

# *Data Pack*

*Lachat Applications in Standard Methods 21st Edition*

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*December 2008*



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# Introduction

The Lachat Instruments Applications team has developed many methods contained in Standard Methods, 21<sup>st</sup> Edition. Lachat recognizes Standard Methods as an important regulatory consensus body and has actively worked with this organization to develop methods that meet the standards of APHA, AWWA, WEF, and other internationally recognized authorities on water and wastewater quality.

This Data Pack provides an example of the available application information for each of the parameters in which Lachat has developed methods contained in Standard Methods, 21<sup>st</sup> Edition. A comprehensive list of all Lachat Methods is available upon request (see contact information below).

Lachat recommends that users always check with their local regulatory authority to ensure a method meets their individual reporting requirements.

For technical assistance, custom method development requests, 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)

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# Inorganic Anions by Ion Chromatography

**Lachat QuikChem method number 10-510-00-1-A**  
(SM 4110 B) Ion Chromatography with Chemical Suppression of Eluent Conductivity

## Inorganic Anions in Waters and Solid Extracts

USEPA 300.0 Part A

0.05 to 5 mg F<sup>-</sup>/L

0.50 to 50 mg Cl<sup>-</sup>/L

0.05 to 5 mg NO<sub>2</sub><sup>-</sup>-N/L

0.05 to 5 mg Br<sup>-</sup>/L

0.05 to 5 mg NO<sub>3</sub><sup>-</sup>-N/L

0.05 to 5 mg HPO<sub>4</sub><sup>2-</sup>-P/L

1.0 to 100 mg SO<sub>4</sub><sup>2-</sup>/L

### – Principle –

This is a chemically-suppressed, ion chromatography (IC) method. The anions are separated based on their affinities toward the stationary phase in the column. A suppressor cartridge, packed with high-capacity cation exchange resin, chemically suppresses the background conductance of the eluent and, at the same time, converts the anionic species into species of higher conductance. The method provides results that are equivalent to those of USEPA method 300.0 Part A, Standard Methods 4110 B, and those of ISO methods 10304-1 and 10304-2.

### – Interferences –

See Section 4 of the method for information on interferences.

### – QuikChem Method 10-510-00-1-A –

#### DETERMINATION OF ANIONS IN WATERS AND SOLID EXTRACTS

##### 1. SCOPE AND APPLICATION

1.1 The method covers the determination of the following inorganic anions:

Bromide	Nitrite-N
Chloride	Ortho-Phosphate-P
Fluoride	Sulfate
Nitrate-N	

1.2 The matrices applicable to this method are as follows:

Drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, solids (using the extraction procedure given in section 8.5), and leachates (when no acetic acid is used).

1.3 The single laboratory Method Detection Limits (MDL defined in Section 3.10) for the above analytes are listed in Table 1, Section 17.3.1 of this method. The MDL for a specified matrix may differ from those listed, depending upon the nature of the sample.

1.4 The range tested for each anion is as follows:

Analyte	Range, mg/L
bromide, fluoride, nitrite-N, nitrate-N, $\sigma$ -phosphate-P	0.05 to 5
chloride	0.5 to 50
sulfate	1 to 100

**Note:** The calibrated range for this method is two orders of magnitude. These ranges were based on a 100  $\mu$ L sample loop.

1.5 This method is recommended for use only by analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatogram.

1.6 When this method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of a fortified sample matrix covering the anions of interest. The fortification procedure is described in Section 9.

1.7 Bromide and nitrite react with most oxidants employed as disinfectants for drinking waters. The utility of measuring these anions in treated water should be considered prior to conducting the analysis.

1.8. Users of the method data should state the data-quality objectives prior to analysis. Users of the method must demonstrate the ability to generate acceptable results with this method, using the procedures described in Section 9, Quality Control.

## – Support Data for USEPA method 300.0 Part A –

**Table 1. Method Detection Limits for Inorganic Anions in Reagent Water**

Analyte	Known Conc. (mg/L)	Determined Conc. (mg/L)	Std. Dev. (mg/L)	MDL (mg/L)	PQL (mg/L)
Fluoride	0.02	0.023	0.001	0.004	0.01
Chloride	0.025	0.038	0.001	0.004	0.01
Nitrite-N	0.02	0.026	0.002	0.008	0.02
Bromide	0.05	0.050	0.006	0.018	0.05
Nitrate-N	0.02	0.025	0.001	0.004	0.01
Phosphate-P	0.04	0.046	0.004	0.012	0.04
Sulfate	0.05	0.074	0.004	0.012	0.04

**Note:** The instrument was calibrated by including the MDL standard in the calibration model (Section 7.11). Seven replicates of a standard were analyzed. The MDL equals the standard deviation of the determined values for seven replicate runs multiplied by 3.142, corresponding to a 99% confidence value. The PQL (practical quantitation limit) equals the standard deviation multiplied by 10.

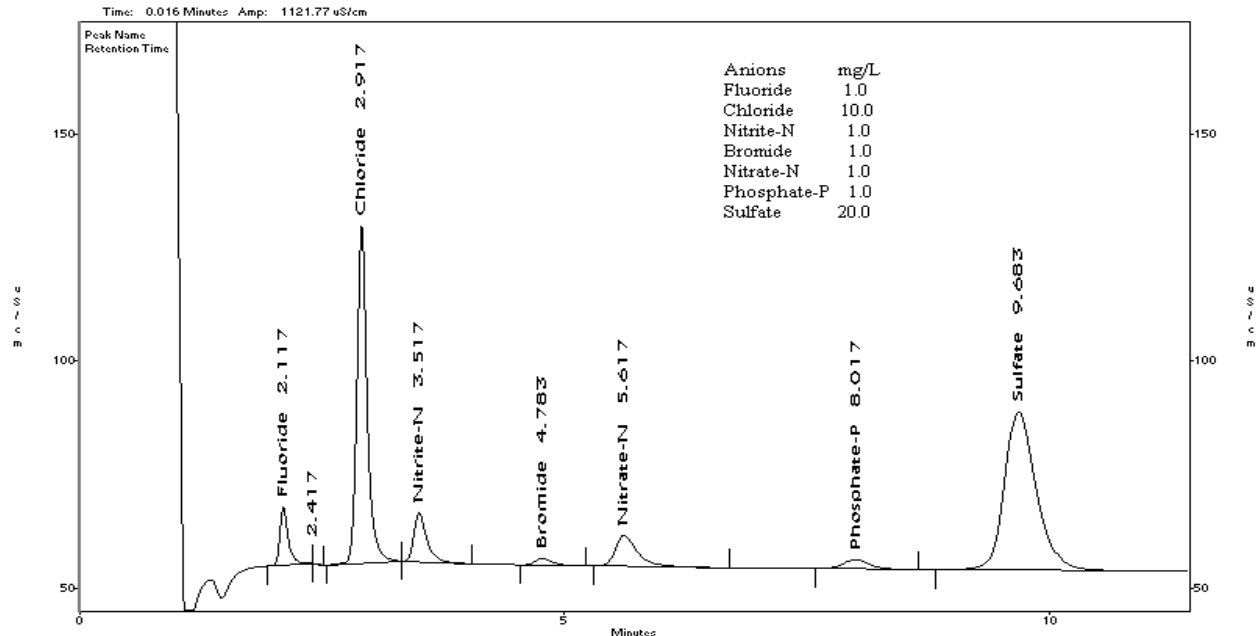
**Table 2. Single-Operator Accuracy and Precision of Standard Anions**

