

# *Data Pack*

## *2-cm Path Length Methods*

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*July 2009*



# Table of Contents

Introduction.....	3
Ammonia.....	5
Nitrate-Nitrite .....	10
Orthophosphorus .....	16
Silicate .....	31

# Introduction

The Lachat Instruments Applications team has developed methods utilizing a 2-cm flow cell detector. These methods are able to reach lower detection limits than previously developed Lachat methods measuring the same parameter because of the increase in signal obtained through the larger flow cell, as well as the optimization of other variables during method development.

This Datapack contains application information on Lachat methods utilizing the 2-cm flow cell detector. Information on currently available parameters are included: Ammonia, Nitrate-Nitrite, Orthophosphorus, and Silicate. The summary table on the following page gives an overview of all currently available 2-cm path length methods. More detailed information, including support data, can be found in the remainder of the Datapack.

Please check with your local Lachat representative for updates on new 2-cm flow cell methods.

If you would like to see a specific method reach lower detection limits with the 2-cm flow cell, please contact Lachat Instruments for a feasibility analysis.

For technical assistance, price information, and ordering, please contact Lachat Instruments:

**In the US:**

(800) 247-7613

**International:**

(970) 669-3050

**On the Web:**

[www.lachatinstruments.com](http://www.lachatinstruments.com)

**E-mail for Support**

[support@lachatinstruments.com](mailto:support@lachatinstruments.com)

**E-mail for Sales**

[sales@lachatinstruments.com](mailto:sales@lachatinstruments.com)

### Lachat 2-cm Path Length Methods (as of July 2009)

Analyte	Range	Waters Method No.	Brackish / Seawater Method No.	Note
Ammonia	1.25 - 100 µg NH <sub>3</sub> -N/L	E10-107-06-3-F	E31-107-06-1-G	Uses DCIC for generation of hypochlorite
Nitrate - Nitrite	0.5 - 14 µg N/L	E10-107-04-1-M	E31-107-04-1-F	NO <sub>2</sub> is measured by keeping cadmium column off-line. <b>EPA Equivalent for NPDES reporting.</b>
Orthophosphate	0.25 - 20.0 µg P/L	E10-115-01-1-W	E31-115-01-1-W	For samples w/ low silicate levels. <b>EPA Equivalent for NPDES reporting.</b>
Orthophosphate	0.50 - 100.0 µg P/L	E10-115-01-1-Y	E31-115-01-1-Y	For samples w/ higher silicate levels. <b>EPA Equivalent for NPDES reporting.</b>
Silicate	2.50 - 100 µg SiO <sub>2</sub> /L	E10-114-27-1-C	E31-114-27-1-E	Oxalate suppresses interference by orthophosphate. <b>EPA Equivalent for NPDES reporting.</b>

Note:

- All 2-cm Path Length Methods require a **2-cm Detector Assembly** (Lachat part no. 58025)
- 2-cm Path Length Methods have large-volume sample loops. For replicates, or to use more than one trace level method simultaneously, the **Trace Level Seawater Kit** (Lachat part no. 58112) contains four 21-position racks and sets of 50 mL plastic and 40 mL glass sample vials.
- If you do not see a 2-cm Path Length Method for a specific application, please contact Lachat Instruments for custom method development advisement.

# Ammonia

QuikChem<sup>®</sup> Method 31-107-06-1-G and 10-107-06-3-F

## Ammonia (Phenolate) in Brackish or Seawater

1.25-100 µg NH<sub>3</sub>-N/L

### – Principle –

This method covers the determination ammonia (phenolate) in brackish and seawater. This method may also be used for the analysis of drinking, ground, surface, and domestic waters.

The method is based on the Berthelot reaction. Ammonia reacts with alkaline phenol, and sodium hypochlorite (using DCIC, **sodium dichloroisocyanurate** [dichloro-s-triazine 2,4,6,(1H,3H,H)-trione sodium salt], 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.

This method should **not** be used with acid-preserved samples.

### – Interferences –

1. Calcium and magnesium ions may precipitate if present in sufficient concentration. EDTA is added to the sample in-line to prevent this problem.
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. Sulfide may interfere at levels greater than 2 mg H<sub>2</sub>S/L. Samples containing concentrations greater than this should be diluted.
4. Salinity does not normally interfere in this method. This may be verified by running the samples through the manifold with all reagents pumping, except hypochlorite, which is replaced by deionized water. The resulting concentrations are then compared to those obtained for samples determined with color formation.
5. Eliminate any marked variation in acidity or alkalinity among samples because intensity of measured color is pH-dependent. Likewise, insure that pH of standard ammonia solutions approximates that of the samples.

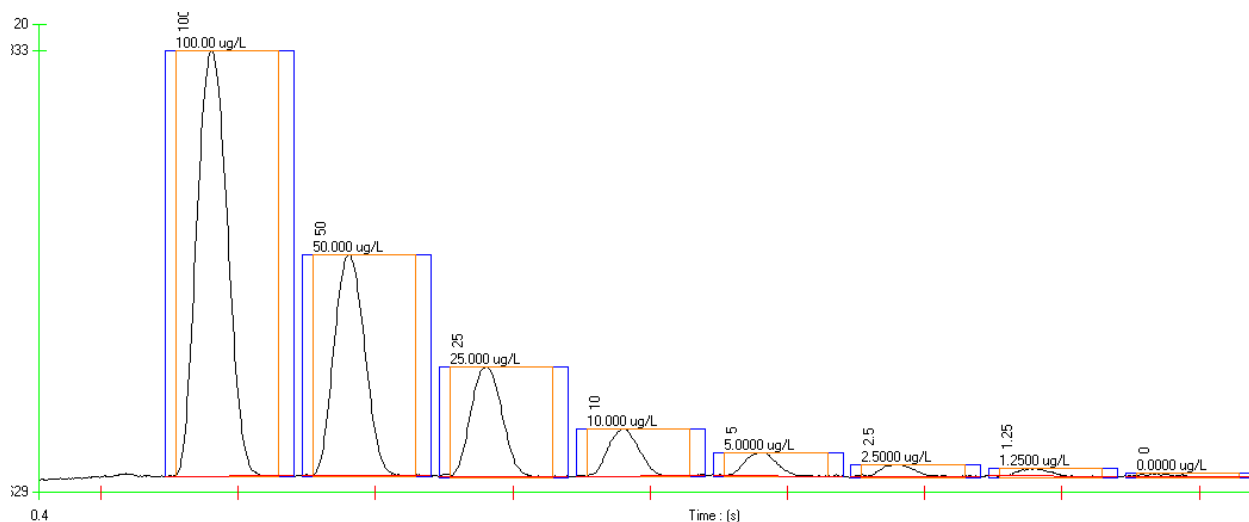
### – Special Apparatus –

Please contact Lachat Sales for ordering information

1. Heating Unit, wrapped with 960 cm of Tefzel Tubing (see manifold diagram)
2. PVC PUMP TUBES MUST BE USED FOR THIS METHOD
3. 2 cm detector apparatus (Lachat Part Number 58025).

## – Support Data for Ammonia QuikChem 8500 –

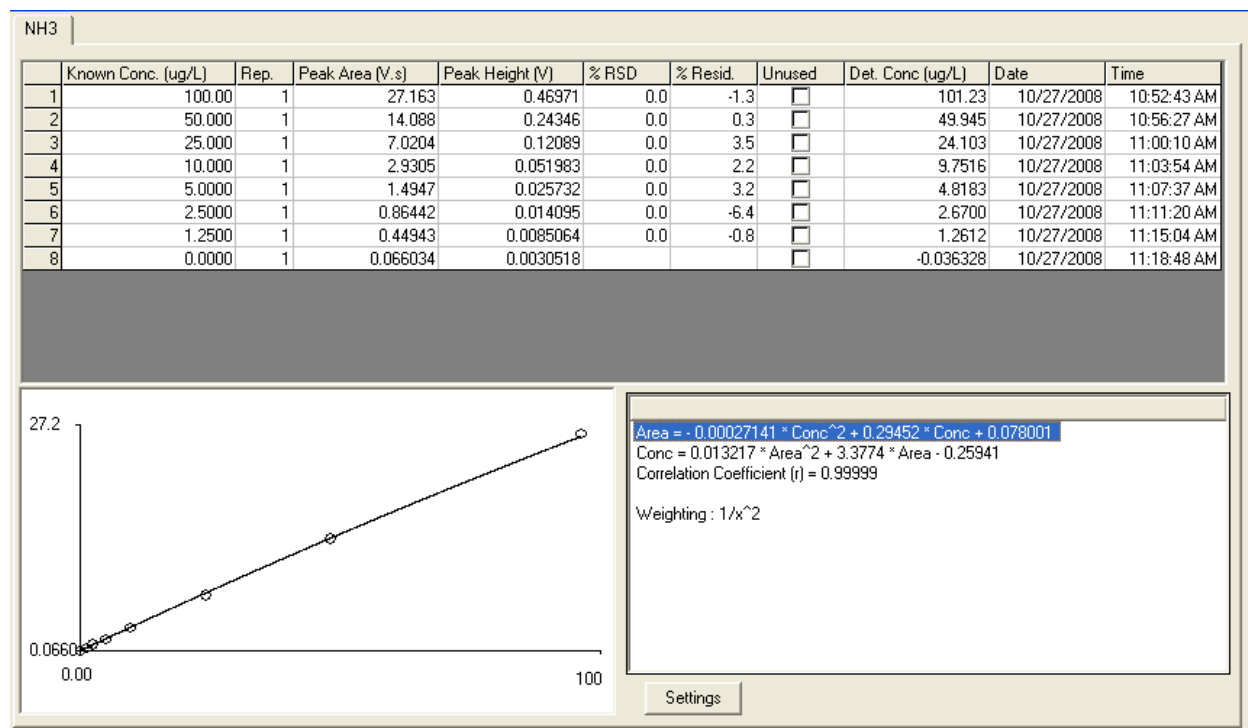
### Calibration Data for Ammonia



File Name: Supp 1 10 27 08.OMN

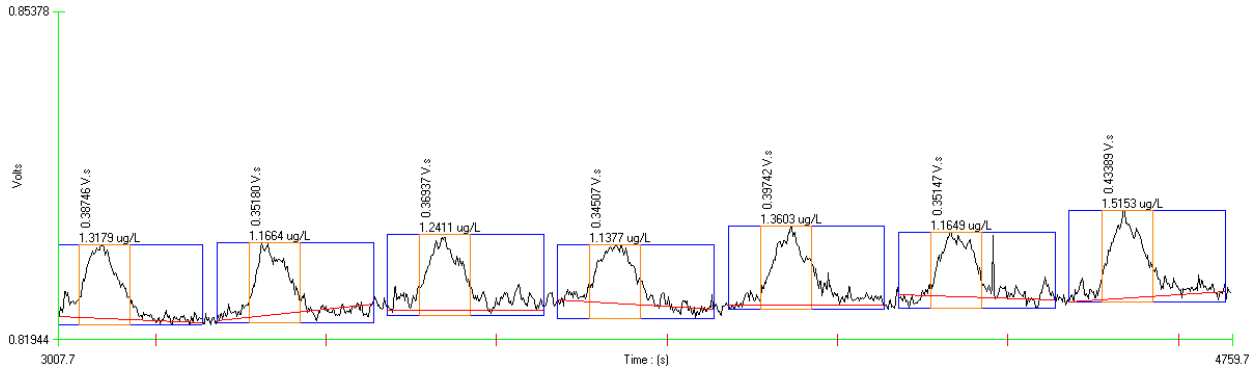
Acq. Date: 27 October 200\*

### Calibration Graph and Statistics



File Name: Supp 1 10 27 08.OMN

Acq. Date: 27 October 2008



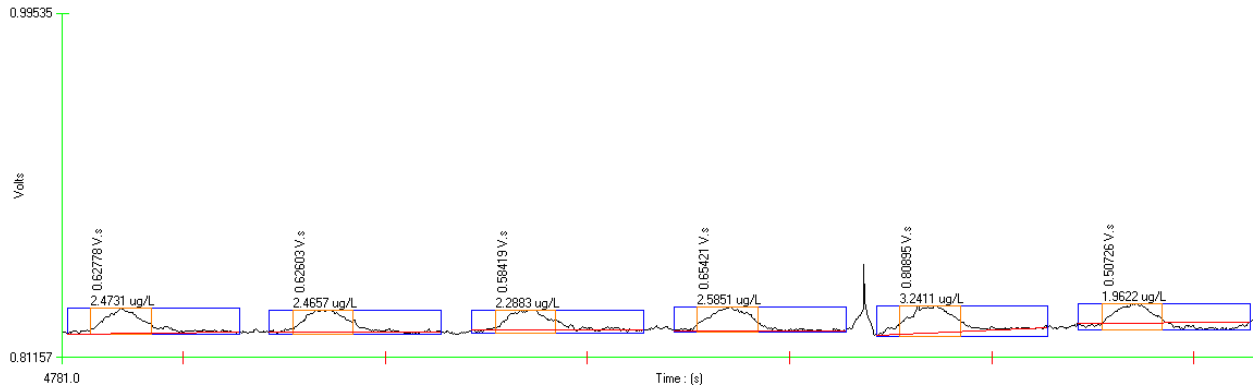
**Method Detection Limit for ammonia using 1.25 µg NH<sub>3</sub>-N/L standard.**

**MDL= 0.41 µg NH<sub>3</sub>-N/L**

Standard Deviation (s) = 0.136 µg NH<sub>3</sub>-N/L, Mean (x) = 1.272 µg NH<sub>3</sub>-N/L, Known value = 1.25 µg NH<sub>3</sub>-N/L

File Name: Supp run 1 10 29 08.OMN

Acq. Date: 29 October 2008

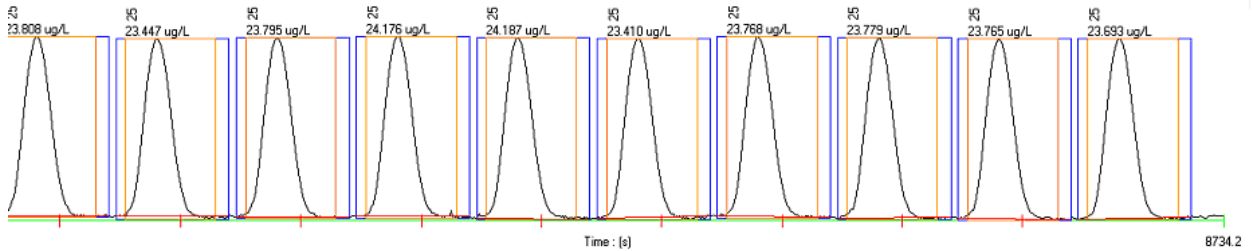


**2.5 µg NH<sub>3</sub>-N/L standard**

Standard Deviation (s) = 0.422 µg NH<sub>3</sub>-N/L, Mean (x) = 2.502 µg NH<sub>3</sub>-N/L, Known value = 2.5 µg NH<sub>3</sub>-N/L

File Name: Supp run 1 10 29 08.OMN

Acq. Date: 29 October 2008



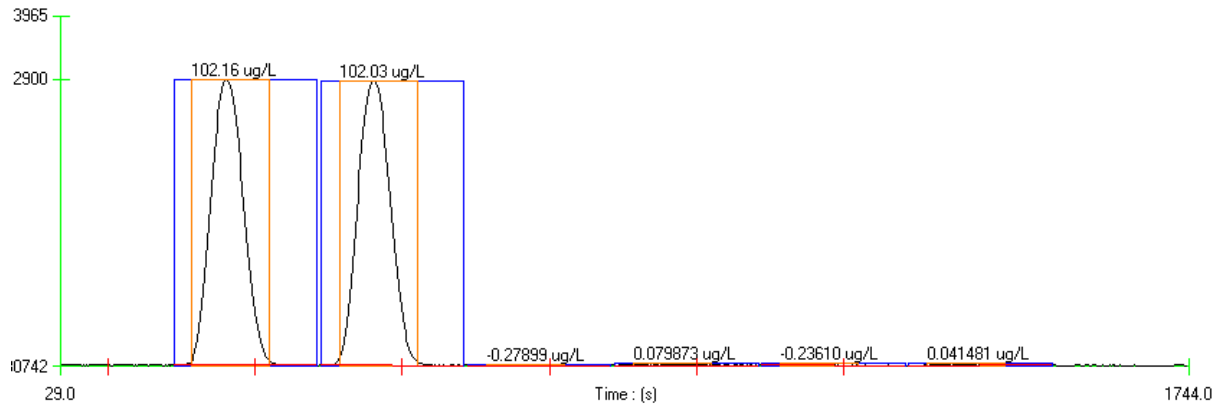
**Precision data for ammonia using 25 µg NH<sub>3</sub>-N/L standard**

**% RSD = 1.06**

Standard Deviation (s) = 0.252 µg NH<sub>3</sub>-N/L Mean (x) = 23.78 µg NH<sub>3</sub>-N/L, Known value = 25 µg NH<sub>3</sub>-N/L

