QuikChem® Method 10-115-01-1-Q

DETERMINATION OF ORTHOPHOSPHATE IN WATERS BY FLOW INJECTION ANALYSIS

LOW FLOW METHOD

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Applications Group

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LACHAT INSTRUMENTS
A Hach Company Brand

QuikChem® Method 10-115-01-1-Q

Orthophosphate in Waters

10 to 200 μg P/L

– Principle –

The orthophosphate ion (PO_4^{3-}) reacts with ammonium molybdate and antimony potassium tartrate under acidic conditions to form a complex. This complex is reduced with ascorbic acid to form a blue complex which absorbs light at 880 nm. The absorbance is proportional to the concentration of orthophosphate in the sample.

– Interferences –

1. Silica forms a pale blue complex which also absorbs at 880 nm. This interference is generally insignificant as a silicate concentration of approximately 30 mg SiO_2/L would be required to produce a 0.005 mg P/L positive error in orthophosphate. See section 17.3.
2. Concentrations of ferric iron (Fe^{3+}) greater than 50 mg/L will cause a negative error due to precipitation of, and subsequent loss, of orthophosphate.
3. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that bias analyte response.
4. Arsenate is determined similarly to phosphate and should be considered when present at concentrations higher than phosphate.

– 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. Glass calibration vials must be used. Lachat part no. 21304 for XYZ samplers.

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QuikChem® Method 10-115-01-1-Q

DETERMINATION OF ORTHOPHOSPHATE BY FLOW INJECTION ANALYSIS COLORIMETRY

1. SCOPE AND APPLICATION

1.1. This method covers the determination of orthophosphate in drinking, ground, and surface waters, and domestic and industrial wastes. This method determines total orthophosphate or if the sample is filtered through a 0.45 micron pore size filter, the result is termed dissolved orthophosphate. The difference between the result of a sample determined directly and filtered is termed insoluble orthophosphate.

1.2. The method is based on reactions that are specific for the orthophosphate (PO_4^{3-}) ion.

1.3. The applicable range is 10 to 200 µg P/L. The method detection limit was determined to be 0.3 µg P/L. 80 samples per hour can be analyzed.

2. SUMMARY OF METHOD

2.1. Only orthophosphate forms a blue color in this test. Polyphosphates and organic phosphorus compounds are not recovered. The sulfuric acid in the molybdate reagent does not have enough contact time with polyphosphates to hydrolyze them.

2.2. The PO_4^{3-} reacts with ammonium molybdate and antimony potassium tartrate under acidic conditions to form a antimony-phosphomolybdate complex. This complex is reduced with ascorbic acid to form a blue complex which absorbs light at 880 nm. The absorbance is proportional to the concentration of PO_4^{3-} in the digested 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. Its 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. Silicate forms a pale blue complex which also absorbs at 880 nm. This interference is generally insignificant as a silicate concentration of approximately 30 mg SiO_2/L would be required to produce a 0.005 mg P/L positive error in orthophosphate.

4.2. Concentrations of ferric iron greater than 50 mg/L will cause a negative error due to competition with the complex for the reducing agent ascorbic acid.

4.3. For dissolved orthophosphate, sample turbidity must be removed by filtration prior to analysis. Sample color that absorbs at 880 nm will also interfere. When in doubt about background absorbance, the background contribution to the total concentration should be determined.

4.4. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that bias analyte response.

4.5. Arsenate is determined similarly to phosphate and should be considered when present at concentrations higher than phosphate.

4.6. Glass calibration vials must be used.

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. Sulfuric Acid

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. Acid-washed glassware: All glassware used in the determination of phosphate should be washed with hot 1:1 HCl and rinsed with distilled water. Preferable, this glassware should be used only for the determination of phosphate and after use it should be rinsed with distilled water and kept covered until needed again. If this is done, the treatment with 1:1 HCl is only required occasionally. Commercial detergent should never be used.