*(Seven replicates of each sample were analyzed)*

Analyte	Sample Type	Spike mg/L	Mean	Standard
			Recovery -- % --	Deviation - mg/L -
Bromide	RW	0.5	100	0.006
	DW	0.5	96	0.007
	SW	0.5	96	0.031
	WW	0.5	101	0.006
	GW	0.5	97	0.021
	SD	0.9	97	0.022
Chloride	RW	5.0	92	0.061
	DW	5.0	102	0.285

	SW	5.0	103	2.295
	WW	5.0	101	0.544
	GW	5.0	98	0.440
	SD	9.1	92	0.329
Fluoride	RW	0.5	100	0.006
	DW	0.5	101	0.022
	SW	0.5	100	0.036
	WW	0.5	100	0.010
	GW	0.5	91	0.029
	SD	0.9	91	0.029
Nitrate-N	RW	0.5	99	0.007
	DW	0.5	99	0.011
	SW	0.5	100	0.058
	WW	0.5	99	0.006
	GW	0.5	96	0.023
	SD	0.9	100	0.029
Nitrite-N	RW	0.5	98	0.006
	DW	0.5	92	0.006
	SW	0.5	90	0.028
	WW	0.5	88	0.007
	GW	0.5	94	0.020
	SD	0.9	101	0.027
O-Phosphate-P	RW	0.5	98	0.009
	DW	0.5	94	0.009
	SW	0.5	98	0.054
	WW	0.5	98	0.008
	GW	0.5	98	0.034
	SD	0.9	99	0.041
Sulfate	RW	10.0	93	0.152
	DW	10.0	103	0.576
	SW	10.0	98	1.034
	WW	10.0	103	0.291
	GW	10.0	101	3.340
	SD	18.2	100	0.558

RW; Reagent Water, WW; Wastewater, DW; Drinking Water, GW; Groundwater, SW; Surface Water, and SD; Solid (soil sample was used; soil-water extract prepared using the procedure described in USEPA method 300.0).



**Figure 1. Chromatogram Showing Separation Using the QS-A1 Anion Profiling Column.**

Note, Example shown is for running IC5000. When running QC8000, the baseline will drop at 0.2 min.

090399.013 Acquired -- 3 September 1999

## Lachat QuikChem Method Number 10-540-00-1-C (SM 4110 D) Ion Chromatographic Determination of Oxyhalides and Bromide

### Inorganic Disinfection Byproducts in Drinking Waters

5 to 50  $\mu\text{g ClO}_2^-/\text{L}$   
5 to 50  $\mu\text{g BrO}_3^-/\text{L}$   
20 to 200  $\mu\text{g ClO}_3^-/\text{L}$   
10 to 100  $\mu\text{g Br}^-/\text{L}$

#### – Principle –

This is a chemically-suppressed ion chromatography (IC) method. The anions are separated based on their affinities toward the stationary phase in the column. A suppressor cartridge, packed with high-capacity cation exchange resin, chemically suppresses the background conductance of the eluent and, at the same time, converts the anionic species into species of higher conductance. The method provides results that are similar to those of USEPA Method 300.1.

#### – Special Apparatus –

- This method is for use with **Omnion 3.0** only.
- Lachat Instruments QuikChem 8500 IC+ Ion Chromatography System

#### – QuikChem Method 10-540-00-1-C –

1.1 The method covers the determination of inorganic disinfection byproducts in waters.

- 1.2 The matrices applicable to this method are as follows:  
Finished and raw drinking waters.
- 1.3 The single laboratory Method Detection Limits (MDL defined in Section 3.11) for chlorite, bromate, chlorate, and bromide in reagent water are 2.61, 1.15, 5.00, and 2.01 µg/L respectively. The MDL for a specified matrix may differ from those listed, depending upon the nature of the sample.
- 1.4 The range tested for this anion is as follows:

Analyte	Range, µg/L	Analyte	Range, µg/L
Chlorite	5 to 50	Chlorate	20 to 200
Bromate	5 to 50	Bromide	10 to 100

**Note:** The calibrated range for this method is 10-fold. These ranges were based on a 200 µL sample loop.

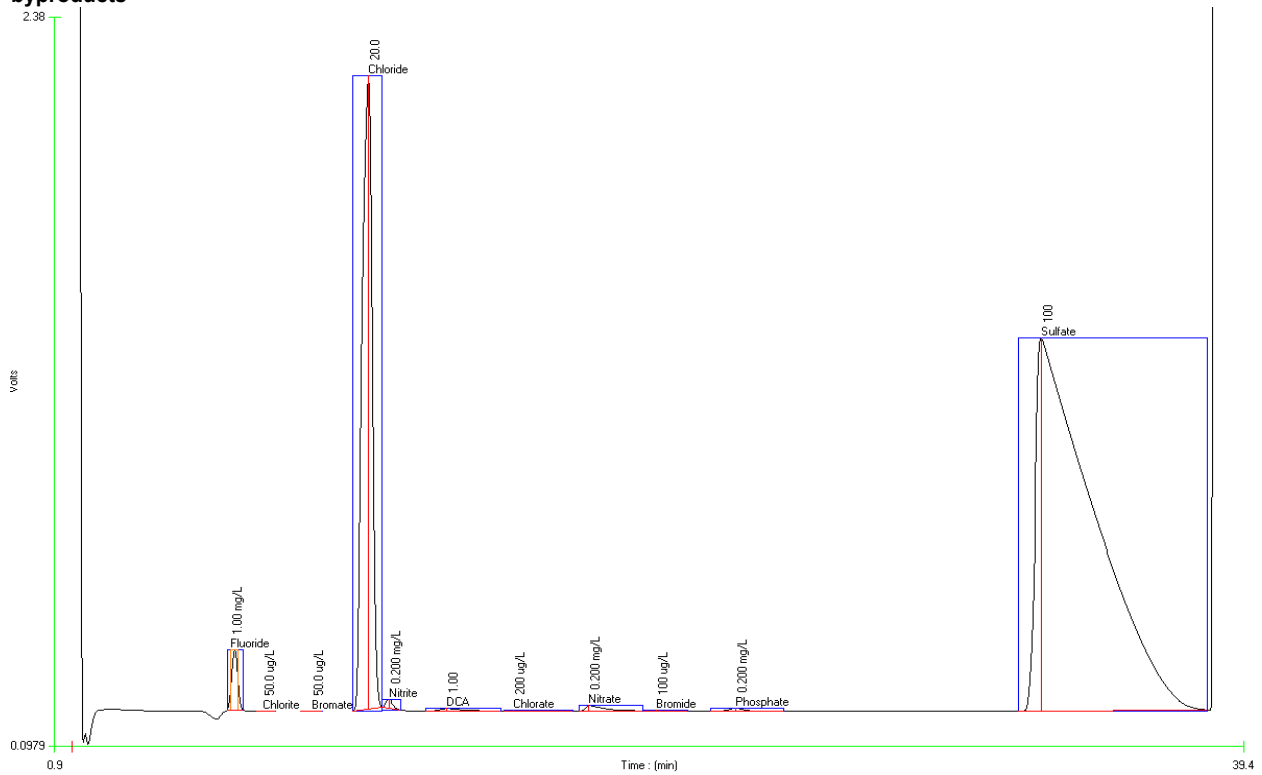
- 1.5 This method is recommended for use only by analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatogram.
- 1.6 When this method is used to analyze unfamiliar samples, peak identification should be supported by the use of a fortified sample matrix covering the anions of interest. The fortification procedure is described in Section 9.4.
- 1.8 Users of the method data should state the data-quality objectives prior to analysis. Users of the method must demonstrate the ability to generate acceptable results with this method, using the procedures described in Section 9.