File Name: Supp 1 10 27 08.OMN

Acq. Date: 27 October 2008



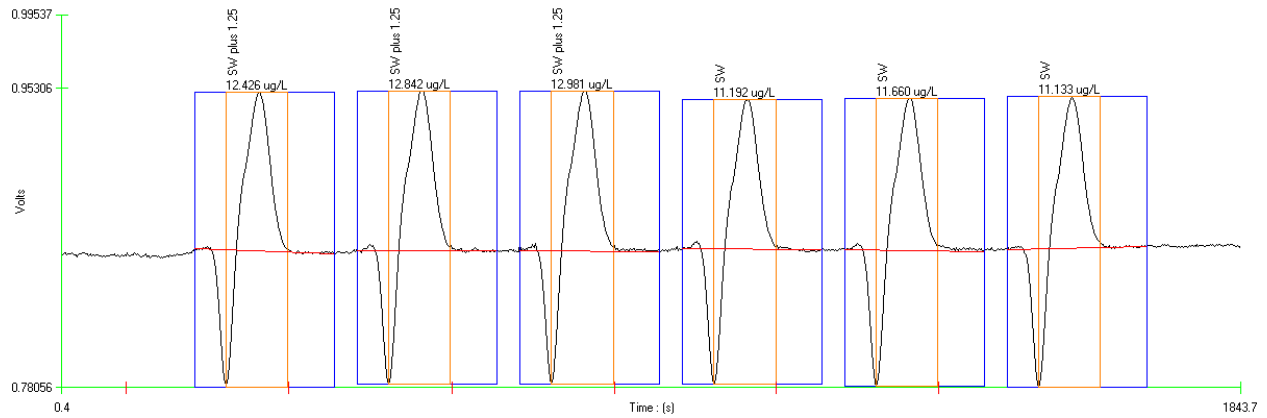
**Carryover Study: 100 µg NH<sub>3</sub>-N/L standard followed by 4 blanks**

Carryover Passed

File Name: Carry 10 27 08.OMN

Acq. Date: 27 October 2008

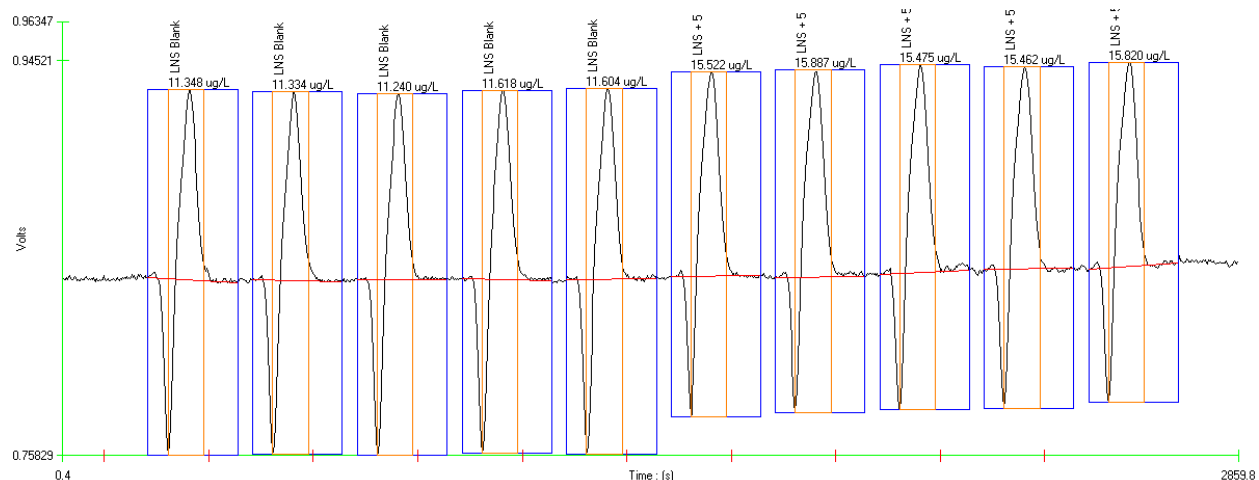
**Seawater spikes**



Sample Type	Initial (µg NH <sub>3</sub> -N/L)	Spiked (µg NH <sub>3</sub> -N/L)	Spike Level (µg NH <sub>3</sub> -N/L)	Spike Recovery
Seawater	11.3	---	---	---
SW + 1.25 µg NH <sub>3</sub> -N/L	---	12.76	1.25	101.67%

File Name: Spikes 10 29 08.OMN

Acq. Date: 27 October 2008



Sample Type	Initial ( $\mu\text{g NH}_3\text{-N/L}$ )	Spiked ( $\mu\text{g NH}_3\text{-N/L}$ )	Spike Level ( $\mu\text{g NH}_3\text{-N/L}$ )	Spike Recovery
Low Nutrient Seawater	11.43	---	---	---
LNS + 5 $\mu\text{g NH}_3\text{-N/L}$	---	15.63	5	95%

File Name: Spikes 2 10 29 08.OMN

Acq. Date: 27 October 2008

Low Nutrient Seawater from Ocean Scientific. Seawater from LUMCON, Louisiana USA.

# Nitrate-Nitrite

QuikChem® Method 31-107-04-1-F and 10-107-04-1-M

## DETERMINATION OF NITRATE AND/OR NITRITE IN BRACKISH WATER BY FLOW INJECTION ANALYSIS

0.25 to 14 µg N/L as NO<sub>3</sub> and/or NO<sub>2</sub>  
(0.018 to 1 µM N as NO<sub>3</sub> and/or NO<sub>2</sub>)

### – 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 diazotization with sulfanilamide under acidic conditions to form a diazonium ion. The diazonium ion is then coupled with N-(1-naphthyl)ethylenediamine dihydrochloride. The resulting pink dye absorbs at 540 nm. Nitrate concentrations are obtained by subtracting nitrite values, which have been previously analyzed, from the nitrite + nitrate values. Though the method is written for seawater and brackish water, it is also applicable to non-saline sample matrixes.

The method is calibrated using standards prepared in deionized water. Heat is used to improve linearity. Once calibrated, samples of varying salinities (0 to 35 ppt) may be analyzed. The determination of background absorbance is necessary only for samples that have color absorbing at 540 nm.

### – Interferences –

1. Sample turbidity interferes. Remove turbidity by filtration with a 0.45 µm pore diameter membrane filter prior to analysis.
2. A positive error could be obtained for samples that contain high concentrations of iron, copper, or other metals. Na<sub>2</sub>EDTA in the buffer helps to prevent this interference.
3. Samples that contain oil and grease will coat the surface of the cadmium, causing it to lose reduction efficiency. This interference can be eliminated by pre-extracting the sample with an organic solvent.
4. Sample color may be subtracted by analyzing the samples with a substitute color reagent, which does not contain the diazotizing agent. This is done by replacing the sulfanilamide-NED-phosphoric acid reagent with a solution containing 100 mL of phosphoric acid per liter.

### – Special Apparatus –

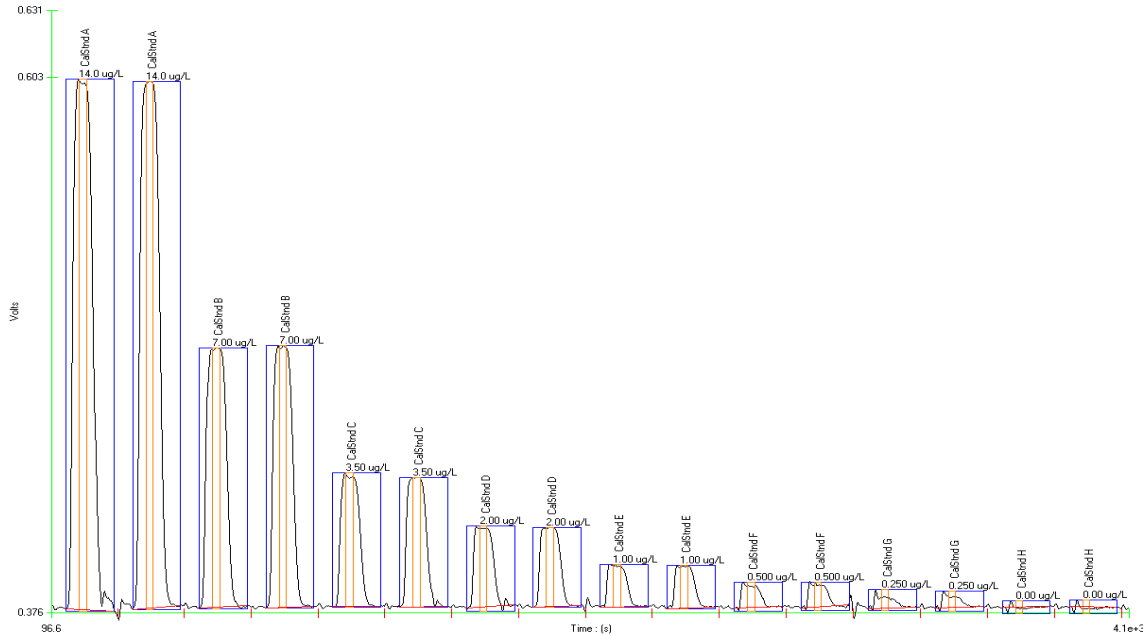
Please contact Lachat Sales for ordering information

1. Heating Unit Lachat Part No. A85X00 (X=1 for 110V, X=2 for 220V)
2. For replicates, or to use more than one trace level method at the same time, Lachat Kit PN 58112 contains 4, 21-position racks, and 50 mL plastic and 40 mL glass sample vials.

3. Sample tubes are needed for 60 Position Samplers (Lachat Part No. 21042)
4. PVC pump tubes must be used for this method.
5. 2-cm Detector Assembly [Lachat Part No. 58025 (Assembly includes 2 cm flow cell, Lachat Part No. 58062)]

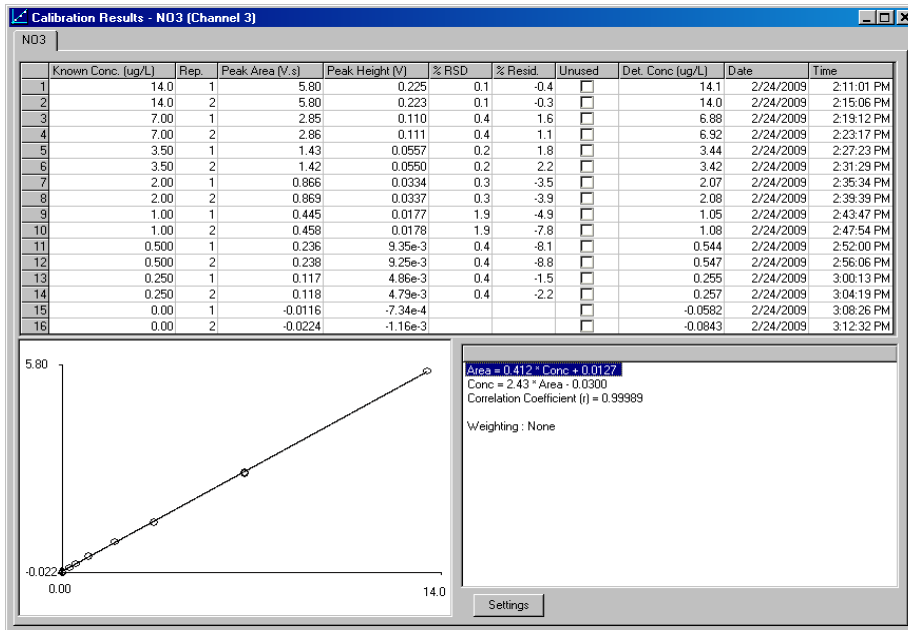
## – Support Data for QuikChem 8500 –

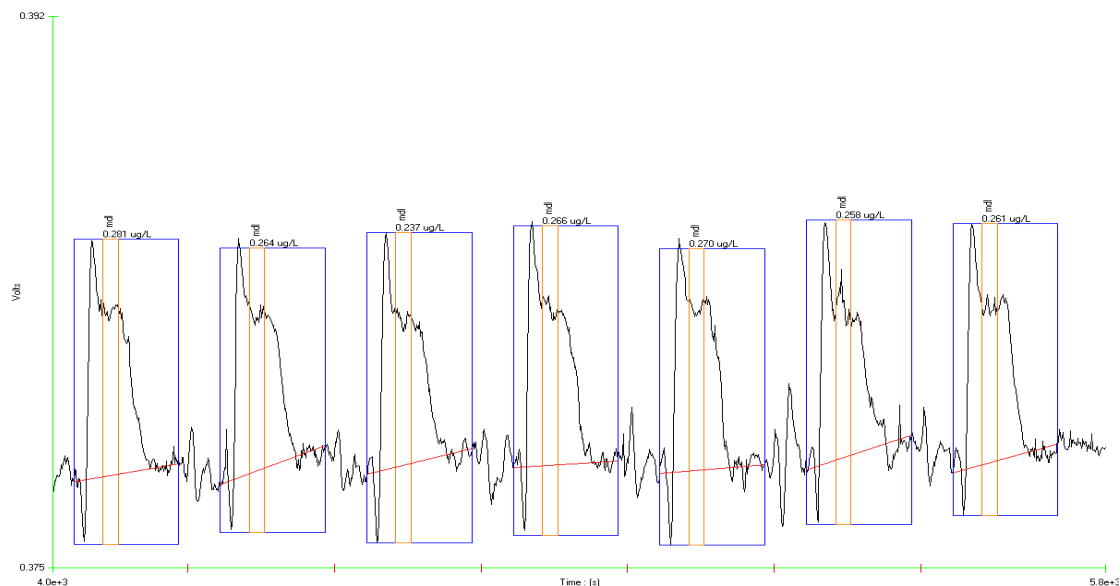
### Calibration Data for Nitrate/Nitrite



File Name: 2-24 cal mdl.omn  
Acq. Date: 24 February 2009

### Calibration Graph and Statistics





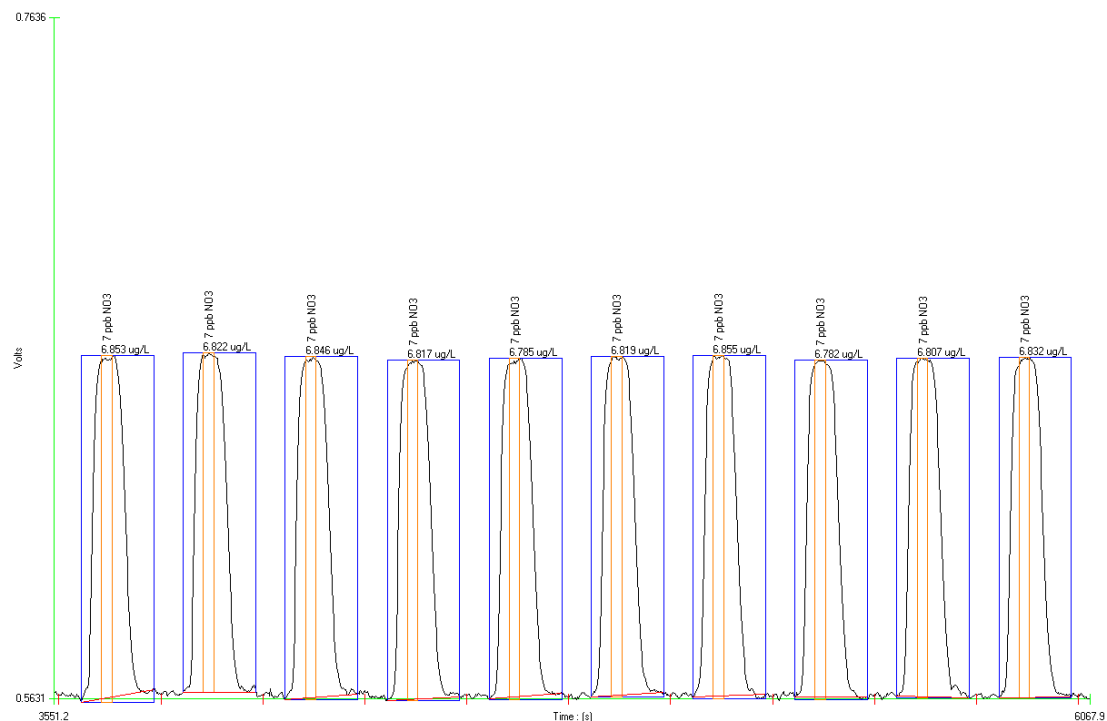
### Method Detection Limit for Nitrate using 0.25 µg N/L standard

**MDL= 0.042 µg N/L (0.003 µM N)**

Standard Deviation (s) = 0.013 µg N/L, Mean (x) = 0.262 µg N/L, Known value = 0.25 µg N/L