6.4. Special Apparatus

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

6.4.2. Glass calibration vials must be used. Lachat part no. 21304 for XYZ samplers.

7. REAGENTS AND STANDARDS

7.1. PREPARATION OF REAGENTS

Use ASTM Type I 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. Stock Ammonium Molybdate Solution

By Volume: In a **1 L** volumetric flask dissolve **40.0 g ammonium molybdate tetrahydrate** [$(NH_4)_6Mo_7O_{24}\cdot 4H_2O$] in approximately **800 mL DI water**. Dilute to the mark with **DI water** stir for two hours. Store in plastic and refrigerate. Maybe stored up to two months when kept refrigerated.

By Weight: To a tared 1 L container add **40.0 g ammonium molybdate tetrahydrate** [$(NH_4)_6Mo_7O_{24}\cdot 4H_2O$] and **983 g DI water**. Stir for two hours. Store in plastic and refrigerate. Maybe stored up to two months when kept refrigerated.

Reagent 2.Stock Antimony Potassium Tartrate Solution

By Volume: In a 1 L volumetric flask, dissolve **3.0 g antimony potassium tartrate** (potassium antimonyl tartrate hemihydrate $K(SbO)C_4H_4O_6\cdot 1/2H_2O$) or dissolve **3.22 g antimony potassium tartrate** (potassium antimonyl tartrate trihydrate $C_8H_4O_{12}K_2Sb_2\cdot 3H_2O$)in approximately **800 mL DI water**. Dilute to the mark with **DI water** and stir for a minimum of four hours. Store in a dark bottle and refrigerate. Maybe stored up to two months when kept refrigerated.

By Weight: To a **1 L** dark, tared container add **3.0 g antimony potassium tartrate** (potassium antimonyl tartrate hemihydrate $K(SbO)C_4H_4O_6\cdot 1/2H_2O$) or dissolve **3.22 g antimony potassium tartrate** (potassium antimonyl tartrate trihydrate $C_8H_4O_{12}K_2Sb_2\cdot 3H_2O$) and **995 g DI water**. Stir for a minimum of four hours. Store in a dark bottle and refrigerate. Maybe stored up to two months when kept refrigerated.

Reagent 3.Molybdate Color Reagent

By Volume: To a 1 L volumetric flask add about **500 mL DI water**, then add **35.0 mL concentrated sulfuric acid** (CAUTION: The solution will get very hot!). Swirl to mix. When it can be comfortably handled, add **72.0 mL Stock Antimony Potassium Tartrate Solution** (Reagent 2) and **213 mL Stock Ammonium Molybdate Solution** (Reagent 1). Dilute to the mark with **DI water** and invert three times. Degas with helium. Prepare fresh weekly.

By Weight: To a tared 1 L container add **680 g DI water**, then **64.4 g concentrated sulfuric acid** (CAUTION: The solution will get very hot!). Swirl to mix. When it can be comfortably handled, add **72.0 g Stock Antimony Potassium Tartrate Solution** (Reagent 2) and **213 g Stock Ammonium Molybdate Solution** (Reagent 1). Shake and degas with helium. Prepare fresh weekly.

Reagent 4.Ascorbic Acid Reducing Solution, 0.33 M

By Volume: In a 1 L volumetric flask dissolve **60.0 g granular ascorbic acid** (Spectrum catalog no. AS102) in about **700 mL DI water**. Dilute to the mark with **DI water** and invert to mix. Add **1.0 g dodecyl sulfate, sodium salt** ($CH_3(CH_2)_{11}OSO_3Na$) (Aldrich catalog no. 86,210-0). Prepare fresh weekly. Discard if the solution becomes yellow. Do not use ascorbic acid powder.

By Weight: To a tared 1 L container, add **60.0 g granular ascorbic acid** (Spectrum catalog no. AS102) and **975 g DI water**. Stir or shake until dissolved. Add **1.0 g dodecyl sulfate, sodium salt** ($CH_3(CH_2)_{11}OSO_3Na$) (Aldrich catalog no. 86,210-0). Prepare fresh weekly. Discard if the solution becomes yellow. Do not use ascorbic acid powder.