### – Precision and MDL Determination –

Anion	Known amount	Mean	Std. Dev.	MDL	PQL	RSD	Recovery
	µg/L	µg/L	µg/L	µg/L	µg/L	%	%
Fluoride	200	207.4	5.91	18.56	59.1	2.85	103.7
Chlorite	10.0	9.75	0.83	2.61	8.31	8.52	97.5
Bromate	10.0	9.96	0.37	1.15	3.66	3.68	99.6
Nitrite-N	40.0	34.54	0.66	2.09	6.65	1.93	86.4
Chlorate	40.0	37.2	1.59	5.00	15.93	4.28	93.0
Bromide	20.0	18.73	0.64	2.01	6.40	3.41	93.6
Nitrate-N	40.0	38.64	0.27	0.85	2.70	0.70	96.6
Phosphate-P	40.0	38.76	0.66	2.09	6.65	1.72	96.9

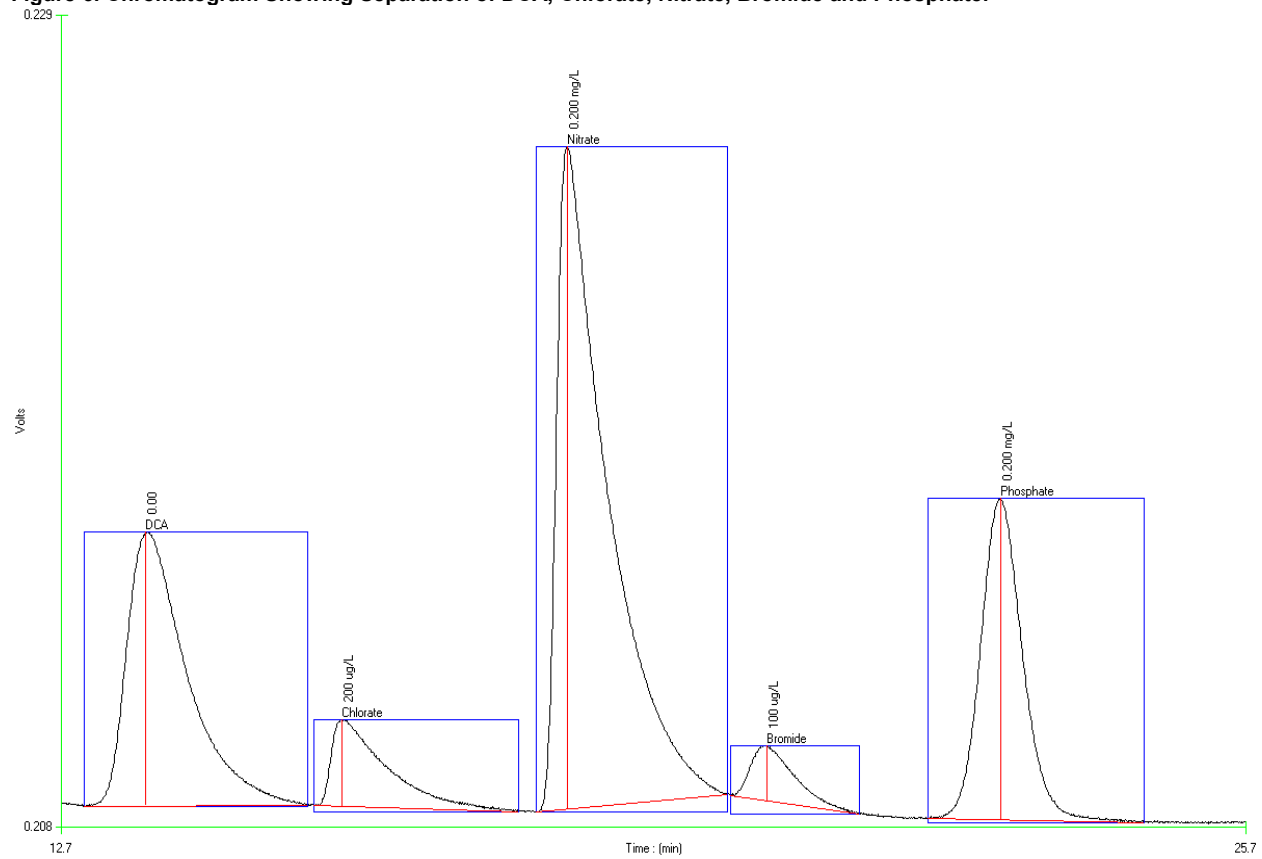
**Note:** Seven replicates of a standard were analyzed. The MDL equals the standard deviation of the determined values for seven replicate runs multiplied by 3.14, corresponding to a 99% confidence value. The PQL (practical quantitation limit) equals the standard deviation multiplied by 10.

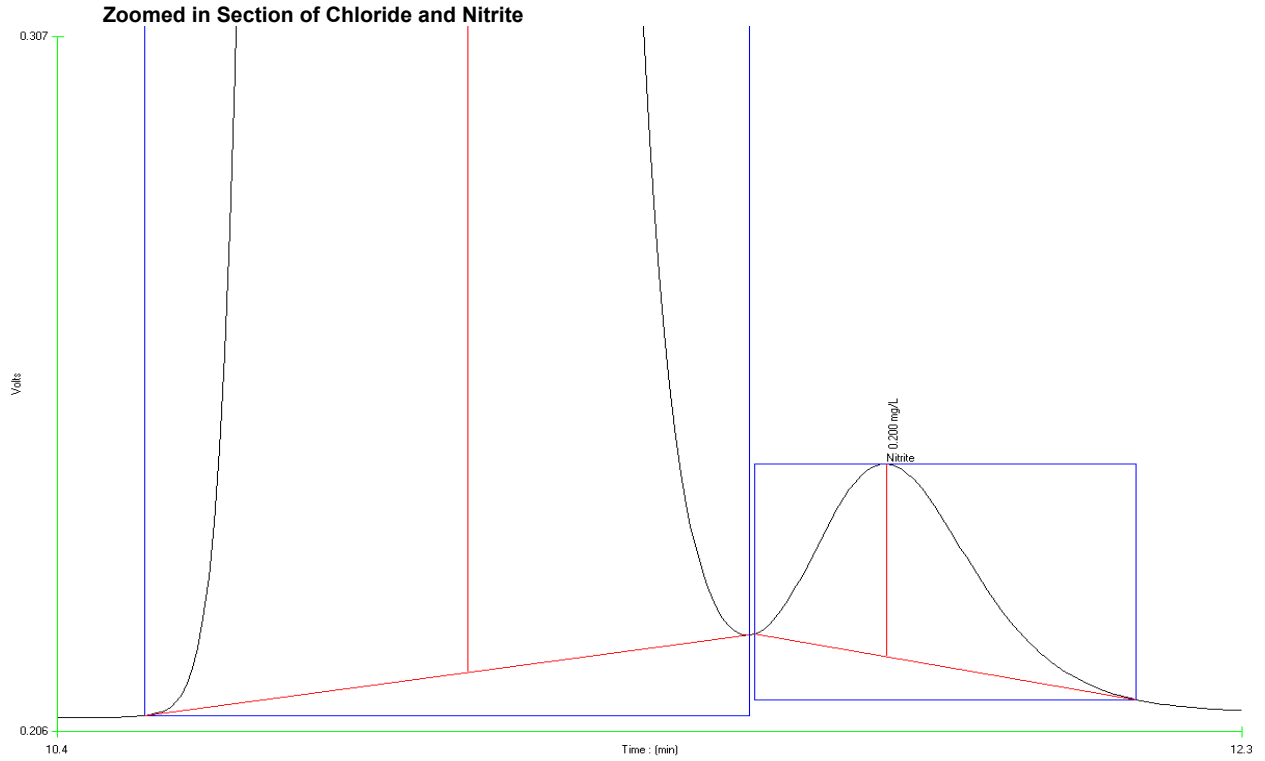
**Figure 1. Chromatogram of Calibration Standard A Showing Separation of common inorganic anions and the disinfection byproducts**