File Name: 2-24 cal mdl.omn

Acq. Date: 24 February 2009



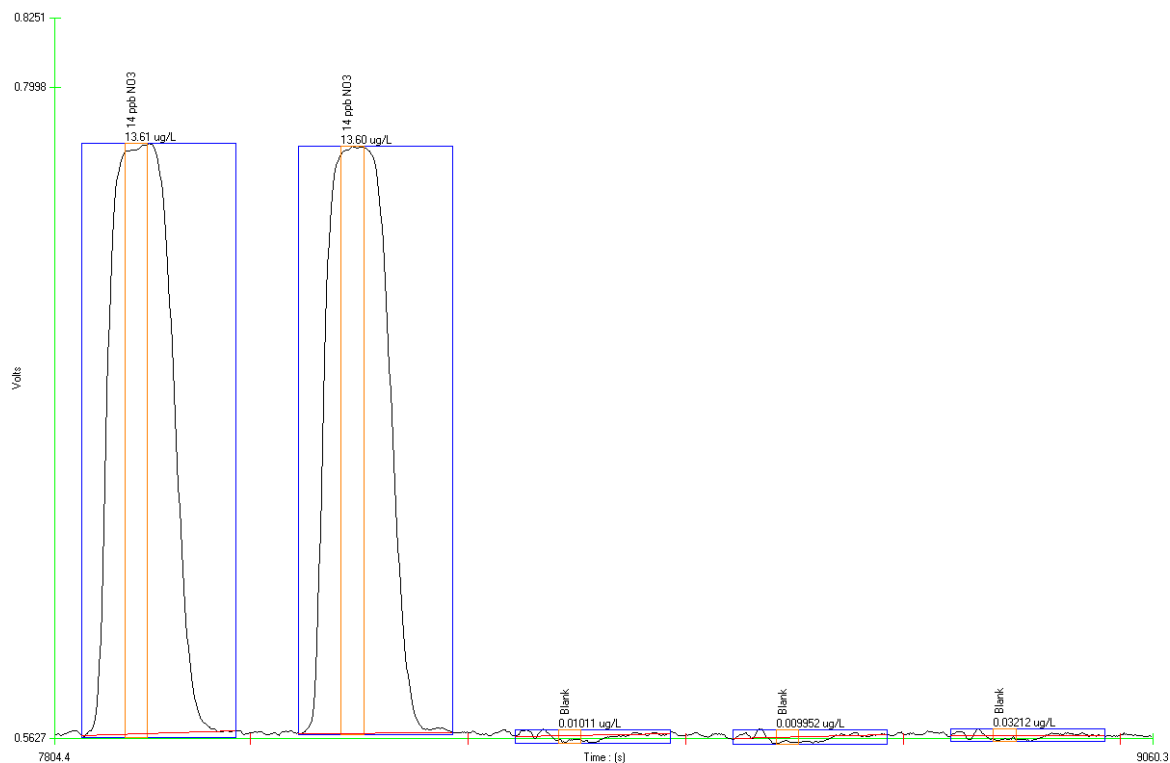
### Precision data for Nitrate using 7.0 µg N/L standard

**% RSD = 0.377**

Standard Deviation (s) = 0.026 µg N/L, Mean (x) = 6.82 µg N/L, Known value = 7.0 µg N/L,

File Name: 7-2 cal support.omn

Acq. Date: 2 July 2008

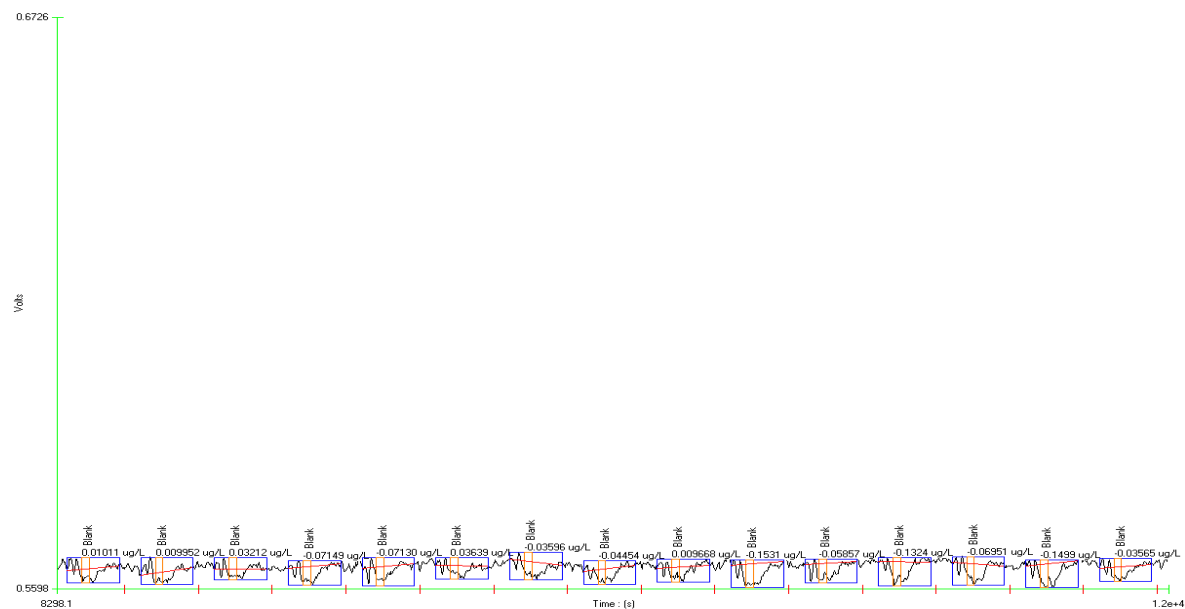


**Carryover Study: Two replicates of 14 µg N/L standard followed by 3 blanks**

Carryover Passed

File Name: 7-2 cal support.omn

Acq. Date: 2 July 2008



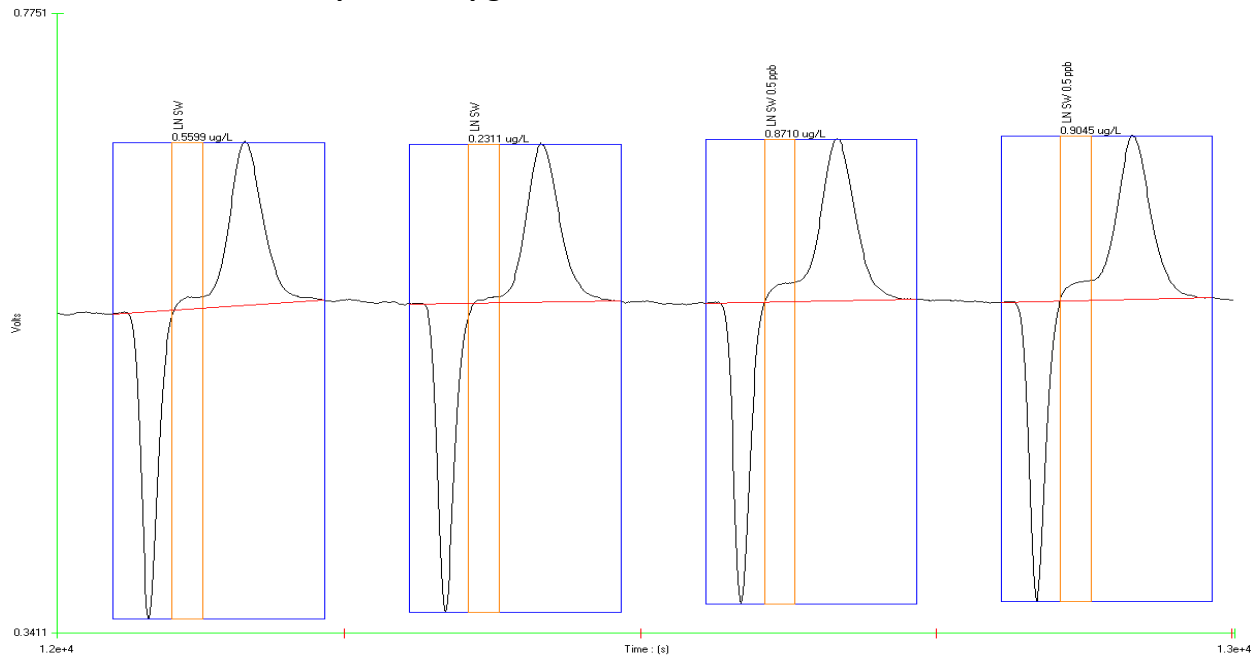
**DIN Blanks**

Average: -0.048 µg N/L, SD = 0.062 µg N/L. Calculated DIN Limits: Detection Limit = 0.186 µg N/L, Decision Limit = 0.373 µg N/L, Determination Limit = 0.560 µg N/L

File Name: 7-2 cal support.omn

Acq. Date: 2 July 2008

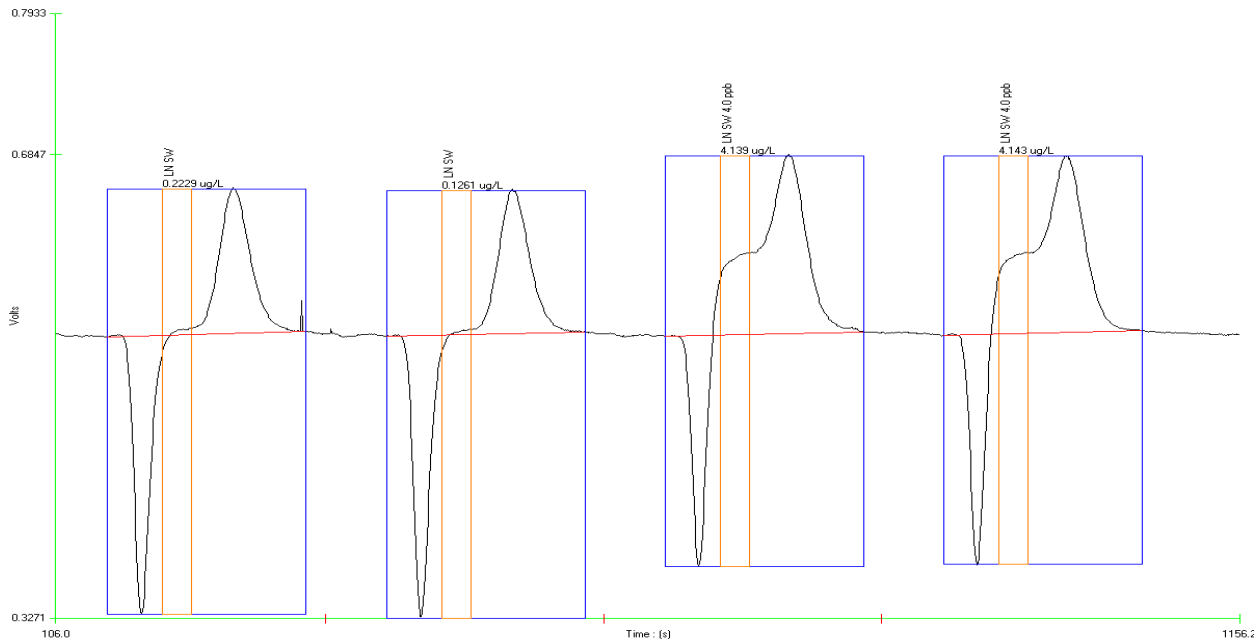
### Low Nutrient Seawater Spikes, 0.5 µg N/L



File Name: 7-2 cal support.omn  
Acq. Date: 2 July 2008

Initial avg (µg N/L)	Spiked avg (µg N/L)	Spike Level (µg N/L)	Spike Recovery
0.396	0.888	0.50	98.5%

### Low Nutrient Seawater Spikes, 0.5 µg N/L



File Name: 7-2 spike2.omn  
Acq. Date: 2 July 2008

Initial avg (µg N/L)	Spiked avg (µg N/L)	Spike Level (µg N/L)	Spike Recovery
0.174	4.14	4.0	99.2%

The Low Nutrient Seawater is supplied by Ocean Scientific International Ltd

**Spike Study Results:**

0.5 and 4.0 µg N/L as NO<sub>3</sub> was spiked into low nutrient seawater, supplied by Ocean Scientific International Ltd. Recoveries of 98.5% and 99.2 % were obtained, respectively. Conclusion: Recoveries of 80-120% can be obtained in this low nutrient seawater matrix.

# Orthophosphorus

QuikChem® Method 31-115-01-1-W and 10-115-01-1-W

## Orthophosphate in Waters

0.25 to 20.0 µg P/L

(For Samples with Very Low (ppb levels) or No Silicate)

### – Principle –

The orthophosphate ion ( $\text{PO}_4^{3-}$ ) 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 significant when measuring P at these trace levels. A silicate concentration of approximately 5 mg  $\text{SiO}_2$ /L will produce approximately 95 µg P/L positive error in orthophosphate. Therefore, this method should be used only for ultra pure waters, or seawaters or brackish waters that have low concentrations of  $\text{SiO}_2$ .
2. Concentrations of ferric iron ( $\text{Fe}^{3+}$ ) greater than 50 mg/L will cause a negative error due to precipitation of, and subsequent loss, of orthophosphate. Samples high in iron can be pretreated with sodium bisulfite to eliminate this interference. Treatment with bisulfite will also remove the interference due to arsenates.
3. Turbidity is removed by filtration. Particles scatter light, causing inaccurate readings.
4. 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 preparations for lab glassware.

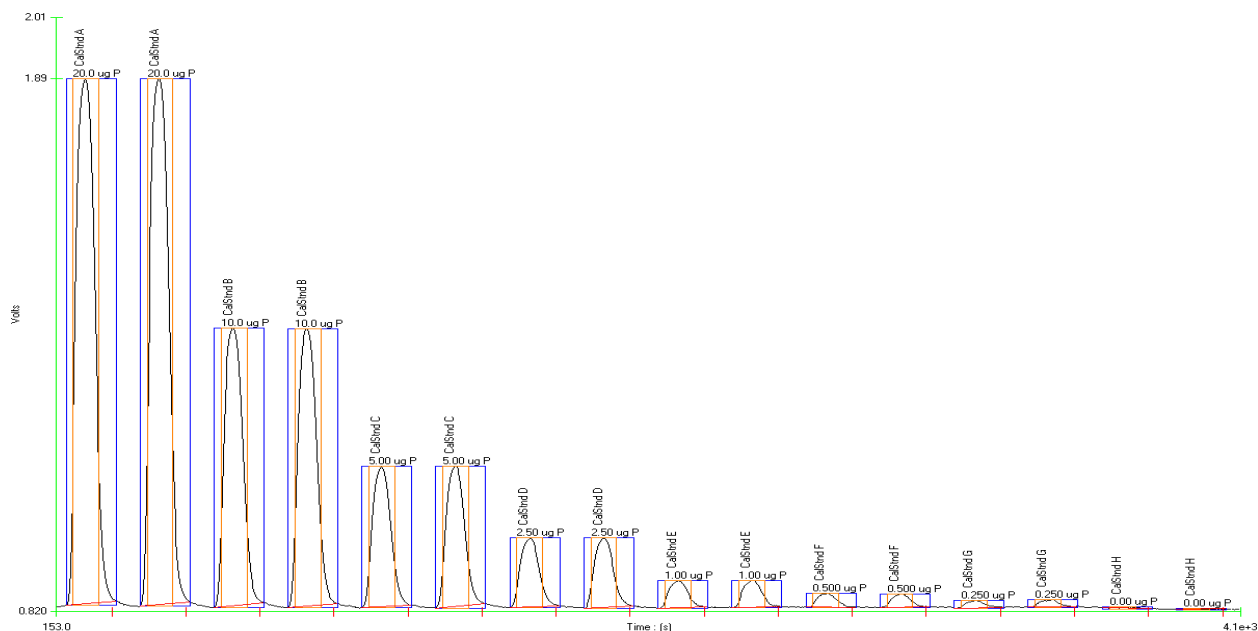
### – Special Apparatus –

Please contact Lachat Sales for ordering information

1. Heating Unit, Lachat Part No. A85X00 (X=1 for 110V, X=2 for 220V), with Hi Temp Insulator Tube (Lachat Part No. 85090)
2. 2-cm Detector Assembly [Lachat Part No. 58025 (Assembly includes 2 cm flow cell, Lachat Part No. 58062)]
3. Glass calibration vials (PN 21304) and 16 x 100 mm sample vials must be used with this method.