Reagent 5.Sodium Hydroxide - EDTA Manifold Rinse

Dissolve **65 g sodium hydroxide** (NaOH) and **6 g tetrasodium ethylenediamine tetraacetic acid** (Na_4EDTA) in **1.0 L** or **1.0 kg DI water**.

7.2. PREPARATION OF STANDARDS

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

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

By Weight: Two 1 L and five 250 mL containers.

Standard 1. Stock Standard 100.0 mg P/L

In a **1 L** volumetric flask dissolve **0.4393 g primary standard grade anhydrous potassium phosphate monobasic** (KH_2PO_4) that has been dried for one hour at 105ºC in about **800 mL DI water**. Dilute to the mark with **DI water** and invert to mix.

Standard 2. Working Stock Standard Solution 0.500 mg P/L

By Volume: In a **1 L** volumetric flask, dilute **5.00 mL Stock Standard** (Standard 1) to the mark with **DI water**. Invert to mix.

By Weight: To a tared **1 L** container add about **5.00 g Stock Standard** (Standard 1). Divide the actual weight of the solution added by **0.005** and make up to this resulting total weight with **DI water**. Shake to mix.

Set of Five Working Standards

Working Standards (Prepare Daily)	A	B	C	D	E
Concentration µg P/L	200	100	50	10	0.00

By Volume

Volume (mL) of **stock standard 2** diluted to 250 mL with DI water	100	50	25	5	0.0

By Weight

Weight (g) of **stock standard 2** diluted to final weight (~250 g) divided by **factor** below with DI water	100	50	25	5	0.0
Division Factor Divide exact weight of the standard by this **factor** to give final weight	0.4	0.2	0.1	0.02	0.0

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and acid rinsed. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.

8.2. Samples should be stored at 4ºC and determined as soon as possible after collection. If sulfuric acid preservation is used, hydrolysis of any polyphosphate species in the sample will occur.

8.3. The USEPA recommends that samples be filtered immediately upon collection, with a maximum holding time of 48 hours.

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. An analytical batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

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 5 percent of all samples (one sample in each batch of no more than twenty 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 no more than twenty 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.

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 of no more than twenty samples. 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 of no more than twenty samples, 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 9.7.1. 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 shown 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 sampler. Input the information required by the data system, such as concentration, replicates and QuikChem 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.

11.1.7. Glassware contamination is a problem in low level phosphorus determinations. Glassware should be washed with 1:1 HCl and rinsed with deionized water. Commercial detergents should rarely be needed but, if they are used, use special phosphate-free detergents for lab glassware.

11.2. SYSTEM NOTES

11.2.1. 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.

11.2.2. Allow 15 min for heating unit to warm up to 37°C.

11.2.3. If necessary, at end of run place the color reagent and ascorbic acid transmission lines into the NaOH - EDTA solution (Reagent 5). Pump this solution for approximately 5 minutes to remove any precipitated reaction products. Then place these lines in water and pump for an additional 5 minutes. Then pump dry all lines.

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 μg P/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 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 the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993, Method 365.1

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, Method I-2601-85.

16.3. Standard Methods for the Examination of Water and Wastewater, 18th Edition, p. 4 - 116, Method 4500-P F (1992)

16.4. Guideline and Format for EMSL-Cincinnati Methods. EPA-600/8-83-020, August 1983.

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:	80 samples/h, 45 s/sample
Pump Speed:	35
Cycle Period:	45

Analyte Data:

Concentration Units:	µg P/L
Peak Base Width:	27.006 s
% Width Tolerance:	100
Threshold:	4900
Inject to Peak Start:	10 s
Chemistry:	Direct

Calibration Data:

Level	1	2	3	4	5
Concentration µg P/L	200	100	50	10	0.00

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

Sampler Timing:

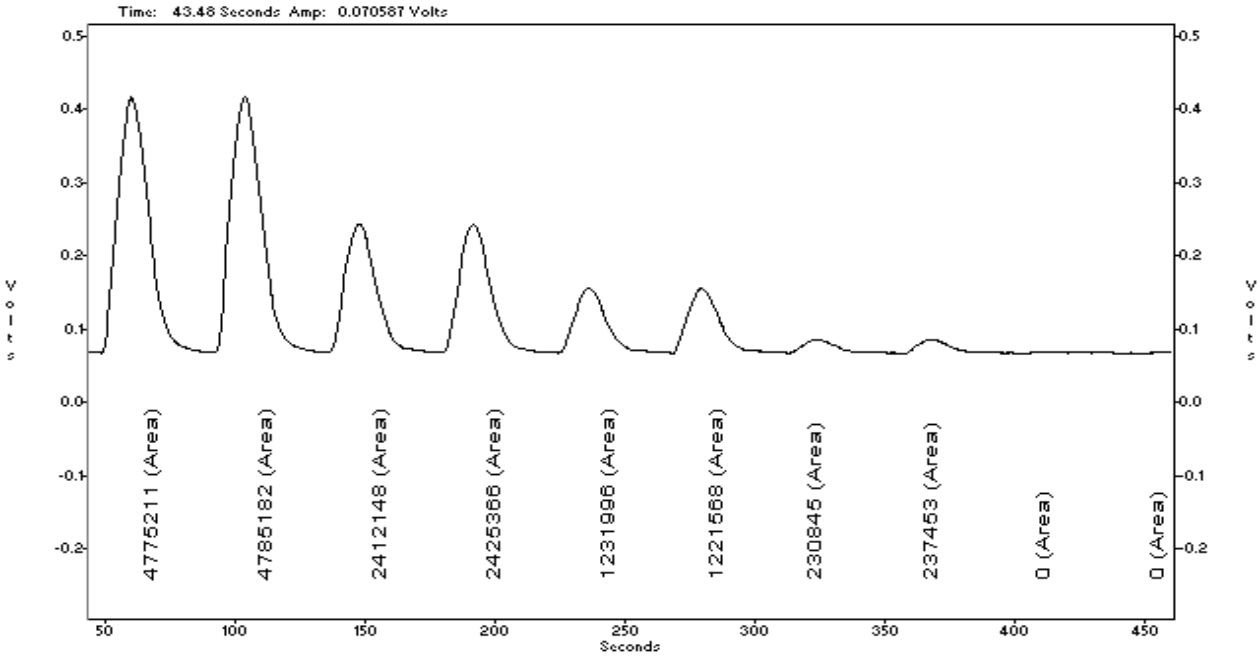
Min. Probe in Wash Period:	14 s
Probe in Sample Period:	20 s

Valve Timing:

Load Time:	0 s
Load Period:	15 s
Inject Period:	30 s

17.2. SUPPORT DATA FOR QUIKCHEM 8000

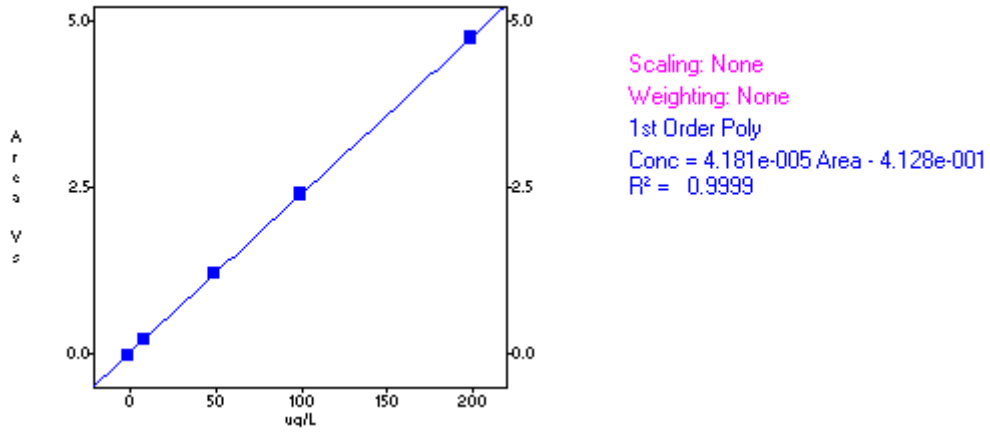
Calibration Data for Orthophoaphate

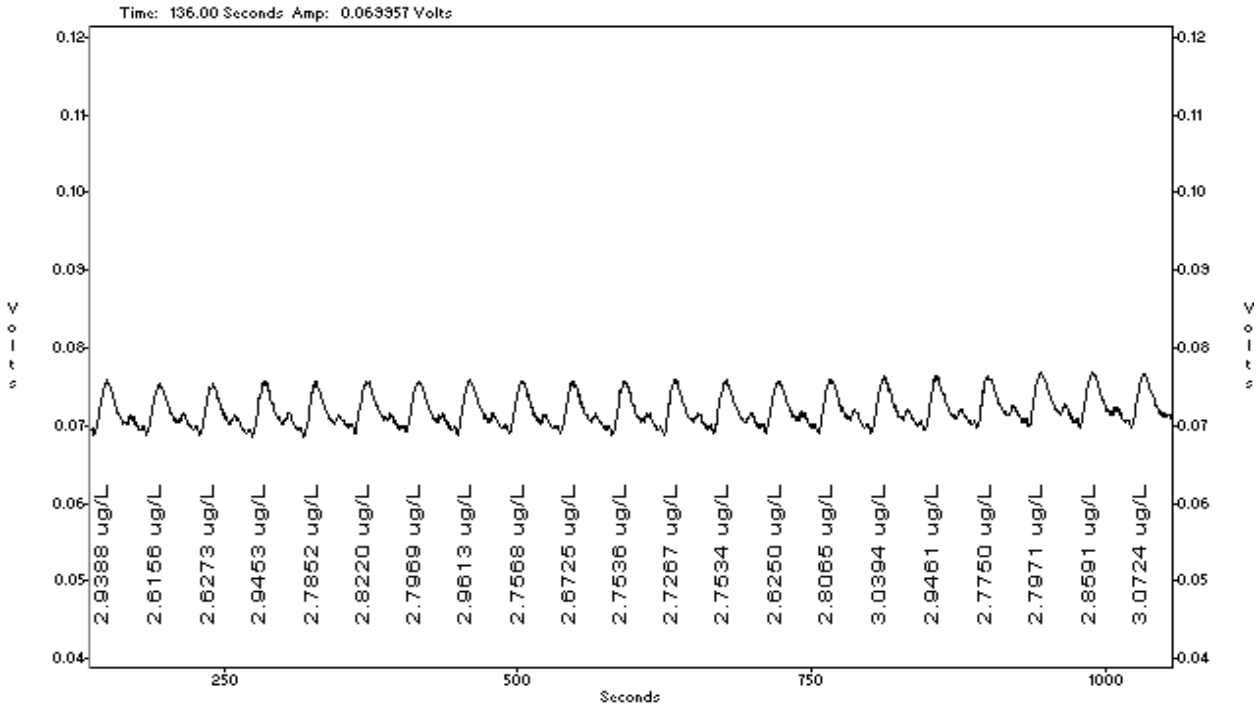


Calibration Data for Orthophosphate in Waters
ACQ Time: November 20, 1995 14:08:13

Calibration Graph and Statistics

Level	Area (V-s)	µg/L	Determined	Replicate %RSD	% residual
1	4780196	200	199	0.1	0.3
2	2418757	100	101	0.4	-0.7
3	1226782	50	51	0.6	-1.8
4	234149	10	9.4	2.0	6.2
5	0	0.0	0.0	0.0	---