File Name: 11-20 cal mdl.omn  
Acq. Date: 20 November 2008

**Figure 6. Chromatogram Showing Separation of DCA, Chlorate, Nitrate, Bromide and Phosphate.**





File Name: 11-20 cal mdl.omn  
Acq. Date: 20 November 2008

# Ammonia

## Lachat QuikChem<sup>®</sup> Method 10-107-06-1-J SM 4500 NH<sub>3</sub> H. Flow Injection Analysis

### Ammonia (Phenolate) in Potable and Surface Waters

0.01 to 2.0 mg N/L as NH<sub>3</sub>

#### – Principle –

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

If distillation is required the sample is buffered at a pH of 9.5 with a borate buffer to decrease hydrolysis of cyanates and organic nitrogen compounds, and is distilled into a solution of boric acid.

#### – Interferences –

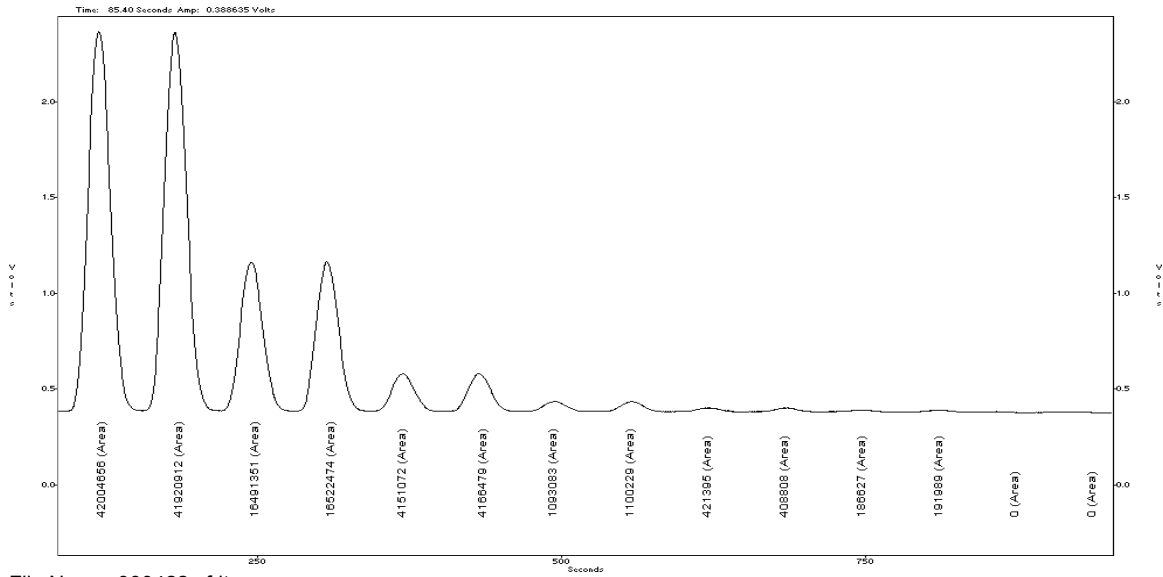
1. Calcium and magnesium ions may precipitate if present in sufficient concentration. Tartrate or EDTA is added to the sample in-line in order to prevent this.
2. Color, turbidity, and certain organic species may interfere. Turbidity is removed by manual filtration. Sample color may be corrected for by running the samples through the manifold without color formation.
3. Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at a pH of 9.5 at which distillation is carried out.
4. Residual chlorine must be removed by pretreatment of the sample with sodium thiosulfate or other reagents before distillation.
5. Method interference may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that bias analyte response.
6. Eliminate any marked variation in acidity or alkalinity among samples because intensity of measured color is pH – dependent. Likewise, ensure that pH of standard ammonia solutions approximates that of sample.

