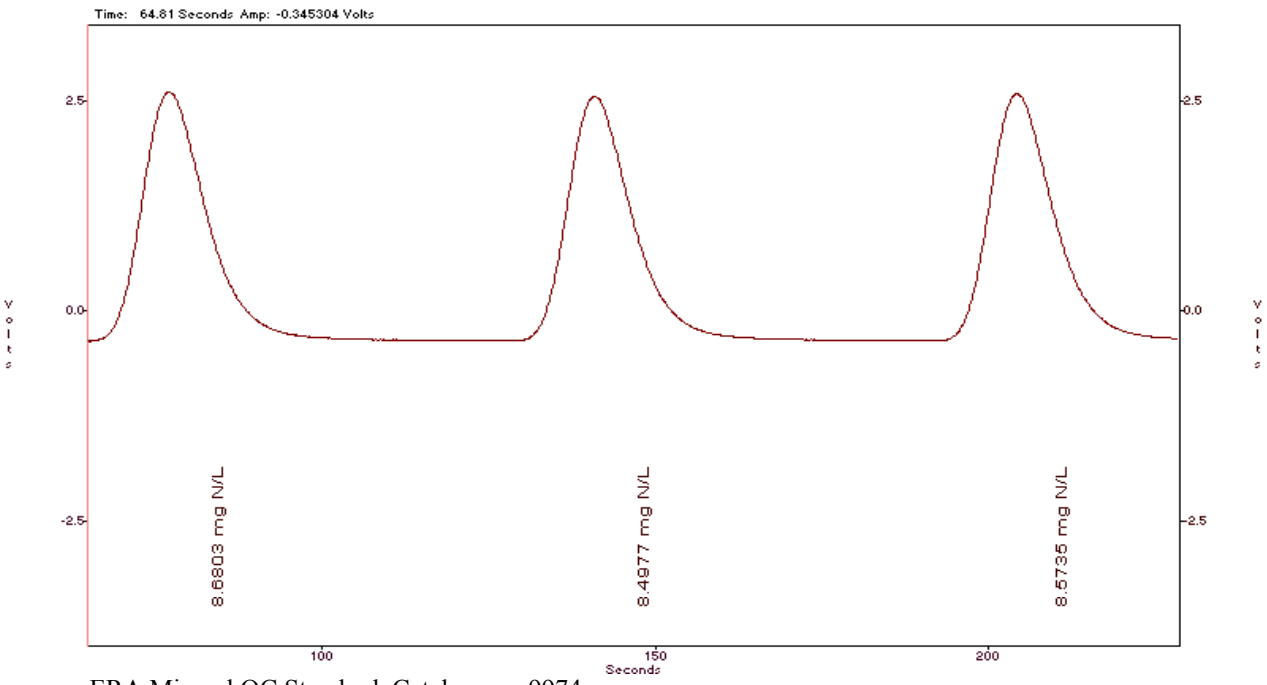


Carryover Study: 20.0 mg N/L standard followed by 10 blanks

Carryover Passed

File Name: 100697co.fdt

Acq. Date: 10 June 1997



ERA Mineral QC Standard, Catalog no. 9974
ERA, Arvada, Colorado
Mean determined concentration: 8.58 mg N/L
Known concentration: 8.66 mg N/L
Interlaboratory acceptance range: 7.71 - 9.61 mg N/L

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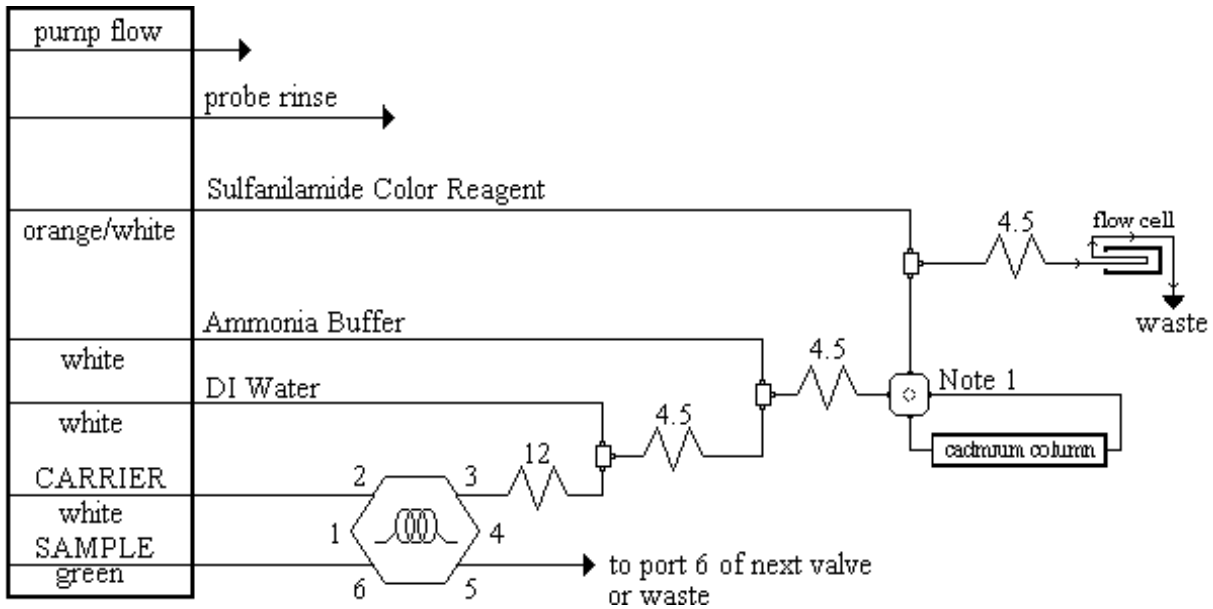
17.3. NITRATE/NITRITE MANIFOLD DIAGRAM



Carrier: Helium degassed DI water
Manifold Tubing: 0.5 mm (0.022 in) i.d. This is 2.54 µL/cm.
AE Sample Loop: 7.5 cm x 0.5 mm i.d.
QC8000 Sample Loop: 13 cm x 0.5 mm i.d.
Interference Filter: 520 nm

Apparatus: An injection valve, a 10 mm path length flow cell, and a colorimetric detector module is required.

4.5: 70 cm of tubing on a 4.5 cm coil support
12: 255 cm of tubing on a 12 cm coil support

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