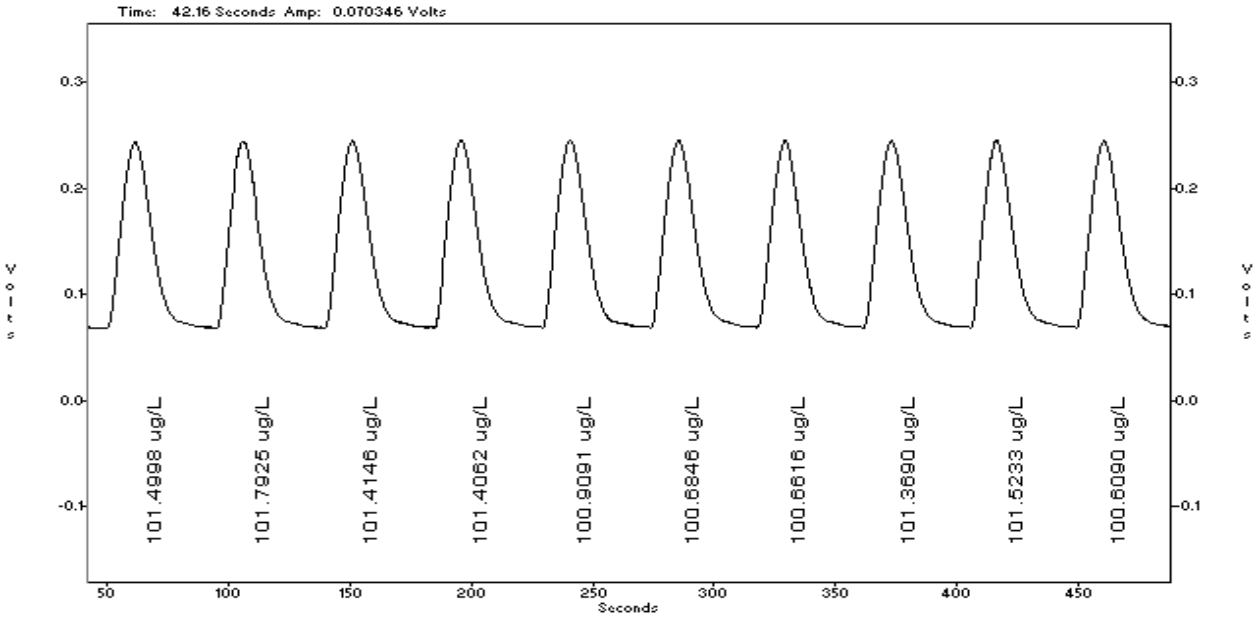


Method Detection Limit for Orthophosphate using a 3.00 µg/L Standard

MDL = 0.32 µg/L

Standard Deviation (s) = 0.128 µg/L Mean (X) = 2.81 µg/L, Known Value = 3.00 µg/L