#### – Special Apparatus –

Please contact Lachat Technical Support for Ordering Information

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

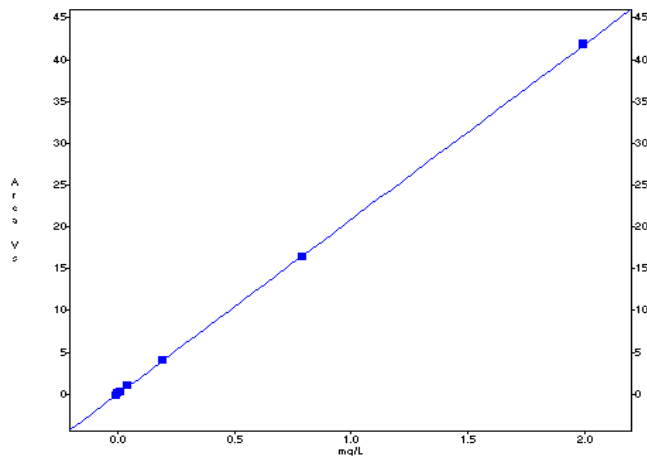
## – Calibration Data for Ammonia –



File Name: 990422c.fdt  
Acq. Date: 22 April 1999

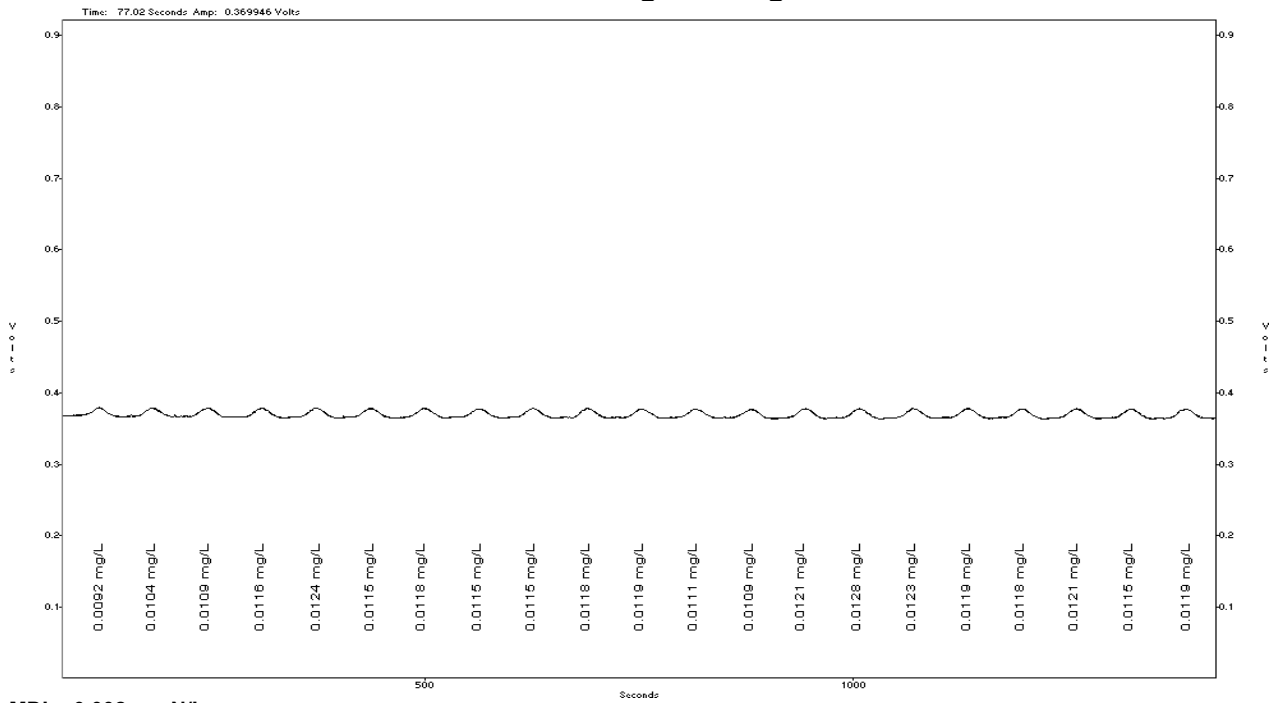
### Calibration Graph and Statistics

Level	Area	mg N/L	Determined	Replicate %RSD	% residual
1	41962784	2.00	2.00	0.1	-0.2
2	16506912	0.80	0.81	0.1	1.3
3	4158776	0.20	0.20	0.3	0.1
4	1096656	0.05	0.05	0.5	-7.5
5	415102	0.02	0.02	2.1	-6.0
6	189308	0.01	0.01	2.0	-4.3
7	0	0.00	---	0.0	---



Scaling: None  
Weighting: None  
1st Order Poly  
Conc = 4.773e-008 Area + 1.394e-003  
r = 1.0000

### Method Detection Limit for ammonia using 0.01 mg N/L standard



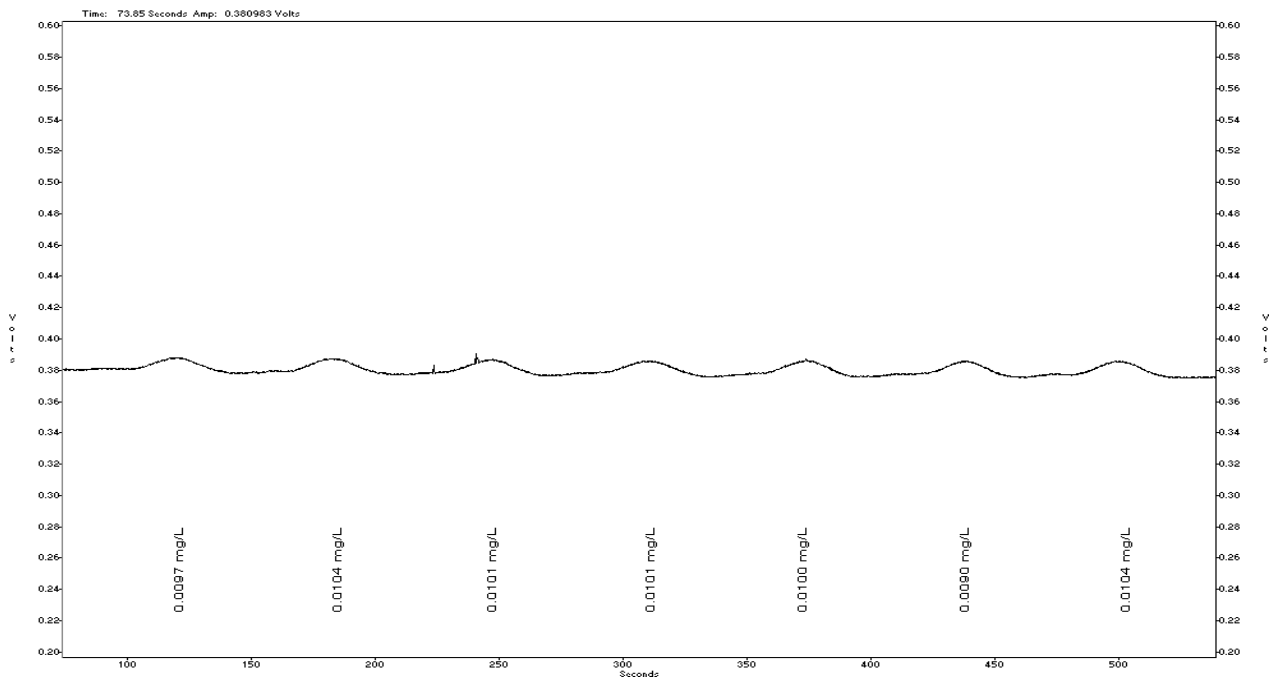
**MDL= 0.002 mg N/L**

Standard Deviation (s) = 0.0008 mg N/L, Mean (x) = 0.012 mg N/L, Known value = 0.01 mg N/L

Acq. Date: 21 April 1999

File Name: 990421m1.fdt

### Method Detection Limit for ammonia using 7 replicates of 0.01 mg N/L standard in DI water



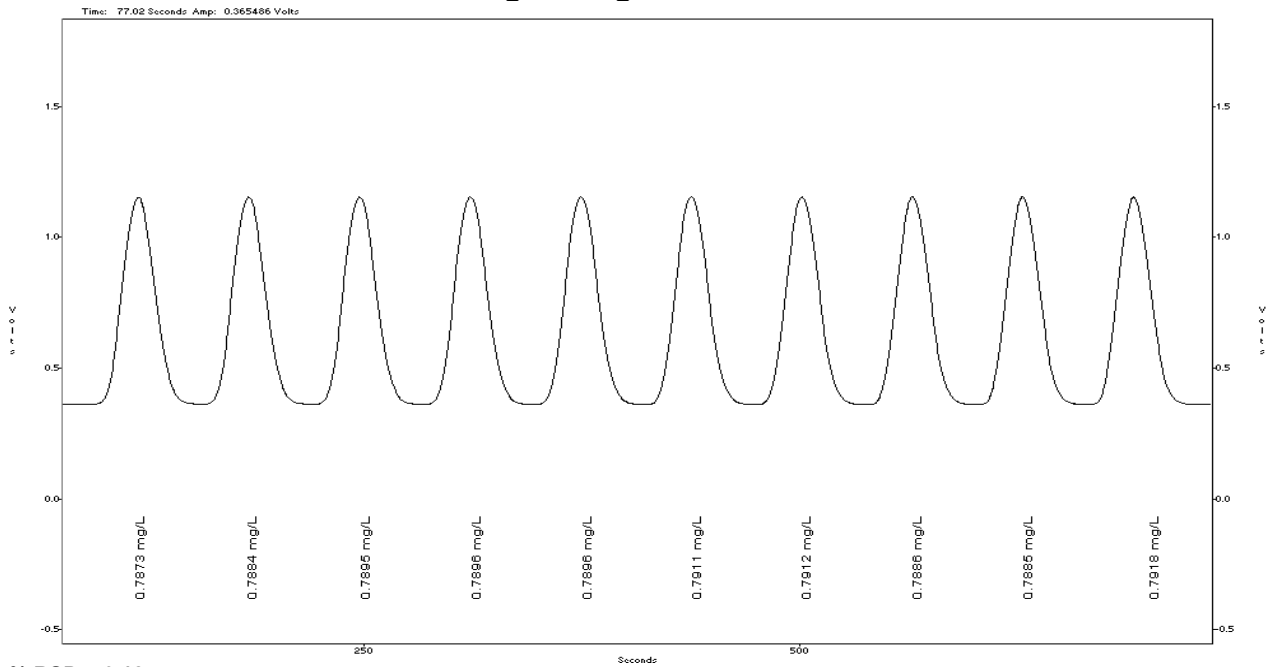
**MDL= 0.0015 mg N/L**

Standard Deviation (s) = 0.0005 mg N/L, Mean (x) = 0.010 mg N/L, Known value = 0.01 mg N/L