## – Support Data for QuikChem 8500 –

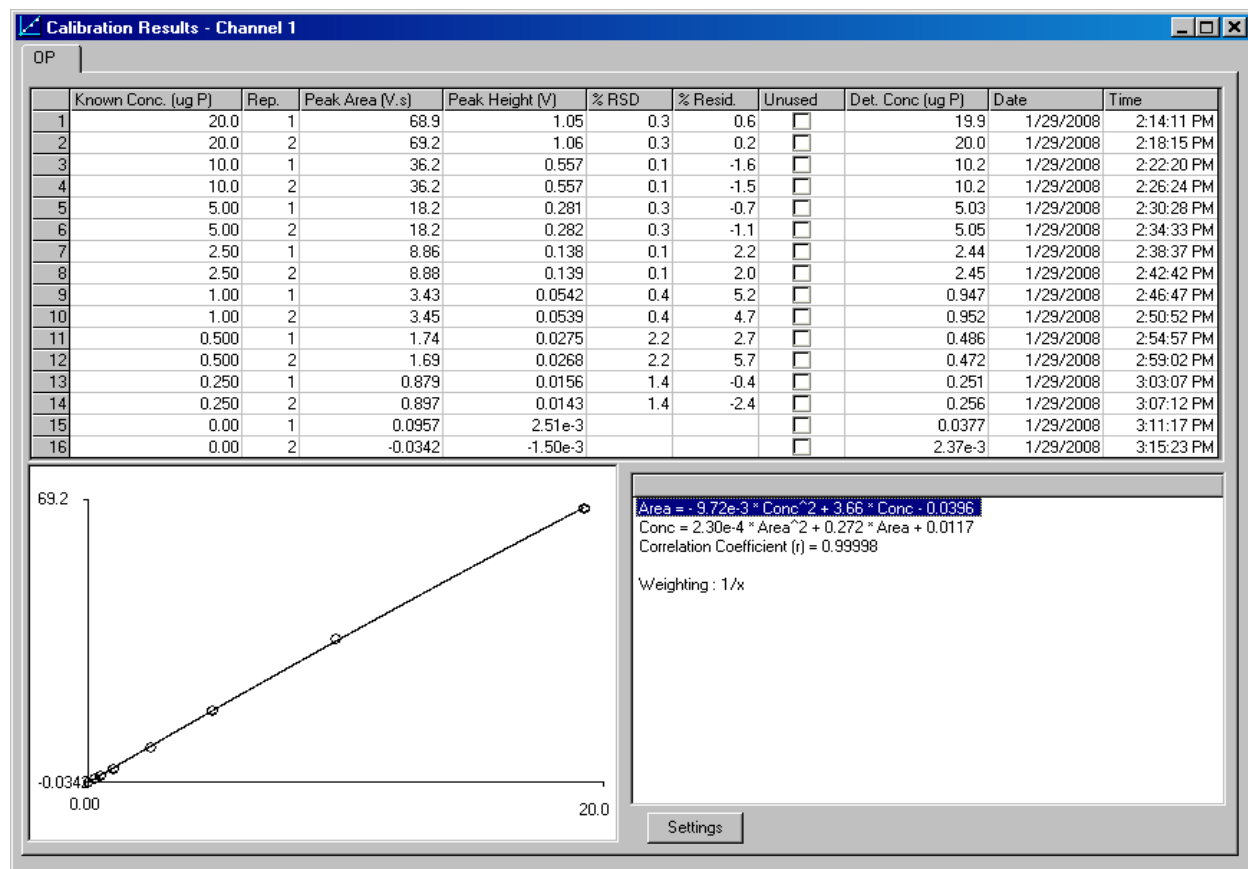
### Calibration Data for Orthophosphate

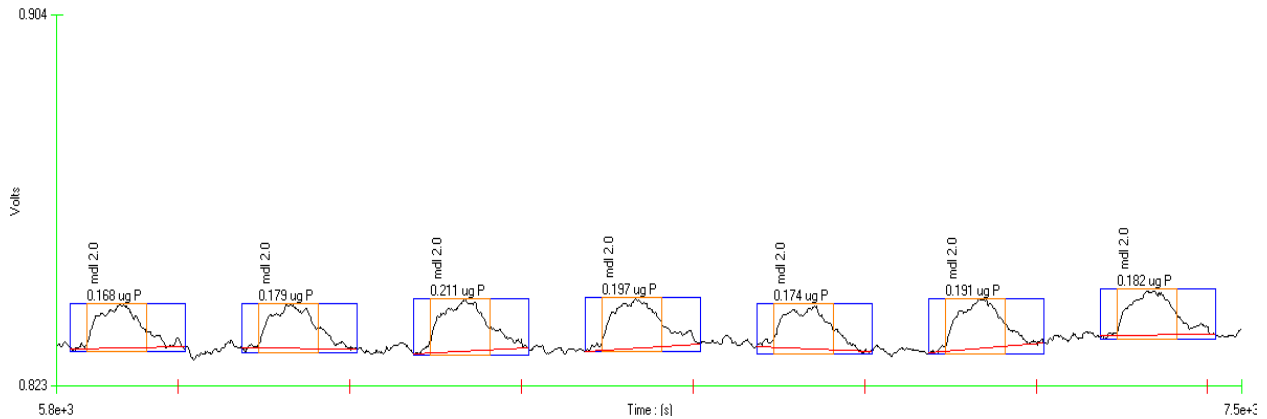


File Name: 1-29 cal support.omn

Acq. Date: 29 January 2008

### Calibration Graph and Statistics





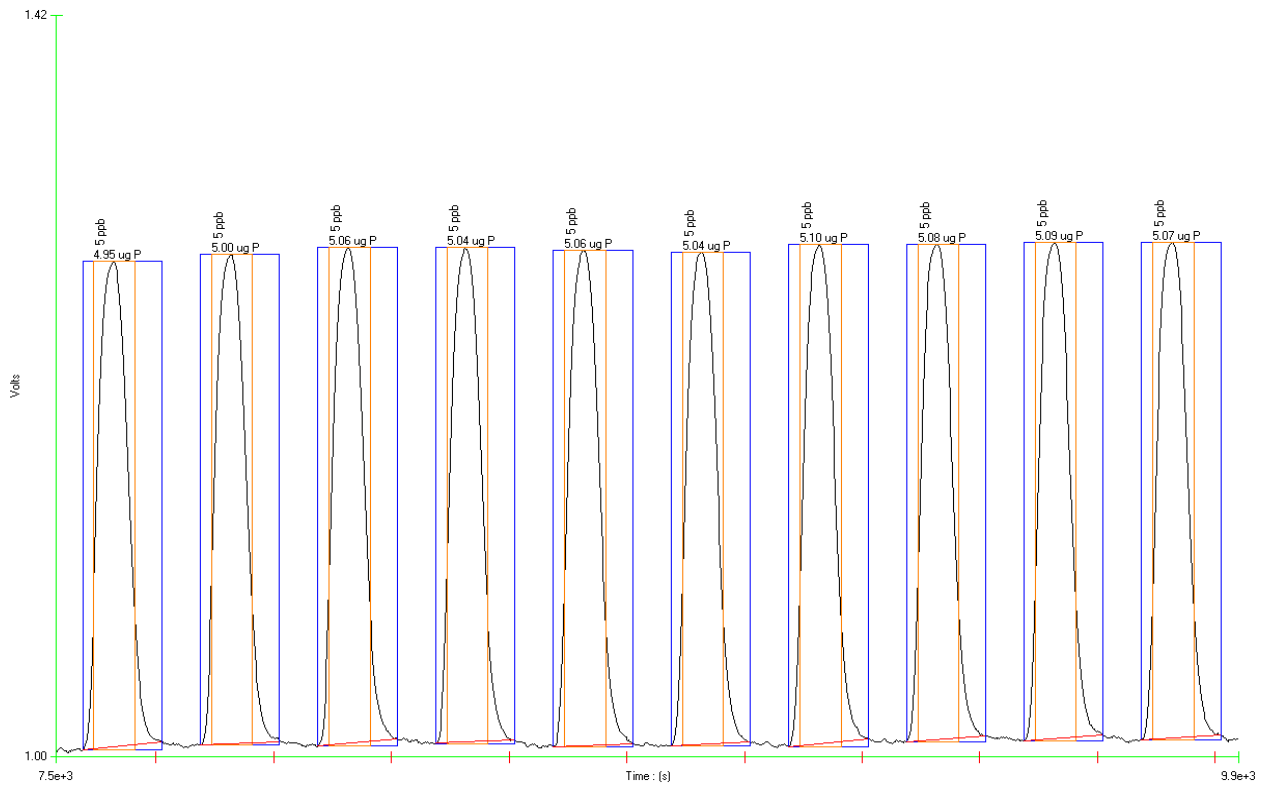
**Method Detection Limit for Orthophosphate using 0.20 µg P/L standard**

**MDL= 0.046 µg P/L**

Standard Deviation (s) = 0.0147 µg P/L, Mean (x) = 0.186 µg P/L, Known value = 0.20 µg P/L

File Name: 1-29 cal support.omn

Acq. Date: 29 January 2008



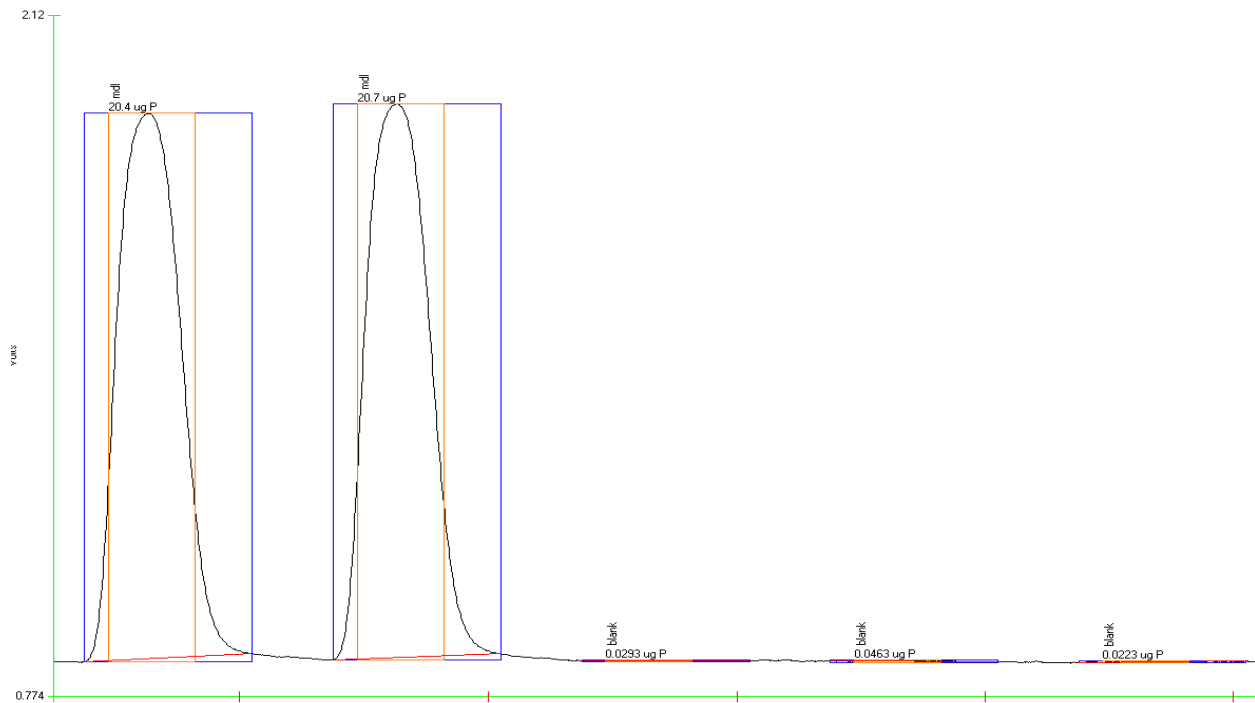
**Precision data for Orthophosphate using 5.0 µg P/L standard**

**% RSD = 0.87**

Standard Deviation (s) = 0.045 µg P/L, Mean (x) = 5.05 µg P/L, Known value = 5.0 µg P/L

File Name: 1-28 cal support.omn

Acq. Date: 28 January 2008

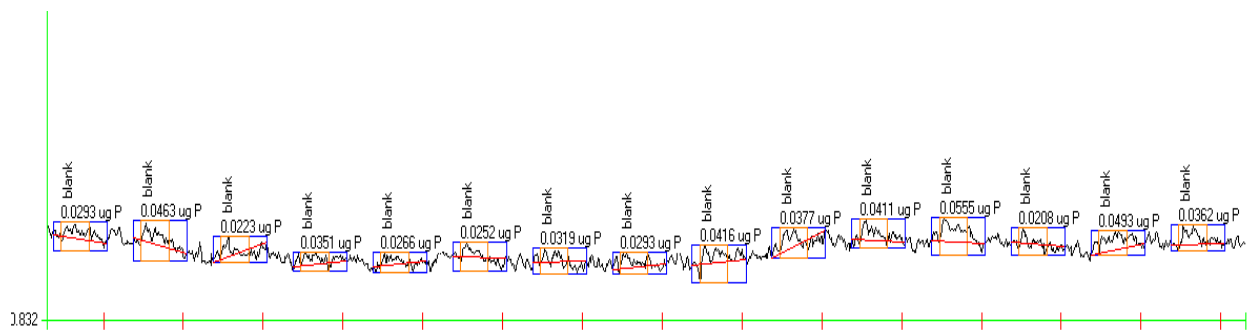


**Carryover Study: 20 µg P/L standard followed by 3 blanks**

Carryover Passed

File Name: 1-30LR spikes din.omn

Acq. Date: 30 January 2008



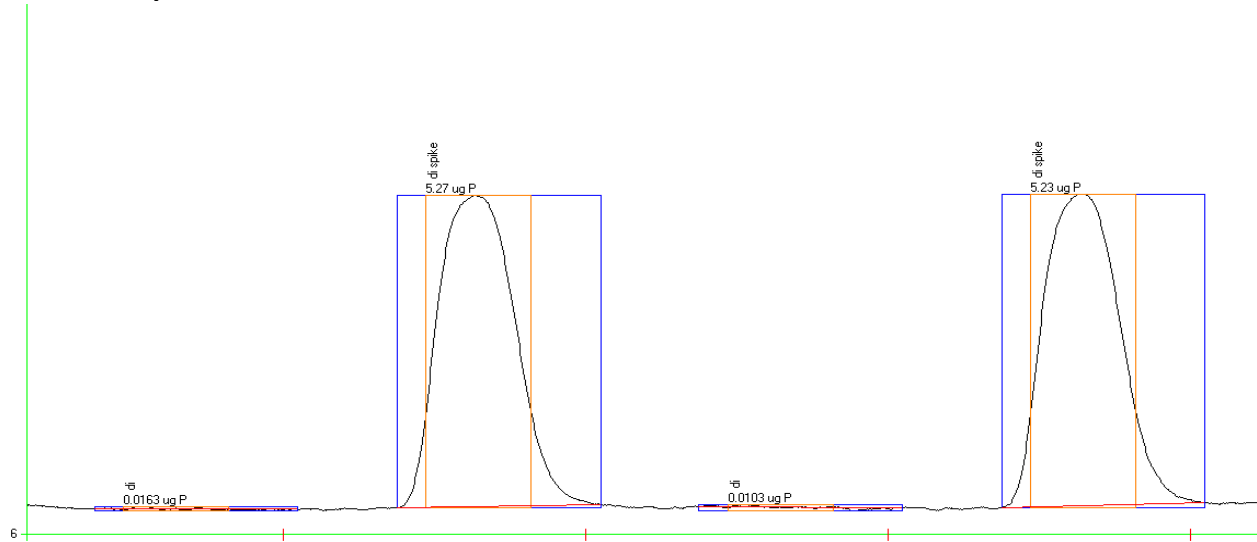
**DIN Blanks**

Average: 0.035µg P/L, SD = 0.010 µg P/L. Calculated DIN Limits: Detection Limit = 0.030 µg P/L, Decision Limit = 0.061 µg P/L, Determination Limit = 0.091 µg P/L

File Name: 1-30LR spikes din.omn

Acq. Date: 30 January 2008

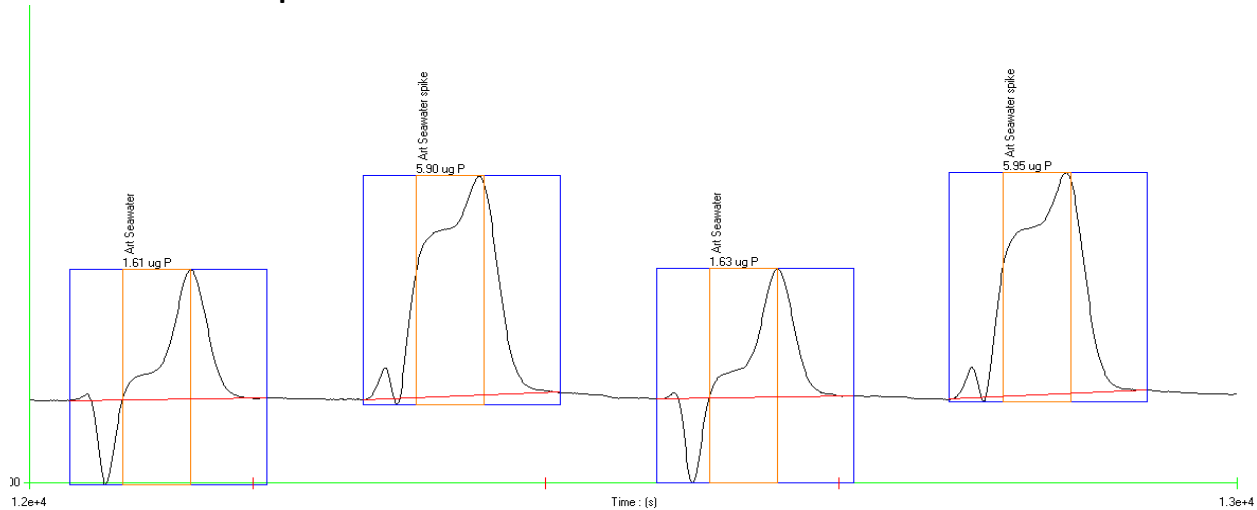
## DI Water Spikes



File Name: 1-30LR spikes din.omn  
Acq. Date: 30 January 2008

Initial ( $\mu\text{g P/L}$ )	Spiked ( $\mu\text{g P/L}$ )	Spike Level ( $\mu\text{g P/L}$ )	Spike Recovery
0.0163	5.27	5.0	105%
0.0103	5.23	5.0	104%