ACQ. Time: November 20, 1995 14:19:51

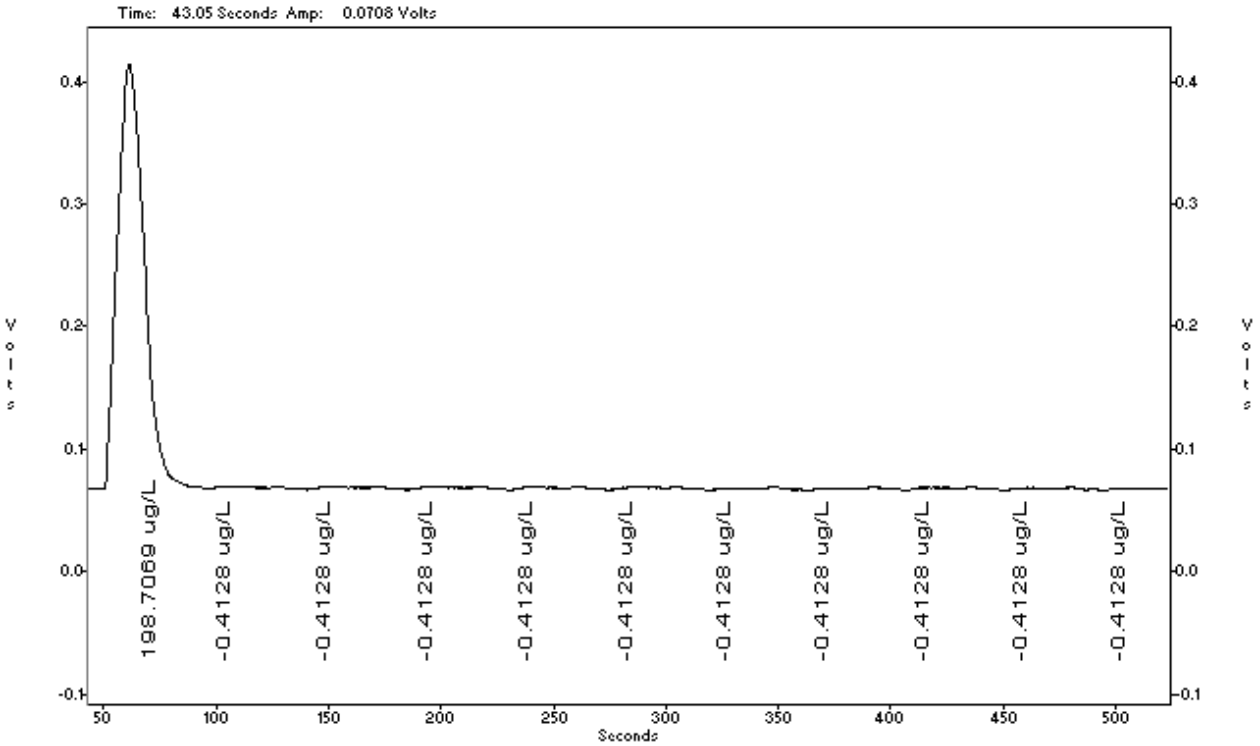


Precision data for Orthophosphate, using a 100 µg/L Standard.