Acq. Date: 21 April 1999

File Name: 990421m.fdt

**Precision data for ammonia using 0.8 mg N/L standard**



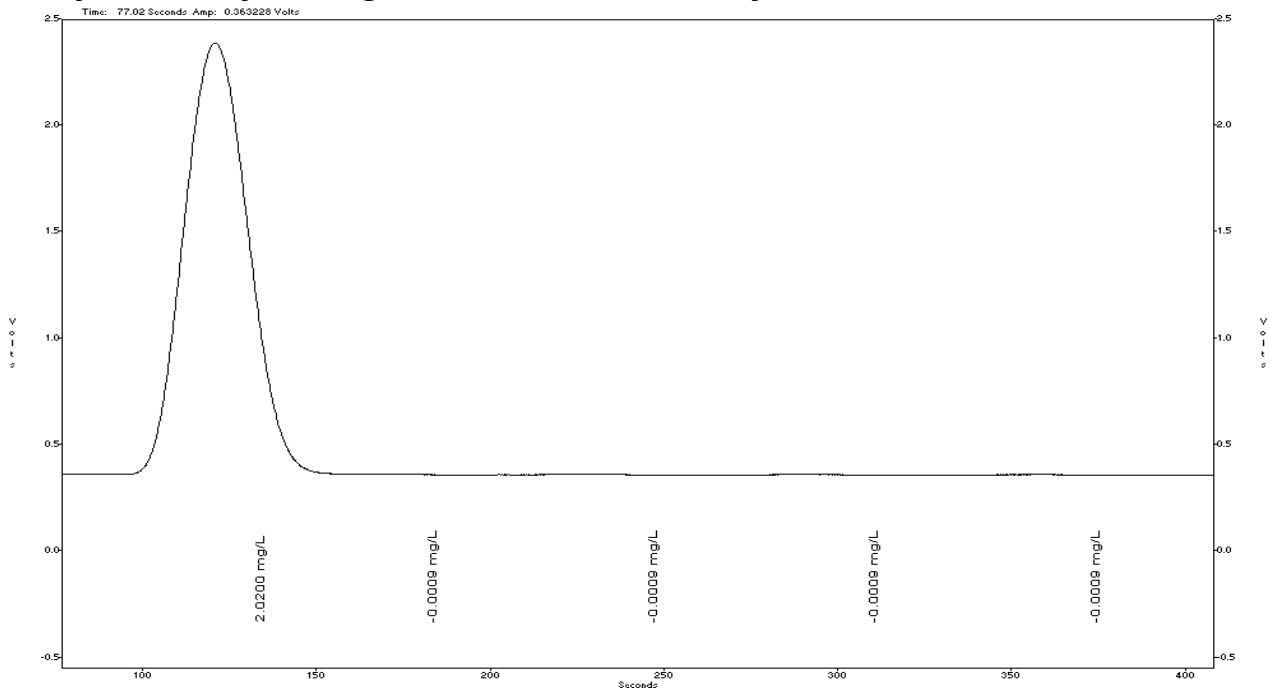
**% RSD = 0.18**

Standard Deviation (s) = 0.0014 mg N/L, Mean (x) = 0.790 mg N/L, Known value = 0.8 mg N/L

Acq. Date: 21 April 1999

File Name: 990421p.fdt

**Carryover Study: 2.0 mg N/L standard followed by 4 DI water blanks**



**Carryover Passed**

Acq. Date: 21 April 1999

File Name: 990421cr.fdt

# Bromide

## Lachat QuikChem<sup>®</sup> Method 30-135-21-1-A SM 4500 Br<sup>-</sup>D. Flow Injection Analysis

### Bromide in Brackish Waters Low Flow Method

0.5 to 10.0 mg Br/L

#### – Principle –

Bromide is oxidized to bromine by chloramine-T, followed by substitution of bromine on phenol red to produce bromophenol blue. The absorbance measured at 590 nm is proportional to the concentration of bromide in the sample. Sodium thiosulfate is added to reduce interference from chloride.

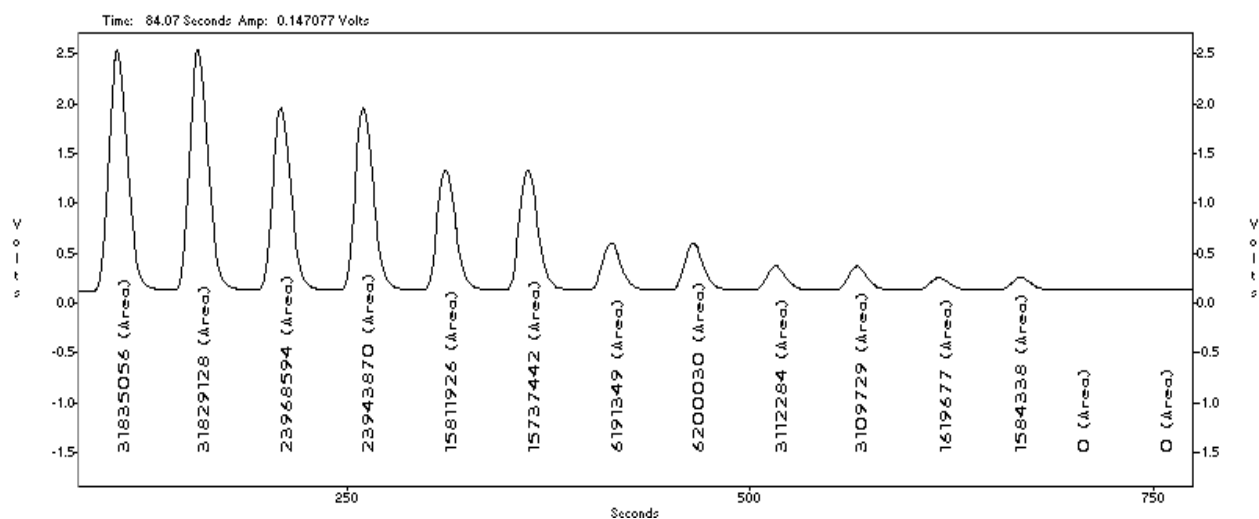
#### – Interferences –

1. Chloride interference is reduced by the addition of sodium thiosulfate. Chloramine-T dissociates in aqueous solution to form hypochlorous acid. The hypochlorous acid can then react with chloride, causing substitution of chloride at positions ortho to the hydroxy groups on phenol red, just as bromination. Sodium thiosulfate reacts with chlorine to reduce this interferent. 5000 mg Cl/L gave a positive interference of 0.15 mg Br/L (See Method Support Data, Section 11.3).