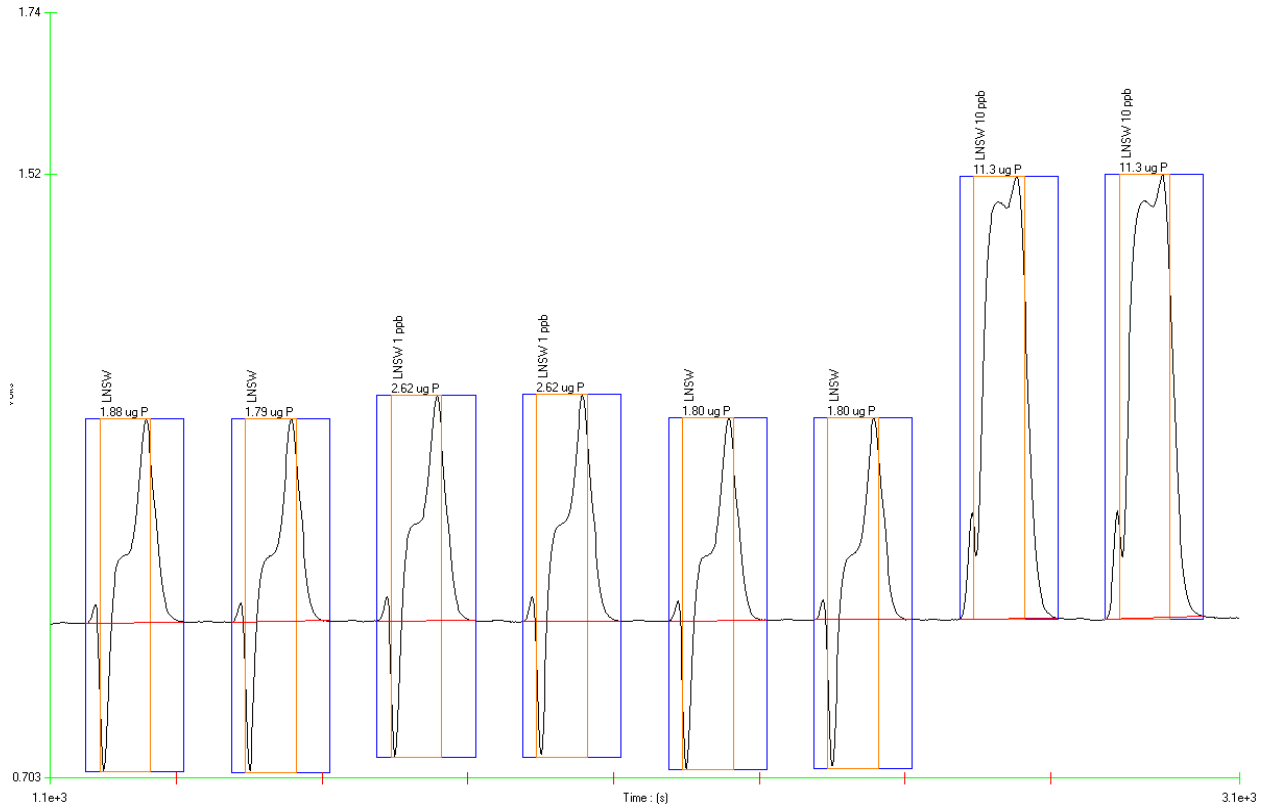
## Artificial Seawater Spikes



File Name: 1-30 seawater.omn  
Acq. Date: 30 January 2008

Initial ( $\mu\text{g P/L}$ )	Spiked ( $\mu\text{g P/L}$ )	Spike Level ( $\mu\text{g P/L}$ )	Spike Recovery
1.61	5.90	5.0	85.8%
1.63	5.95	5.0	86.4%

## Low Nutrient Seawater

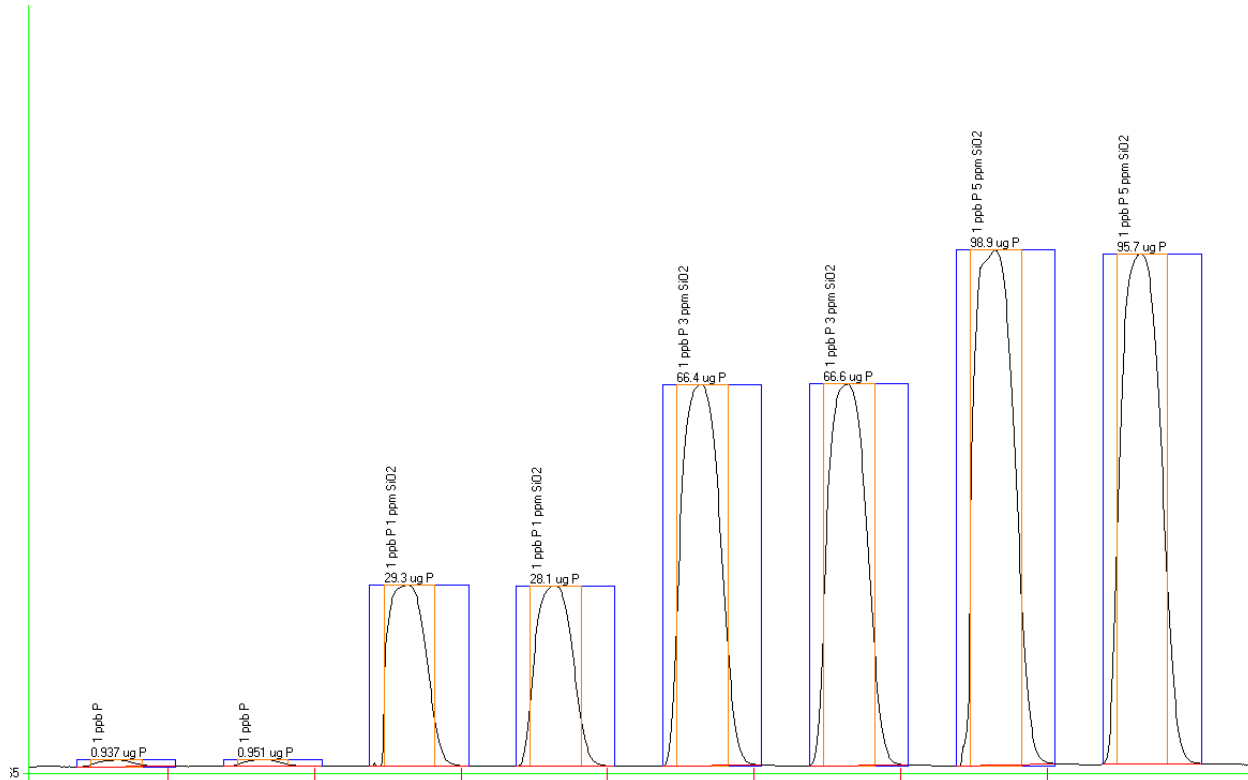


File Name: 2-25 LNSW spike.OMN  
Acq. Date: 25 February 2008

Initial avg ( $\mu\text{g P/L}$ )	Spiked avg ( $\mu\text{g P/L}$ )	Spike Level ( $\mu\text{g P/L}$ )	Spike Recovery
1.83	2.62	1.0	79%
1.80	11.3	10.0	95%

The Low Nutrient Seawater was purchased from Ocean Scientific International Ltd

## Interference Study using Phosphorus Standard Spiked with Silicate



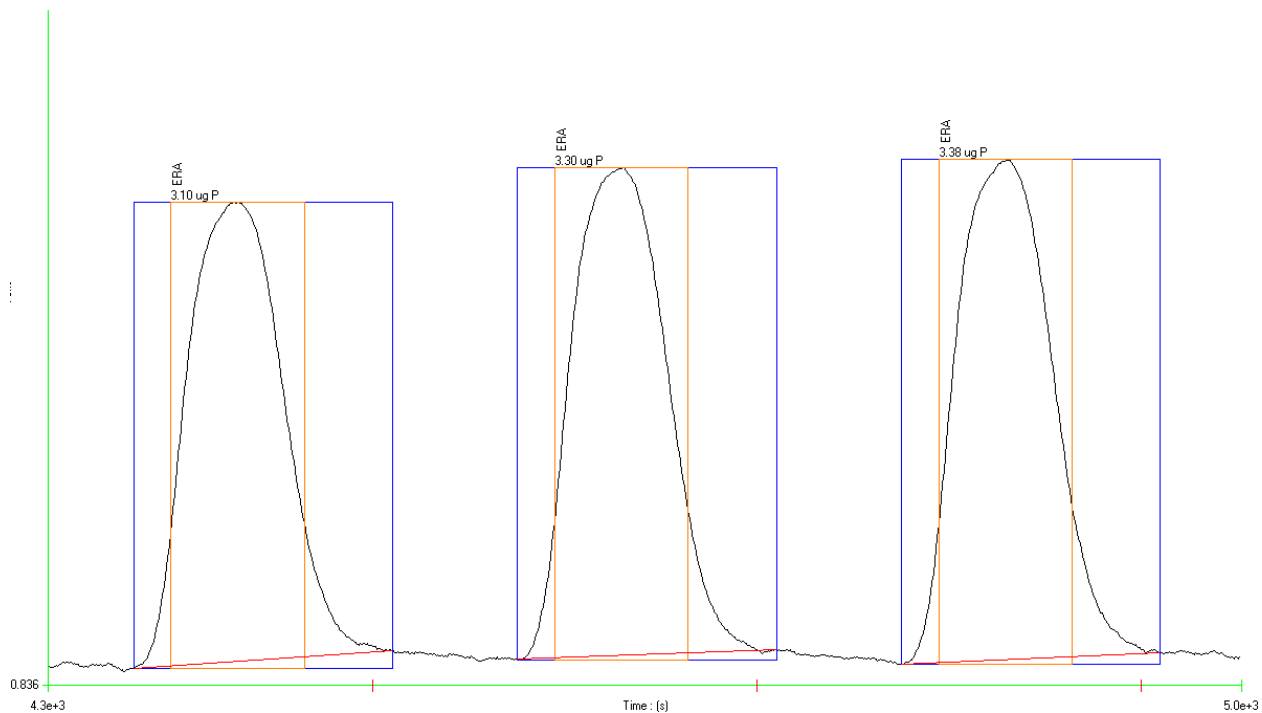
File Name: 1-28 SiO<sub>2</sub> interference.omn

Acq. Date: 28 January 2008

A 1 µg P/L standard was spiked with various amounts of silicate. The non-spiked and spiked standards were then run in duplicate to determine how silicate would affect phosphate quantitation. The following table summarizes the results obtained from this study.

Sample	Results µg P/L	Average µg P/L	Selectivity against Silicate (SiO <sub>2</sub> conc / P interference)
1 µg P/L	0.937 0.951	0.944	--
1 µg P/L + 1 mg SiO <sub>2</sub> /L	29.3 28.1	28.7	34.84
1 µg P/L + 3 mg SiO <sub>2</sub> /L	66.4 66.6	66.5	45.11
1 µg P/L + 5 mg SiO <sub>2</sub> /L	98.9 95.7	97.3	51.39

**Conclusion:** Silicate is an interferent with this method. If SiO<sub>2</sub> is suspected to be present, the concentration should be measured to be sure this will not be an interference in the method.



File Name: 1-30LR spikes din.omn  
 Acq. Date: 30 January 2008

ERA QC Standard WasteWatR Simple Nutrients, Catalog no.  
 505, Lot No. PO95505  
 ERA, Arvada, Colorado, US Phone: 303-431-8454  
 Mean Determined Concentration: **3.26 µg P/L**

**Known Concentration:** 3.22 mg P/L  
 Interlaboratory Acceptance Range: 2.79 – 3.53 mg P/L  
**Diluted known standard 1000 fold to produce 3.22 µg P/L**



**ENVIRONMENTAL  
 RESOURCE ASSOCIATES**  
 The Industry Standard™

# QuikChem® Method 31-115-01-1-Y and 10-115-01-1-Y

## Orthophosphate in Waters and Seawater

**0.50 to 100.0 µg P/L**

**For samples with silicate present at higher levels**

### **– Principle –**

The orthophosphate ion ( $\text{PO}_4^{3-}$ ) 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 can be significant when measuring P at these trace levels. A silicate concentration of approximately 5 mg  $\text{SiO}_2$ /L will produce approximately 7 µg P/L error in orthophosphate. Therefore, this method can be used for waters, seawaters and brackish waters with high silicate levels.
2. Concentrations of ferric iron ( $\text{Fe}^{3+}$ ) greater than 50 mg/L will cause a negative error due to precipitation of, and subsequent loss, of orthophosphate. Samples high in iron can be pretreated with sodium bisulfite to eliminate this interference. Treatment with bisulfite will also remove the interference due to arsenates.
3. 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 preparations for lab glassware.

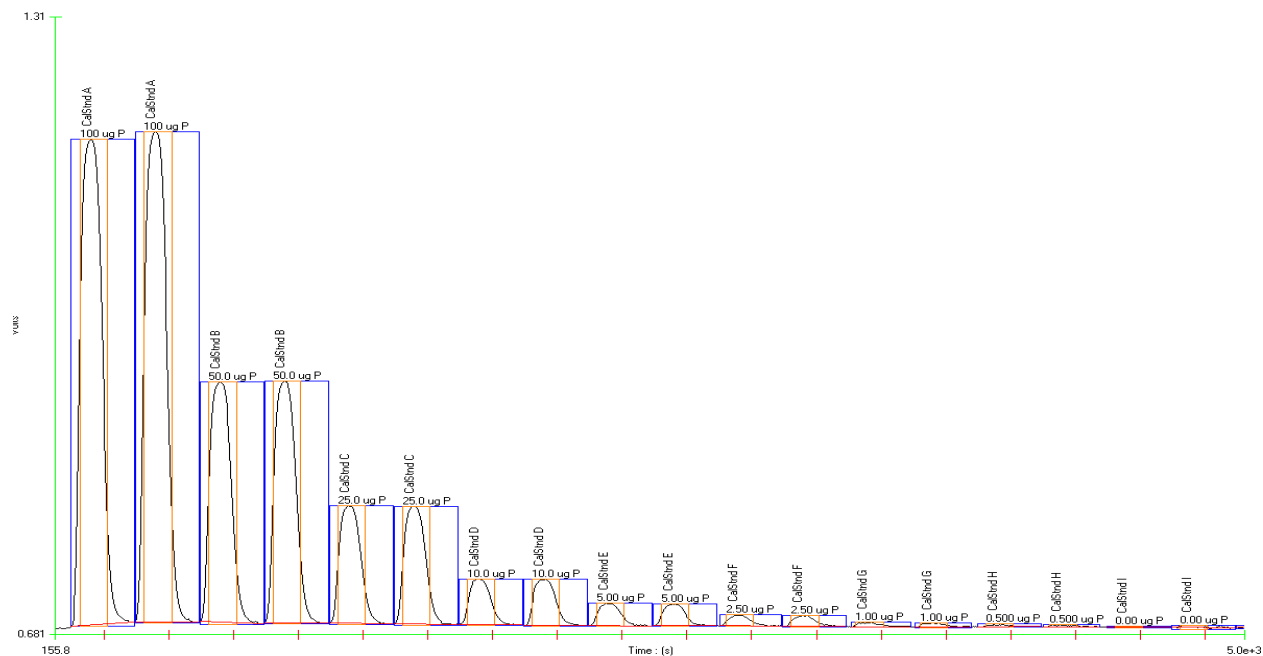
### **– Special Apparatus –**

Please contact Lachat Sales for ordering information

1. Heating Unit, Lachat Part No. A85X00 (X=1 for 110V, X=2 for 220V), with Hi Temp Insulator Tube (Lachat Part No. 85090)
2. 2-cm Detector Assembly [Lachat Part No. 58025 (Assembly includes 2 cm flow cell, Lachat Part No. 58062)]
3. Glass