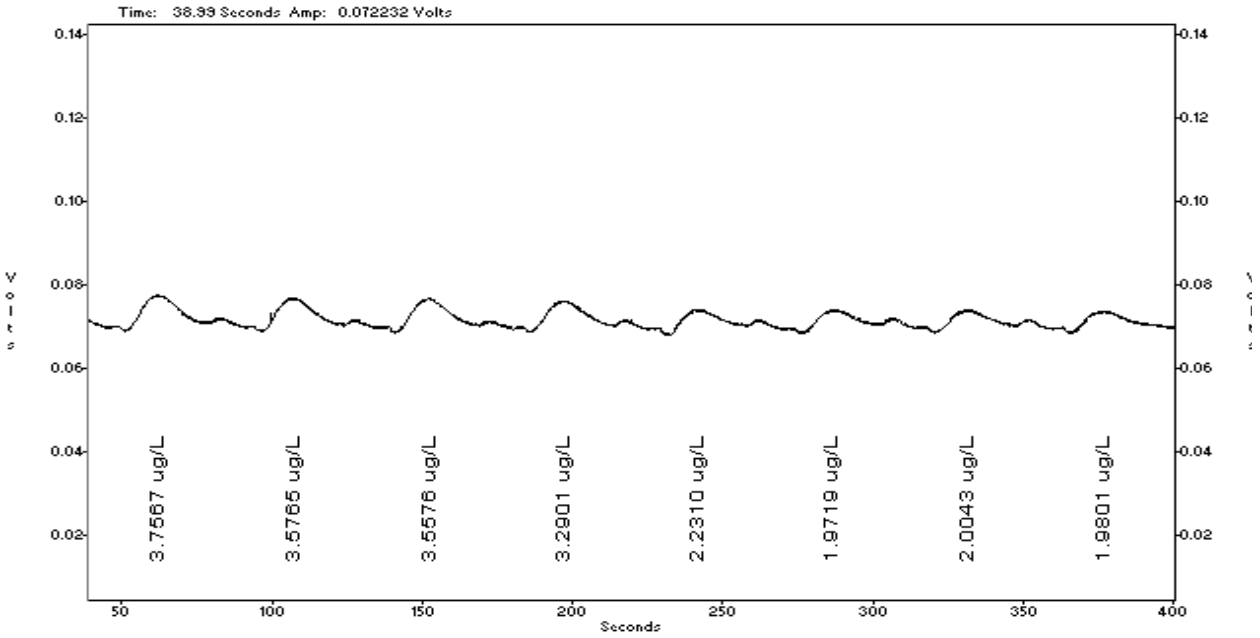
%RSD = 0.401%

Standard Deviation (s) = 0.406 µg/L, Mean (X) = 101.18 µg/L, Known Value = 100 µg/L

ACQ. Time: November 20, 1995 15:12:07

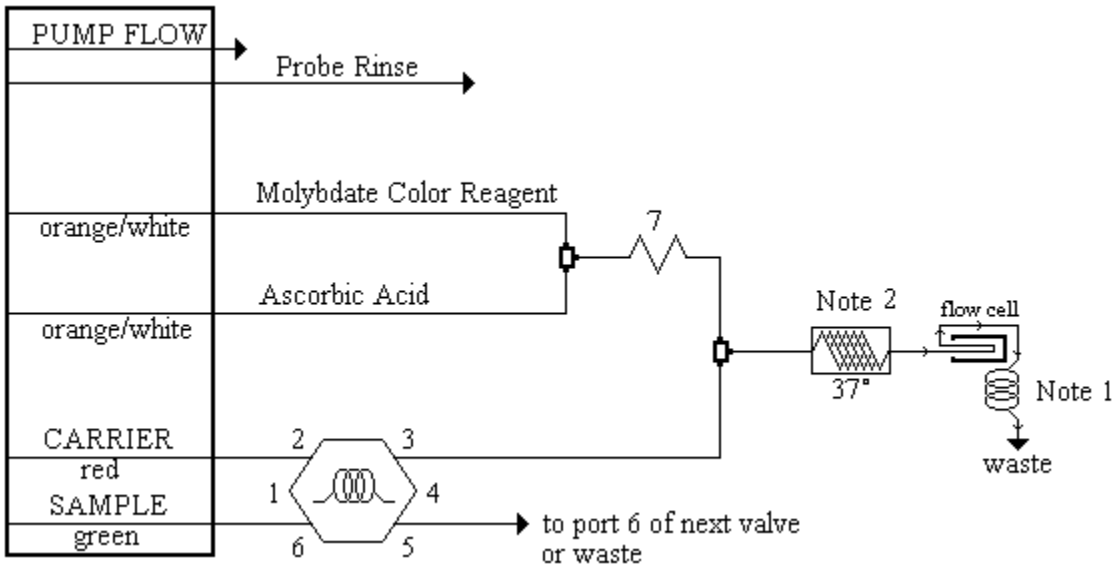


Carryover Study: 200 µg/L Standard followed by 10 blanks
Carryover passed
ACQ Date: November 20, 1995



Silicate Interference, Injections 1-4, 10 mg SiO_2/L, Injections 5-8, 5 mg SiO_2
ACQ Time: November 20, 1995 15:04:23

17.3. ORHTOPHOSPHATE MANIFOLD DIAGRAM



Carrier: **DI water**
Manifold Tubing: **0.5 mm (0.022 in) i.d.** This is **2.5 µL/cm**.
AE Sample Loop: 130.5 cm
QC8000 Sample Loop: 130.5 cm
Interference Filter: 880 nm

Apparatus: An injection valve, a 10 mm path length flow cell, and a colorimetric detector module is required. The  shows 175 cm of tubing wrapped around the heater block at the specified temperature.

7: **135** cm of tubing on a 7 cm coil support

Note 1: Back pressure loop 300 cm x 0.022 i.d.
Note 2: The heating block is wrapped with 175 cm of 0.032 i.d. tubing. This tubing is larger in diameter than the green manifold tubing.