#### –QuikChem<sup>®</sup> Method 30-135-21-1-A –

1. This method covers the determination of bromide in waters containing up to 20,000 mg Cl/L.
2. This includes drinking, ground, and surface waters, as well as domestic and industrial wastes. This method determines total bromide or if the sample is filtered through a 0.45 micron pore size filter, the result is termed dissolved bromide. The difference between the result of a sample determined directly and filtered is termed insoluble bromide.
3. The applicable range is 0.5 to 10.0 mg Br/L. The method detection limit is 0.005 mg Br/L. The method throughput is 72 injections per hour.
4. Chloride interference is reduced by the addition of sodium thiosulfate. Chloramine-T dissociates in aqueous solution to form hypochlorous acid. The hypochlorous acid can then react with chloride, causing substitution of chloride at positions ortho to the hydroxy groups on phenol red, just as bromination. Sodium thiosulfate reacts with chlorine to reduce this interferent. 5000 mg Cl/L gave a positive interference of 0.15 mg Br/L. (See Method Support Data)
5. Samples must be free of turbidity. Filtration through paper or a 0.45 uM membrane is recommended for turbid samples.

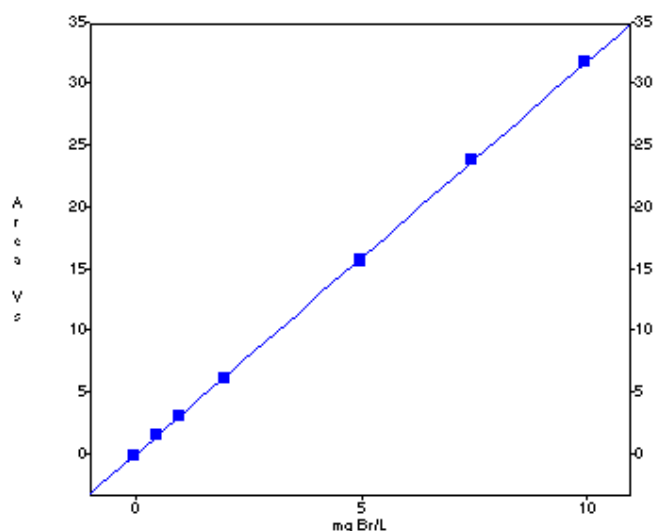
### Calibration Data for Bromide



Method File Name: 961025c1.fdt  
Acq. Date: 25 October 1996

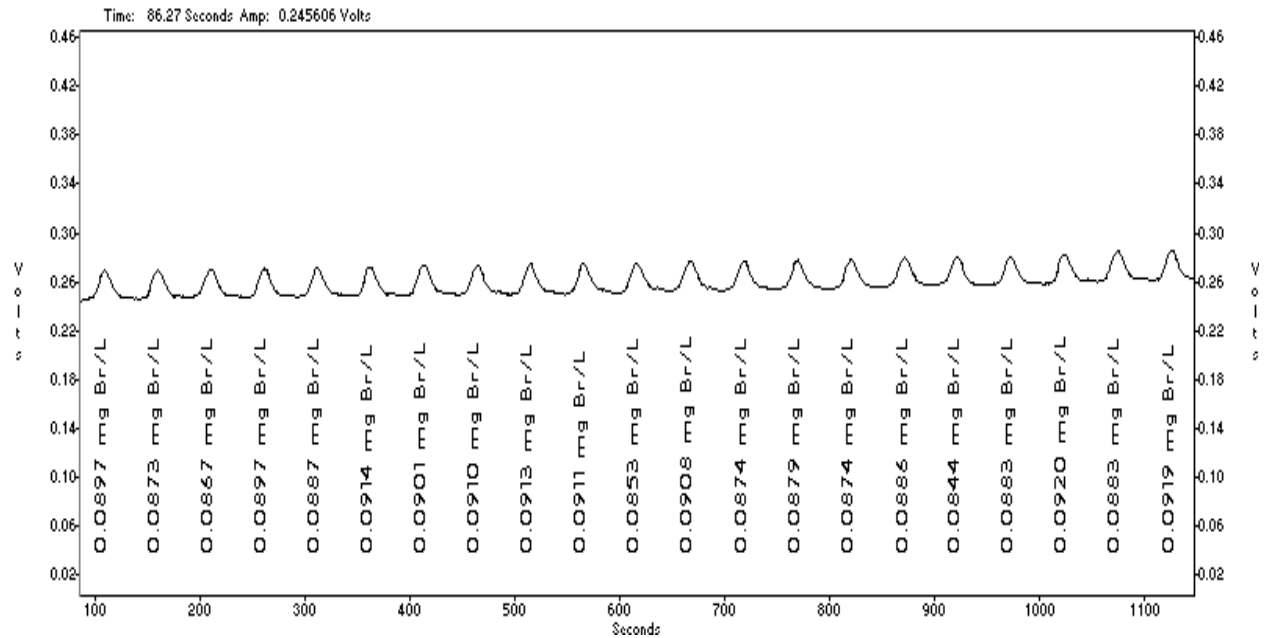
### Calibration Graph and Statistics

Level	Avg.(Area)	mg Br/L	Determined	Replicate %RSD	% residual
1	31832092	10.0	10.04	0.0	-0.4
2	23956232	7.50	7.55	0.1	-0.7
3	15774684	5.00	4.98	0.3	0.5
4	6195689	2.00	1.95	0.1	2.3
5	3111007	1.00	0.98	0.1	1.9
6	1602007	0.50	0.51	1.6	-1.0
7	0	0	0	0.0	-----



Scaling: None  
Weighting: 1/X  
1st Order Poly  
Conc = 3.153e-007 Area + 8.978e-009  
R<sup>2</sup> = 0.9999

### Method Detection Limit for bromide using 0.10 mg Br/L standard



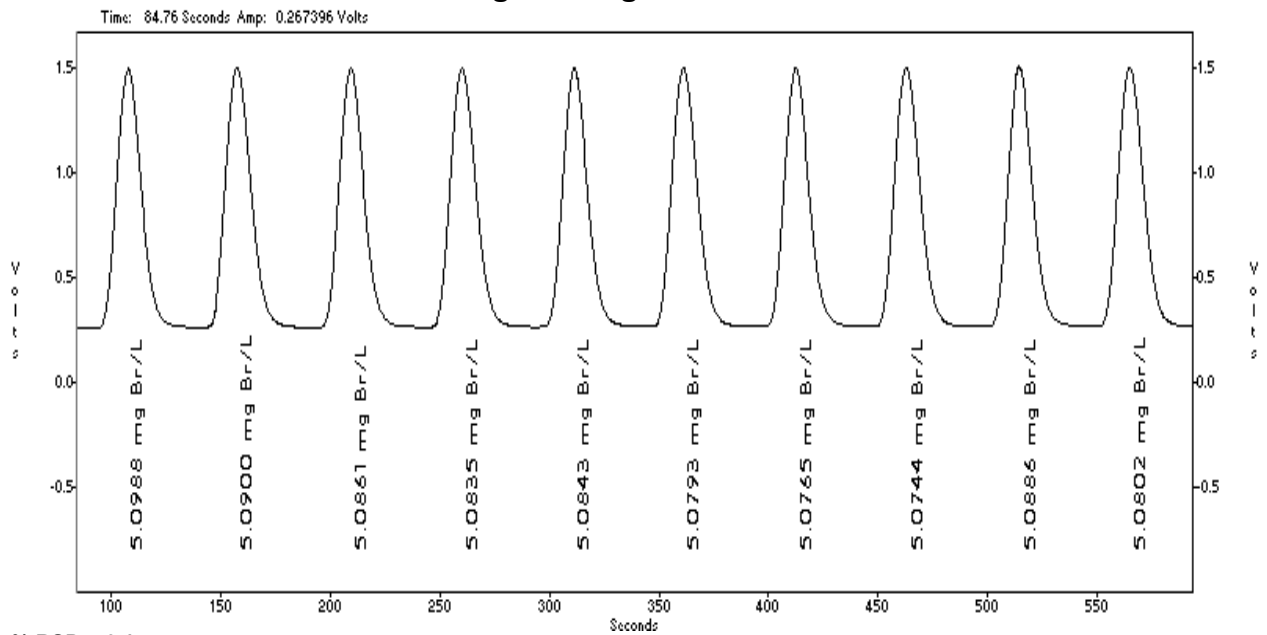
**MDL = 0.0053 mg/L**

Standard Deviation (s) = 0.0021 mg Br/L, Mean (x) = 0.089 mg Br/L, Known value = 0.10 mg Br/L