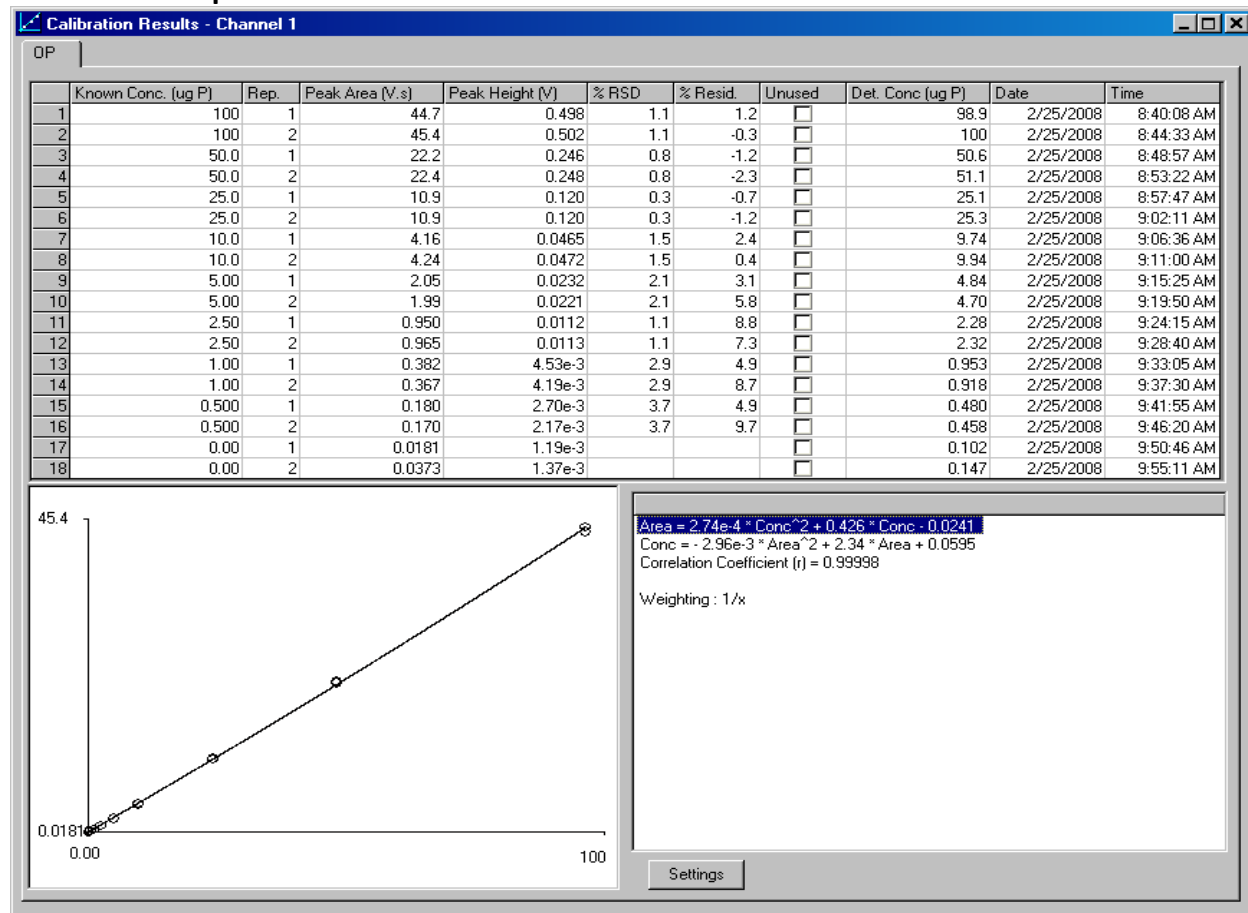
## – Support Data for QuikChem 8500 –

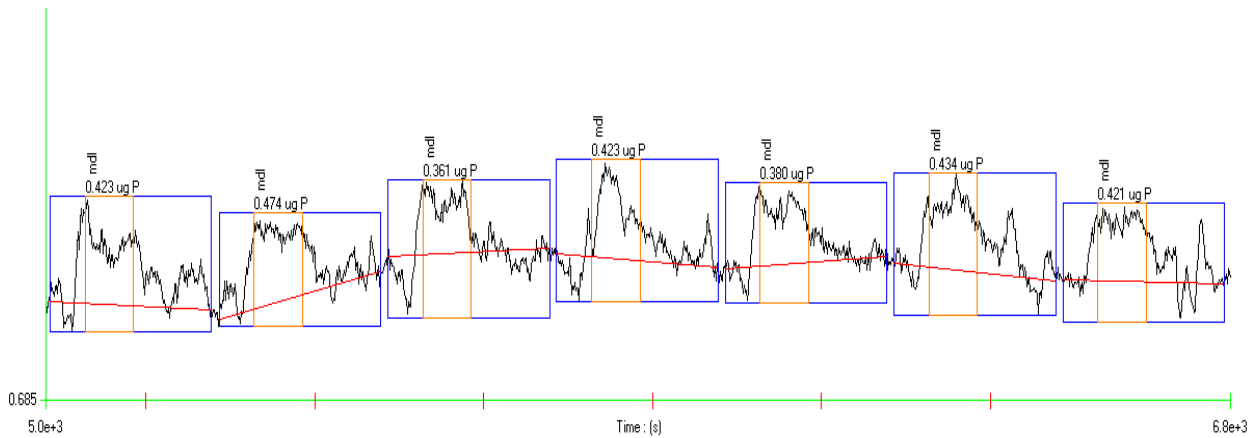
### Calibration Data for Orthophosphate



File Name: 2-25 cal.omn  
Acq. Date: 25 February 2008

### Calibration Graph and Statistics





### Method Detection Limit for Orthophosphate using 0.40 µg P/L standard

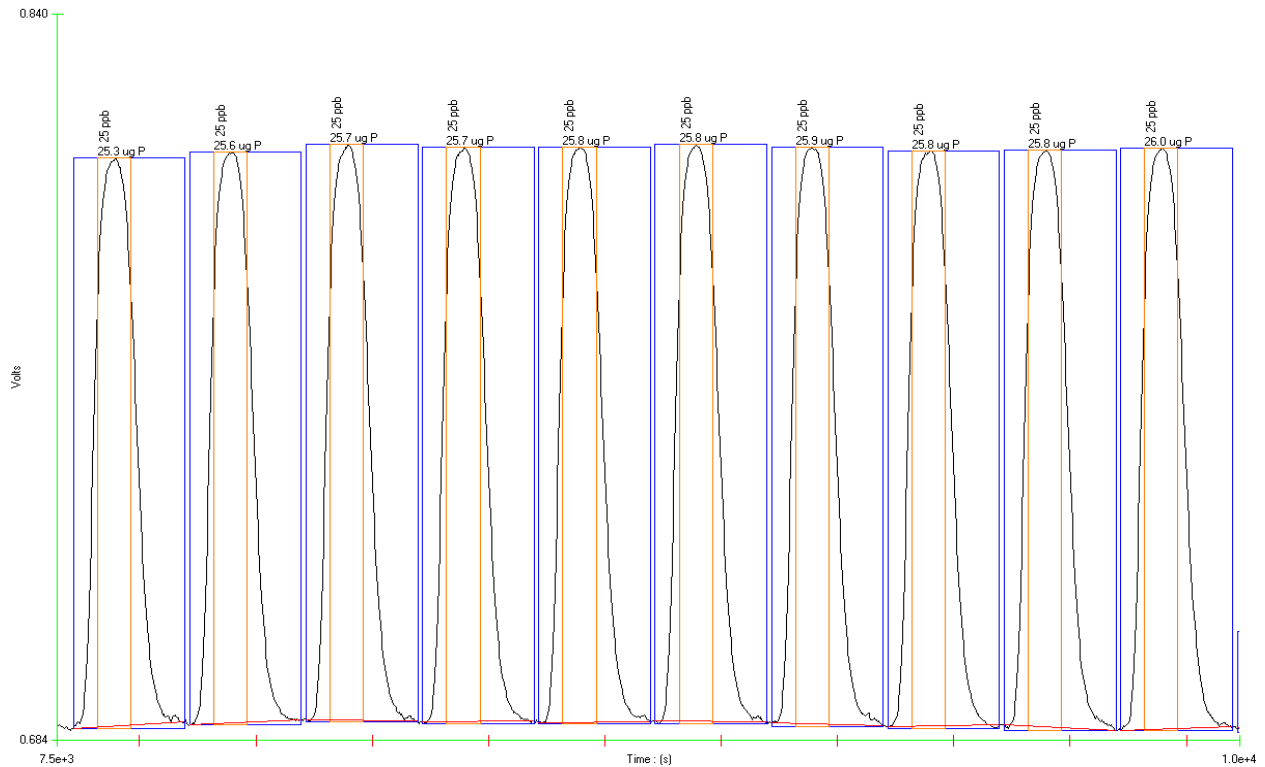
Calculated MDL = 0.116 µg P/L

Reporting MDL = 0.164 µg P/L due to blanks in the Carry Over Study produced values higher than the calculated MDL. The reported MDL is an average of the DIN blanks, see DIN Blanks below.

Standard Deviation (s) = 0.037 µg P/L, Mean (x) = 0.42 µg P/L, Known value = 0.40 µg P/L

File Name: 2-25 cal support.omn

Acq. Date: 25 February 2008



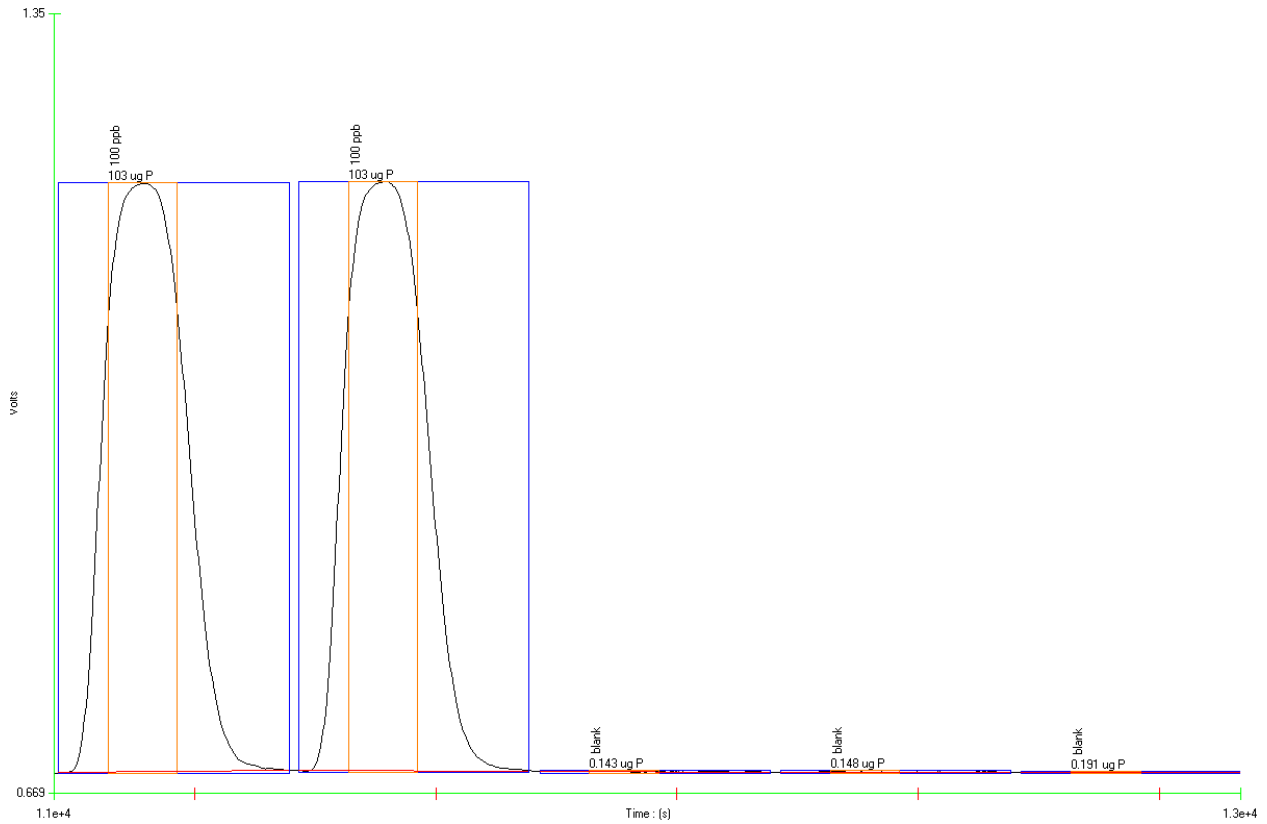
### Precision data for Orthophosphate using 25.0 µg P/L standard

% RSD = 0.74

Standard Deviation (s) = 0.190 µg P/L, Mean (x) = 25.74 µg P/L, Known value = 25.0 µg P/L

File Name: 2-25 cal support.omn

Acq. Date: 25 February 2008

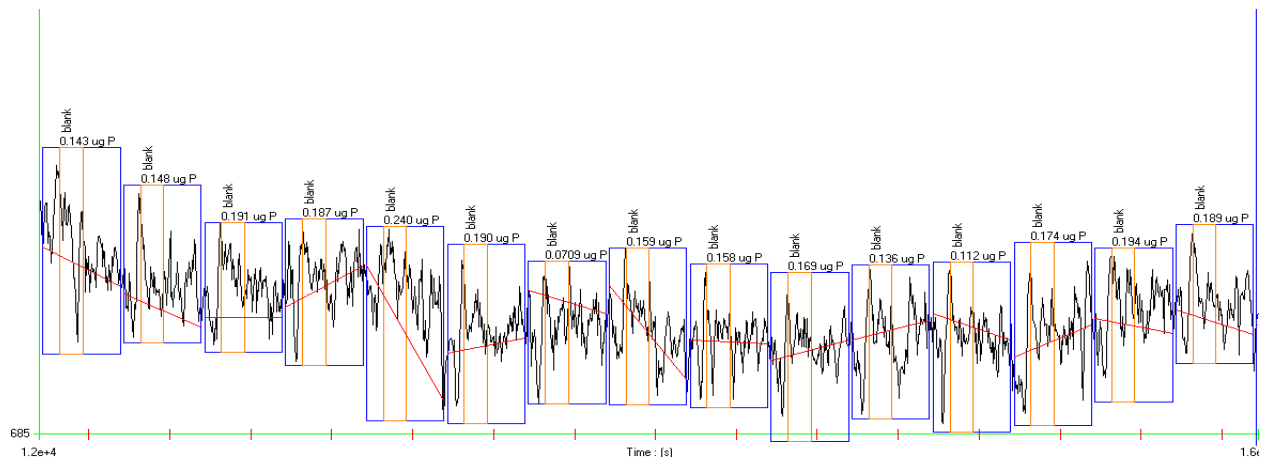


**Carryover Study: 100 µg P/L standard followed by 3 blanks**

Carryover Failed, blanks are higher than the calculated MDL, reporting the average of the DIN calculation of 0.164 µg P/L

File Name: 2-25 cal support.omn

Acq. Date: 25 February 2008



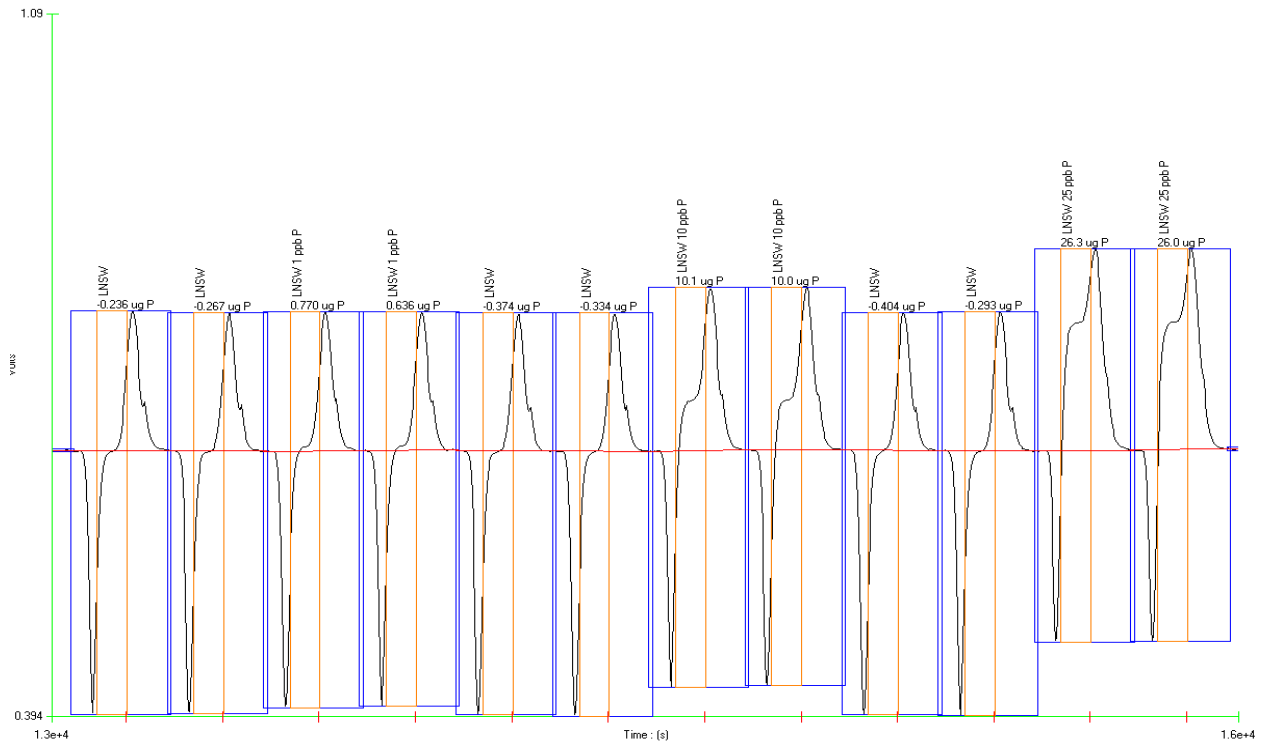
**DIN Blanks**

Average: 0.164 µg P/L, SD = 0.040 µg P/L. Calculated DIN Limits: Detection Limit = 0.120 µg P/L, Decision Limit = 0.239 µg P/L, Determination Limit = 0.359 µg P/L

File Name: 2-25 cal support.omn

Acq. Date: 25 February 2008

## Low Nutrient Seawater



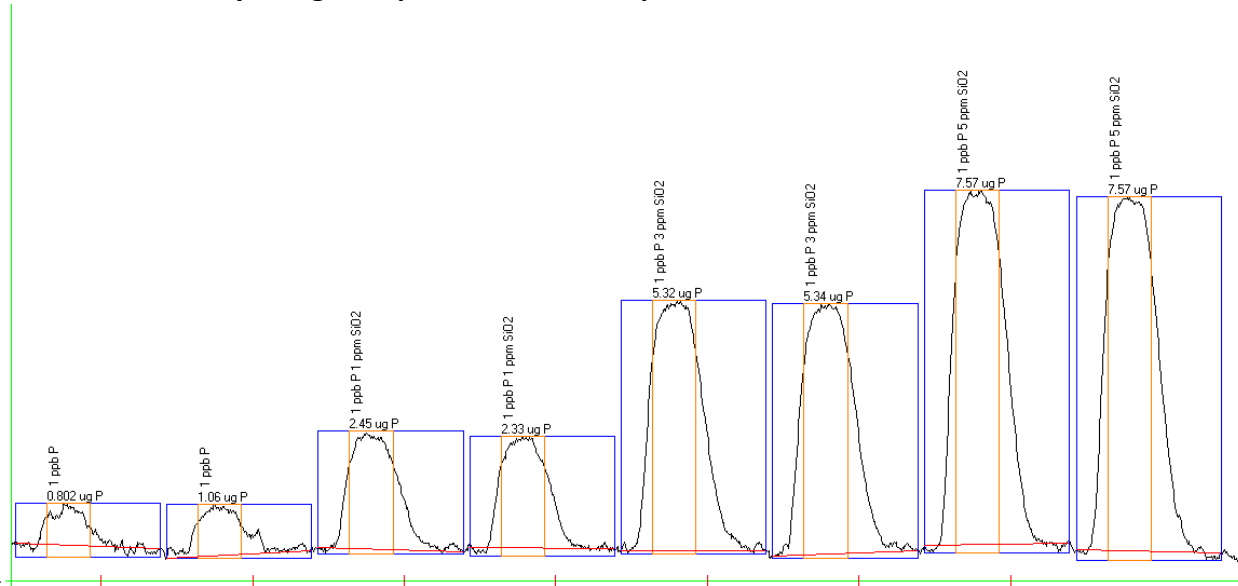
File Name: 2-22 seawater spikes.omn

Acq. Date: 22 February 2008

Initial avg ( $\mu\text{g P/L}$ )	Spiked avg ( $\mu\text{g P/L}$ )	Spike Level ( $\mu\text{g P/L}$ )	Spike Recovery
-0.252	0.703	1.00	95.5%
-0.354	10.05	10.0	104.3%
-0.348	26.15	25.0	105.9%

The Low Nutrient Seawater is supplied by Ocean Scientific International Ltd

## Interference Study using Phosphorus Standard Spiked with Silicate

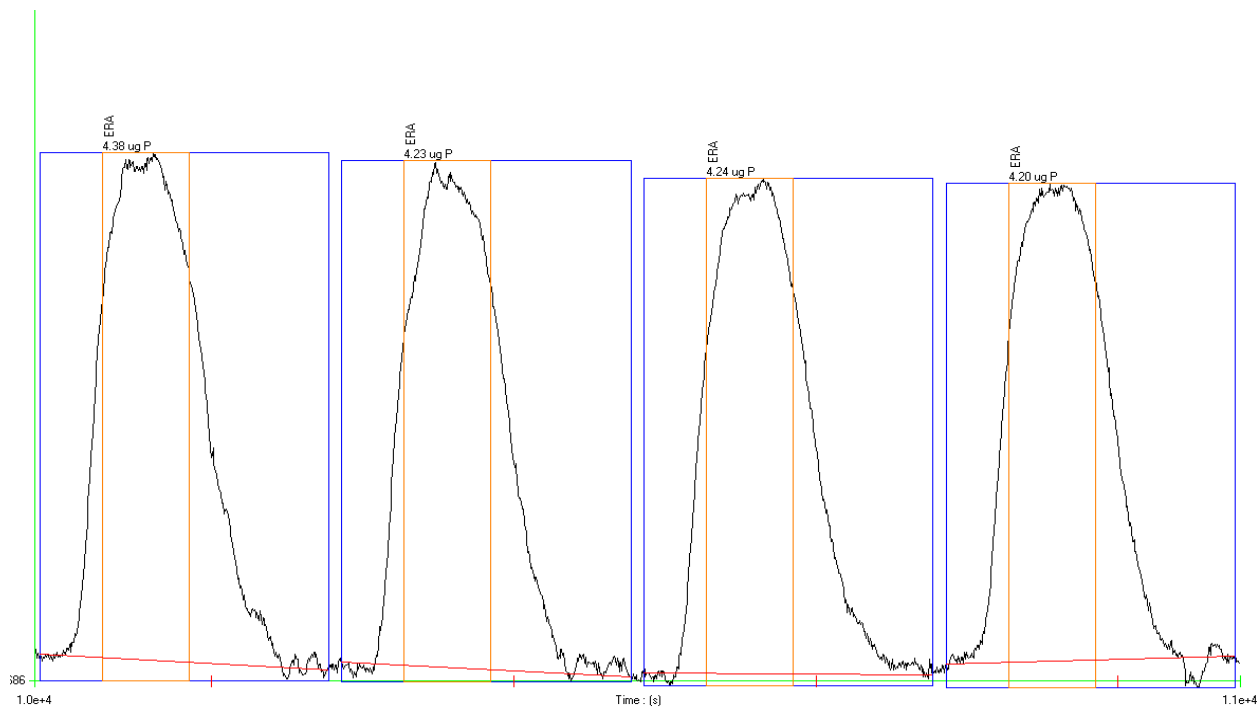


File Name: 2-25 cal support.omn  
Acq. Date: 25 February 2008

A 1 µg P/L standard was spiked with various amounts of silicate. The non-spiked and spiked standards were then run in duplicate to determine how silicate would affect phosphate quantitation. The following table summarizes the results obtained from this study.

Sample	Results µg P/L	Average µg P/L	Selectivity against Silicate (SiO <sub>2</sub> conc / P interference)	% Error
1 µg P/L	0.802 1.06	0.932	--	--
1 µg P/L + 1 mg SiO <sub>2</sub> /L	2.45 2.33	2.39	418	156
1 µg P/L + 3 mg SiO <sub>2</sub> /L	5.32 5.34	5.33	563	472
1 µg P/L + 5 mg SiO <sub>2</sub> /L	7.57 7.57	7.57	660	712

**Conclusion:** Silicate is an interferent with this method, which is not as selective as other ultra low range orthophosphate methods. The SiO<sub>2</sub> concentration should be measured if phosphorus spikes produce high recoveries.



File Name: 2-25 cal support.omn  
 Acq. Date: 25 February 2008

ERA QC Standard WasteWatR Simple Nutrients, Catalog no.  
 505, Lot No. P148-505  
 ERA, Arvada, Colorado, US Phone: 303-431-8454  
 Mean Determined Concentration: **4.26 µg P/L**



**ENVIRONMENTAL  
 RESOURCE ASSOCIATES**  
 The Industry Standard™

**Known Concentration:** 4.32 mg P/L  
 Interlaboratory Acceptance Range: 3.97 – 4.93 mg P/L  
 Diluted known standard 1000 fold to produce 4.32 µg P/L

# Silicate

QuikChem® Method 31-114-27-1-E and 10-114-27-1-C

## Silicate in Waters and Brackish Waters

2.50 to 100  $\mu\text{g SiO}_2/\text{L}$

### – Principle –

Silicate reacts with molybdate under acidic conditions to form yellow beta molybdosilicic acid. This acid is subsequently reduced with stannous chloride to form a heteropoly blue complex which has an absorbance maximum at 820 nm. Oxalic acid is added to reduce the interference from phosphate.

### – Interferences –

1. The interference due to phosphate is reduced by the addition of oxalic acid. A solution of 1000  $\mu\text{g P/L}$  was determined as 20  $\mu\text{g SiO}_2/\text{L}$ . The extent of phosphate interference should be verified by determining a solution of phosphate at the highest concentration that is expected to be encountered. If the 7 cm reaction coil after the oxalic acid does not sufficiently reduce phosphate interference, a longer coil can be used.
2. Tannin and large amounts of iron or sulfides are interferences. Sulfides can be removed by boiling and acidifying the sample. Addition of disodium EDTA will eliminate the interference due to iron. Treatment with oxalic acid decreases interference from tannin.
3. Sample color and turbidity can interfere. The presence of these interferences can be determined by analyzing samples without the presence of molybdate.
4. Silica contamination may be avoided by storing samples, standards, and reagents in plastic. Deionize glass-distilled water before use to remove silica.

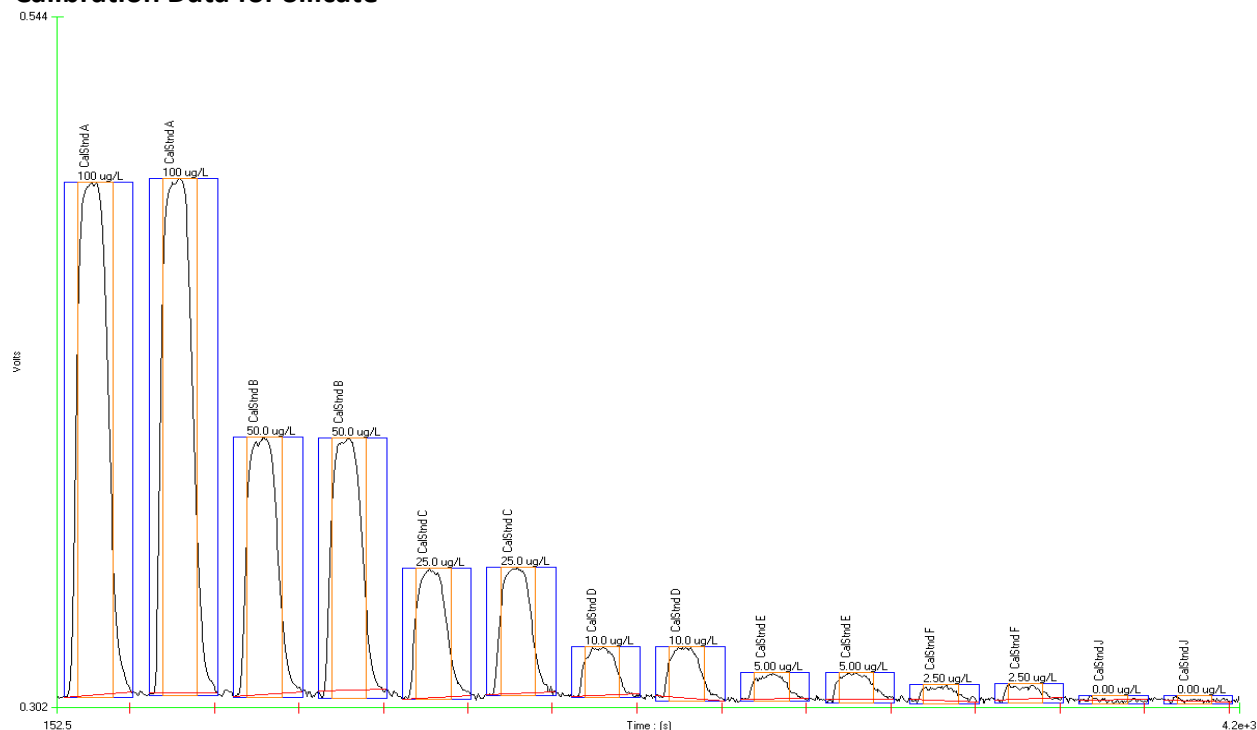
### – Special Apparatus –

Please contact Lachat Sales for ordering information

1. Heating Unit Lachat Part No. A85X00 (X=1 for 110V, X=2 for 220V)
2. Plastic sample (PN 21042) and calibration vials (PN 21409) must be used with this method.
3. Glass line weights must NOT be used with this method.
4. 2-cm Detector Assembly [Lachat Part No. 58025 (Assembly includes 2 cm flow cell, Lachat Part No. 58062)]

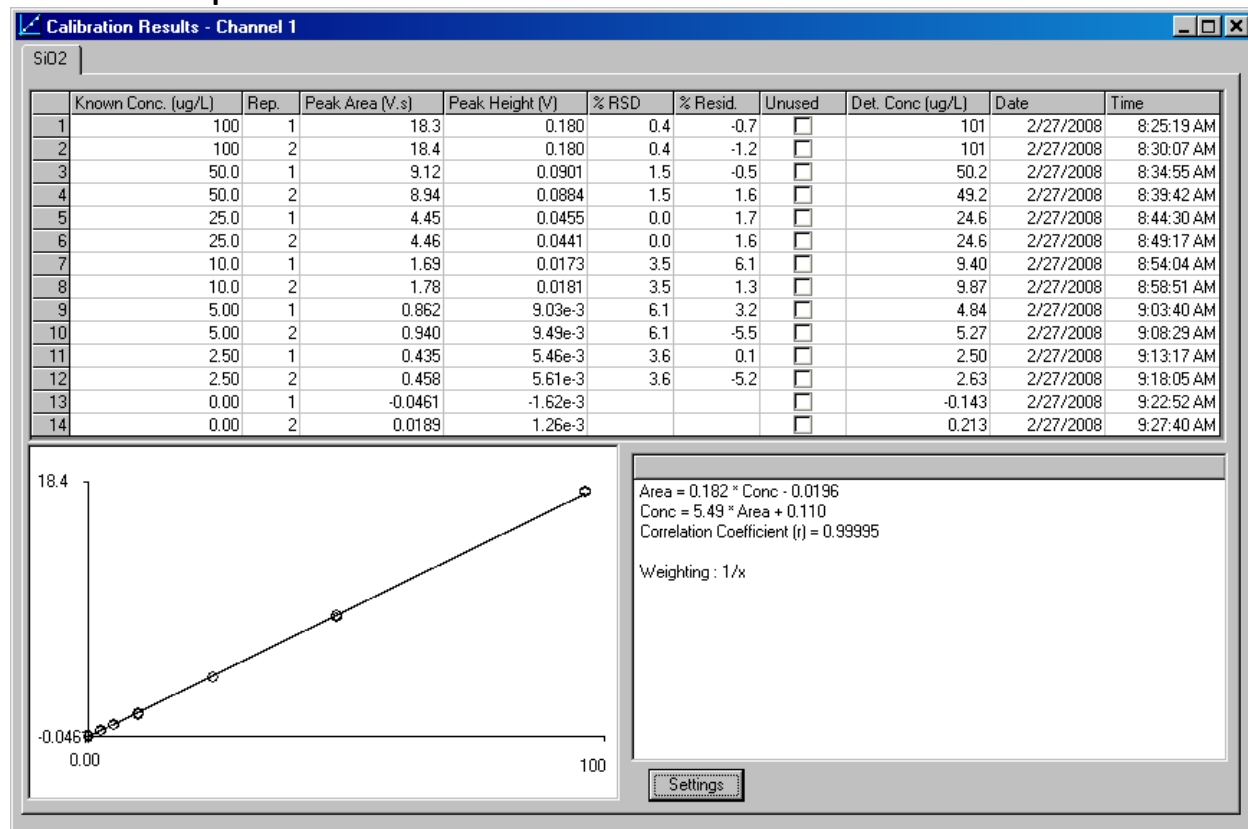
## – Support Data for QuikChem 8500 –

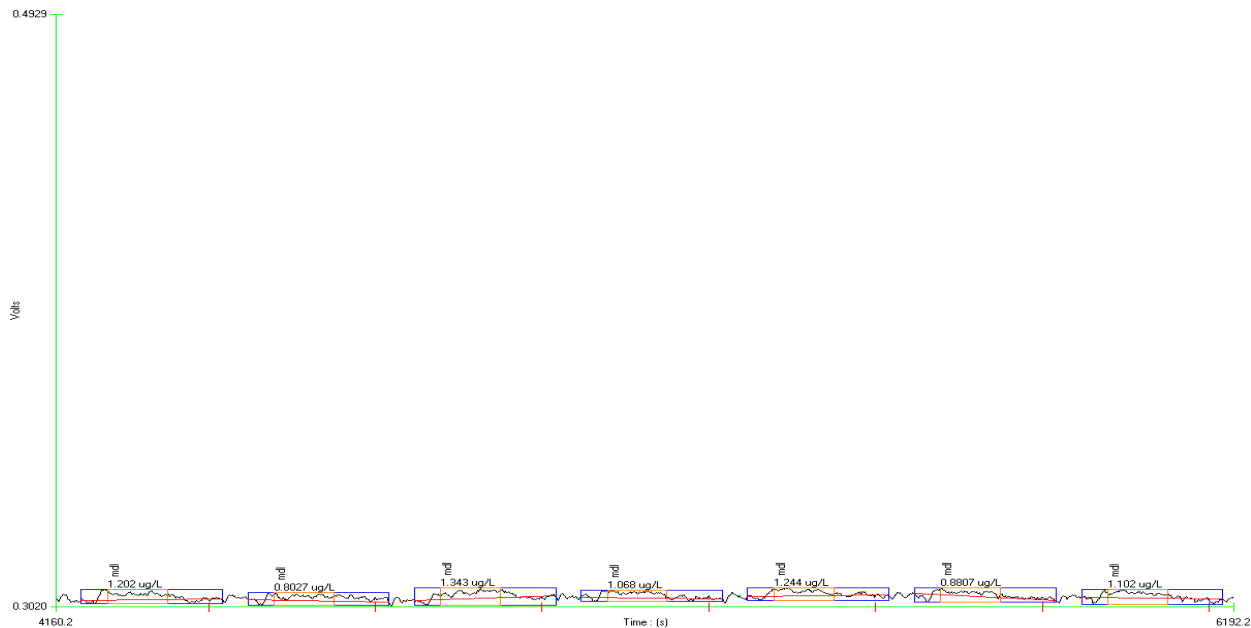
### Calibration Data for Silicate



File name: 2-27 cal.omn  
Acq. Date: 27 February 2008

### Calibration Graph and Statistics





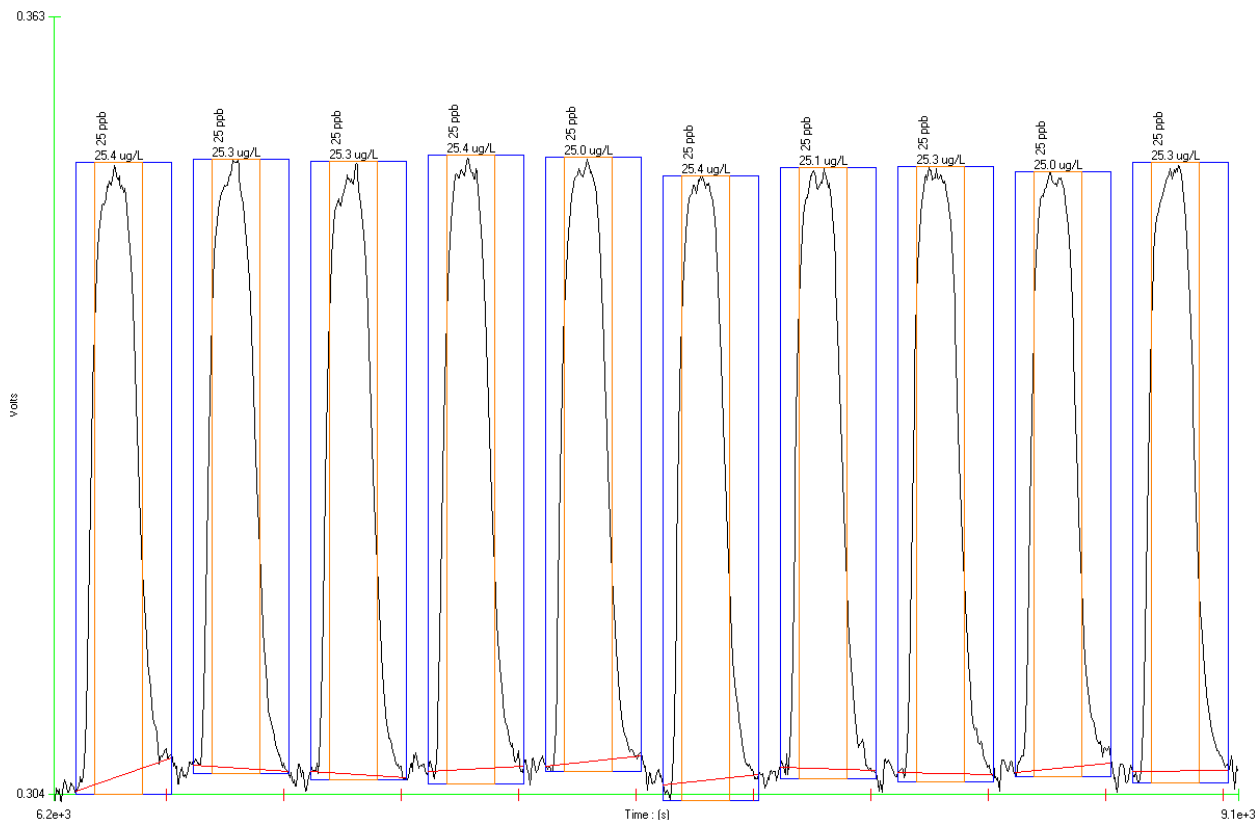
**Method Detection Limit for silicate using 1.0 µg SiO<sub>2</sub>/L standard**

**MDL= 0.606 µg SiO<sub>2</sub>/L**

Standard Deviation (s) = 0.193 µg SiO<sub>2</sub>/L, Mean (x) = 1.09 µg SiO<sub>2</sub>/L, Known value = 1.0 µg SiO<sub>2</sub>/L

File name: 2-27 support.omn

Acq. Date: 27 February 2008



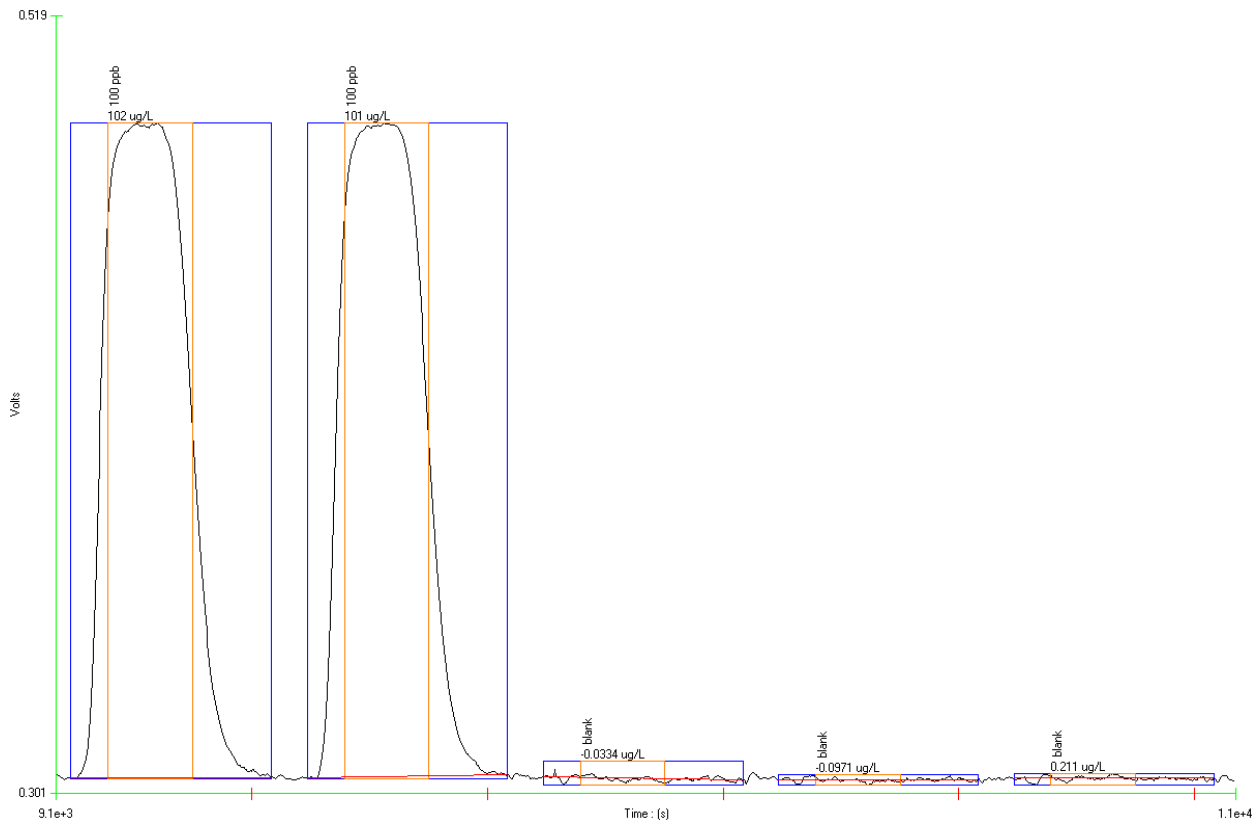
**Precision data for silicate using 25.0 µg SiO<sub>2</sub>/L standard**

**% RSD = 0.63%**

Standard Deviation (s) = 0.158 µg SiO<sub>2</sub>/L, Mean (x) = 25.25 µg SiO<sub>2</sub>/L, Known value = 25.0 µg SiO<sub>2</sub>/L

File name: 2-27 cal.omn

Acq. Date: 27 February 2008

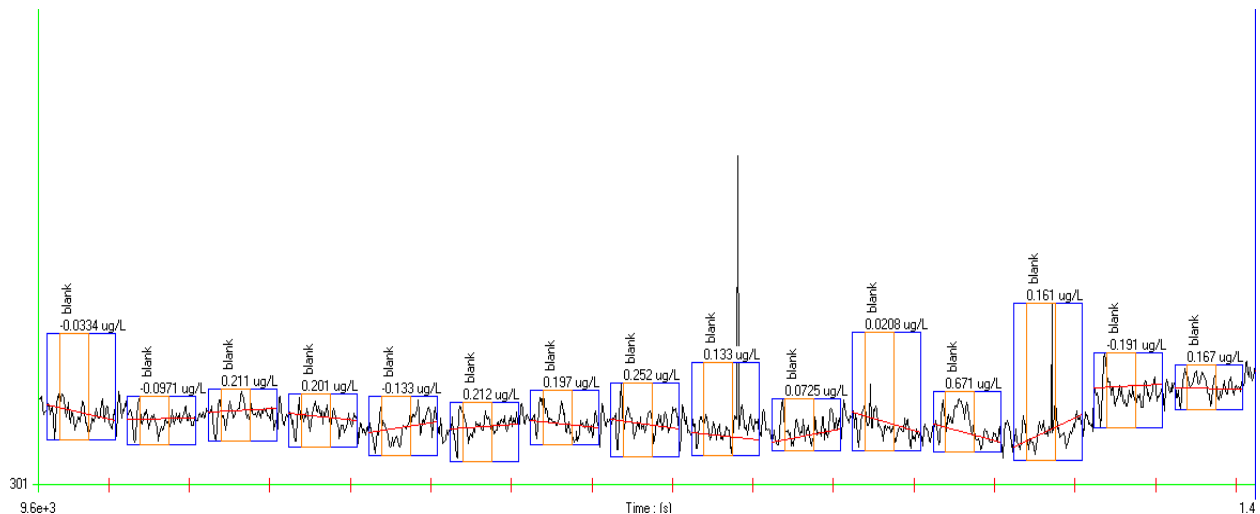


**Carryover Study: 2 replicates of  $\mu\text{g SiO}_2/\text{L}$  standard followed by 3 blanks**

Carryover Passed

File name: 2-27 support.omn

Acq. Date: 27 February 2008



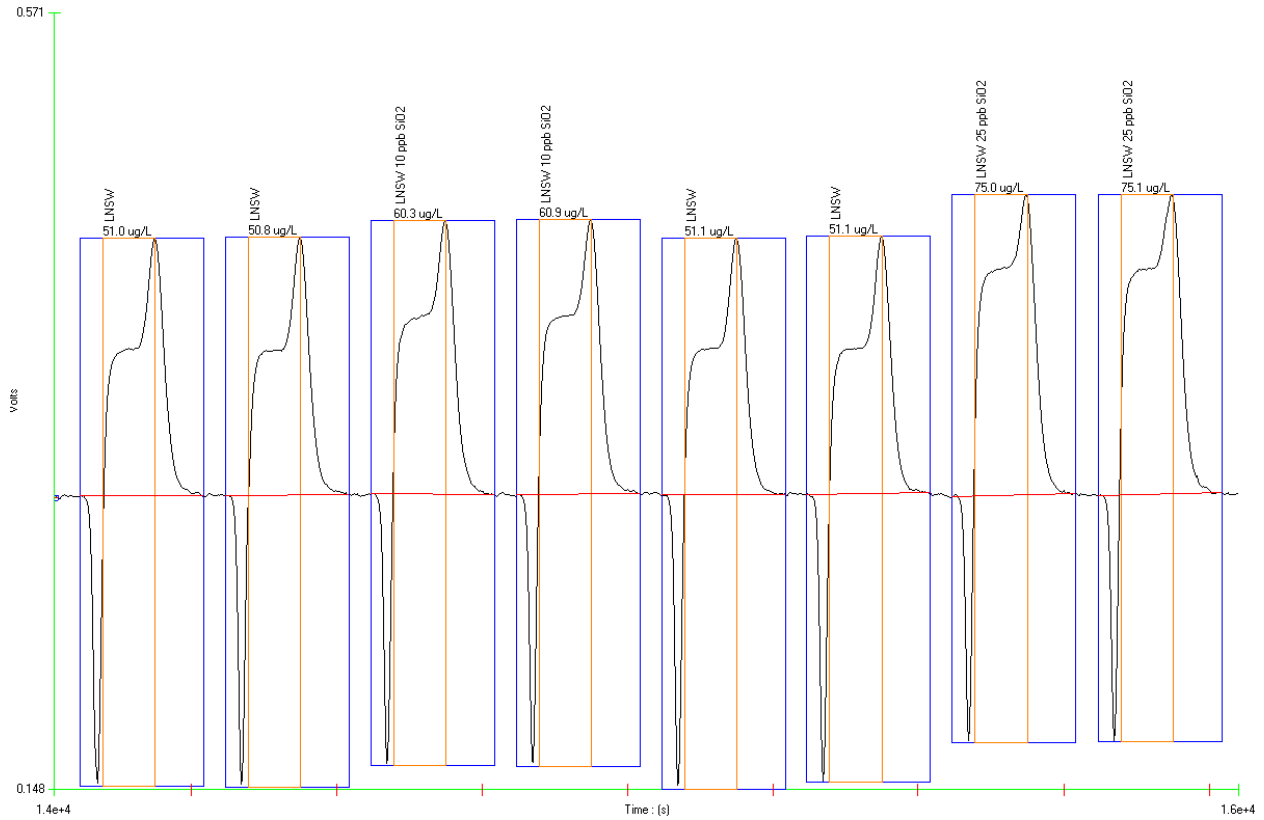
**DIN Blanks**

Average:  $0.123 \mu\text{g SiO}_2/\text{L}$ , SD =  $0.207 \mu\text{g SiO}_2/\text{L}$ . Calculated DIN Limits: Detection Limit =  $0.62 \mu\text{g SiO}_2/\text{L}$ , Decision Limit =  $1.24 \mu\text{g SiO}_2/\text{L}$ , Determination Limit =  $1.86 \mu\text{g SiO}_2/\text{L}$ ;

File name: 2-27 support.omn

Acq. Date: 27 February 2008

### Low Nutrient Seawater SiO<sub>2</sub> Spikes

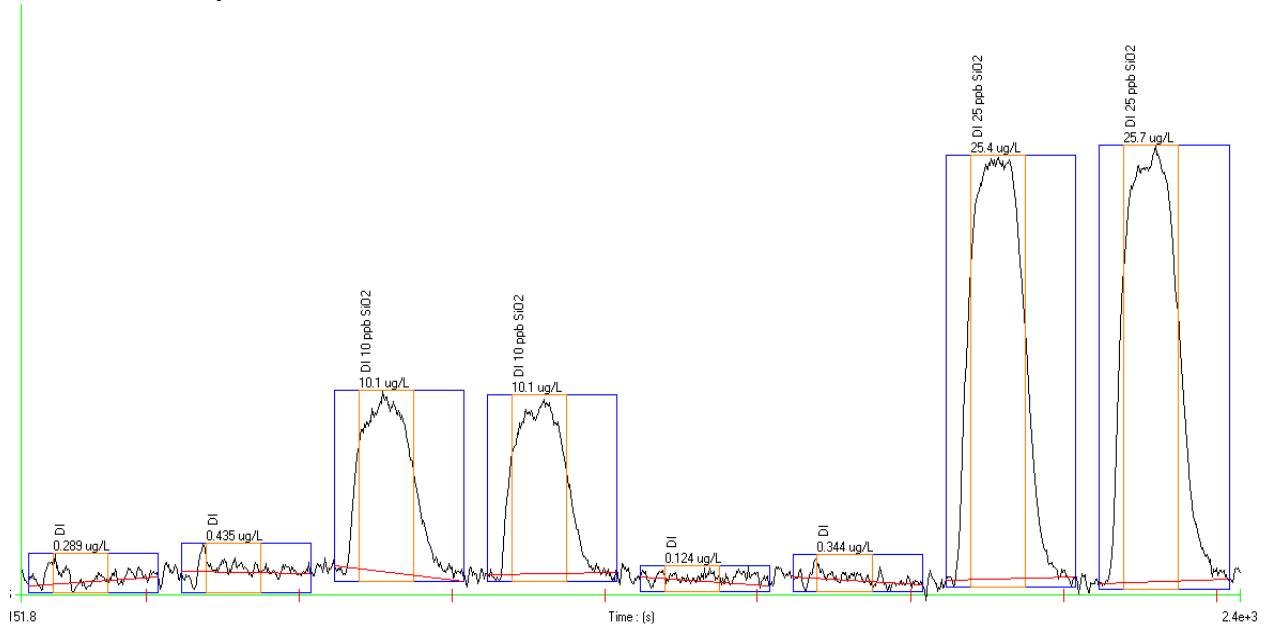


File name: 2-27 support.omn  
 Acq. Date: 27 February 2008

Initial avg ( $\mu\text{g SiO}_2/\text{L}$ )	Spiked avg ( $\mu\text{g SiO}_2/\text{L}$ )	Spike Level ( $\mu\text{g SiO}_2/\text{L}$ )	Spike Recovery
50.9	60.6	10.0	97.0%
51.1	75.0	25.0	95.6%

The Low Nutrient Seawater is supplied by Ocean Scientific International Ltd

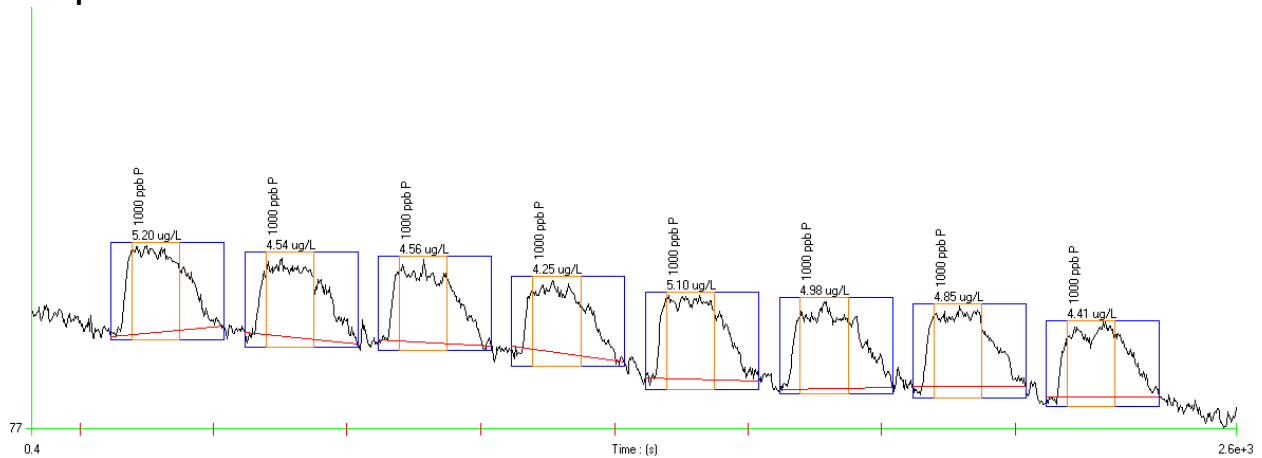
## DI Water SiO<sub>2</sub> Spikes



File name: 2-28 spikes.omn  
Acq. Date: 28 February 2008

Initial avg ( $\mu\text{g SiO}_2/\text{L}$ )	Spiked avg ( $\mu\text{g SiO}_2/\text{L}$ )	Spike Level ( $\mu\text{g SiO}_2/\text{L}$ )	Spike Recovery
0.362	10.1	10.0	97.1%
0.234	25.6	25.0	101.3%

## Phosphorus Interference



File name: 2-28 P interference.omn  
Acq. Date: 28 February 2008

**Conclusion:** 1000  $\mu\text{g P/L}$  standard produced an average of 4.7  $\mu\text{g SiO}_2$  interference, based on 8 phosphorus standard injections.