Data File name 961925m2.fdt

Acq. Date: 25 October 1996

### Precision data for bromide using 5.00 mg Br/L standard



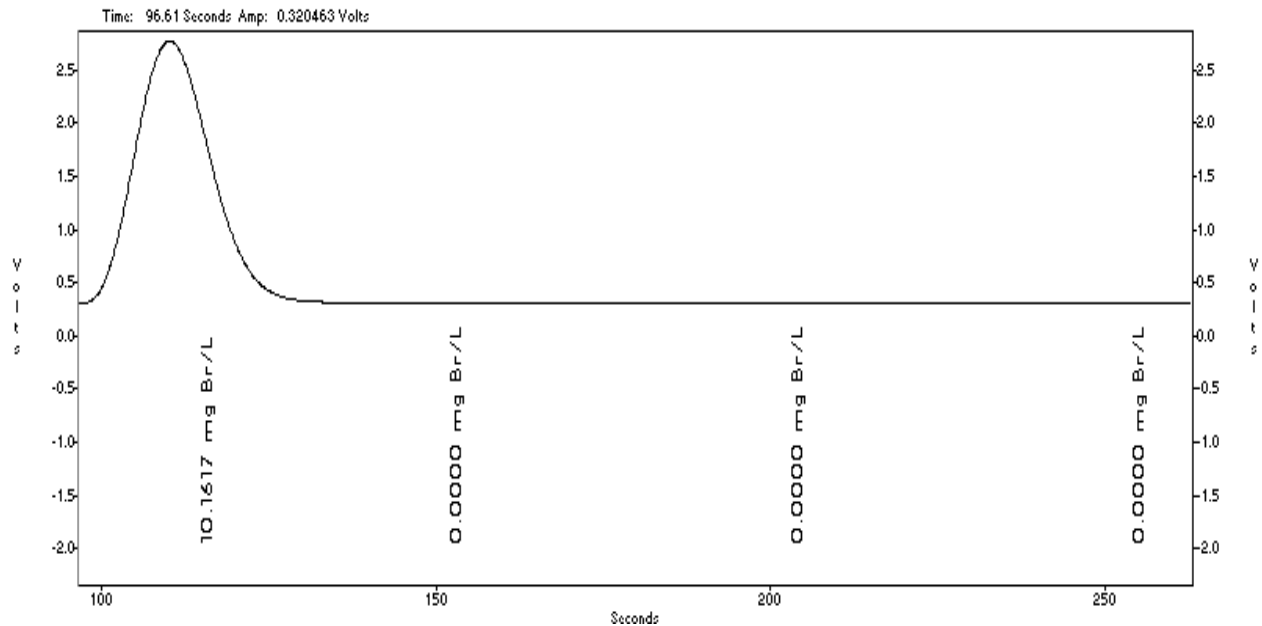
**% RSD = 0.14**

Standard Deviation (s) = 0.007 mg Br/L, Mean (x) = 5.08 mg Br/L, Known value = 5.00 mg Br/L

Data File name 961025p1.fdt

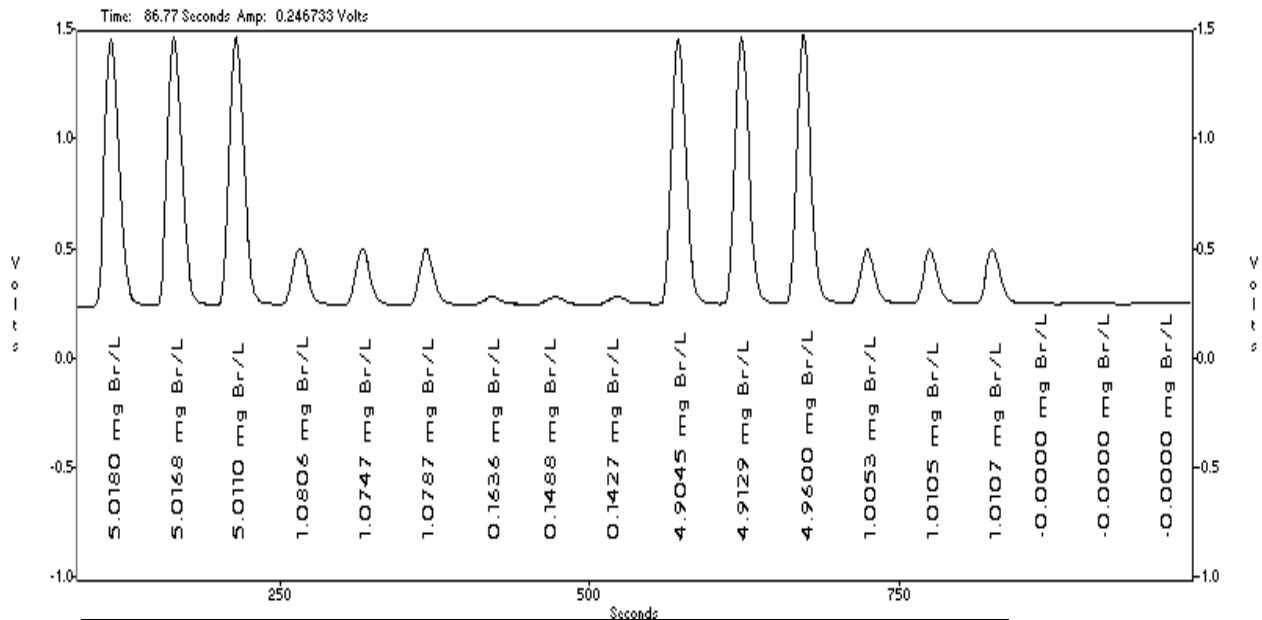
Acq. Date: 25 October 1996

### Carryover Study: 10 mg Br/L standard followed by 3 blanks



**Carryover Passed**  
Data File name 961025r2.fdt  
Acq. Date: 25 October 1996

### Effect of Chloride on Bromide; 5.000 mg Cl/L



Peaks	Concentration
5,000 mg Cl/L	
1-3, 5.00 mg Br/L	5.02
4-6, 1.00 mg Br/L	1.08
7-9, Blank, 5000 mg Cl/L	0.15
DI water (No chloride)	
10-12, 5.00 mg Br/L	4.93
13-15, 1.00 mg Br/L	1.01
16-18, Blank - DI water	0.00

# Cyanide

## Lachat QuikChem<sup>®</sup> Method 10-204-00-1-A

SM 4500 CN<sup>-</sup> Total Cyanide after distillation, by Flow Injection Analysis

### Determination of Cyanide (Macro Distillation Method) in Waters

0.005 to 0.500 mg CN<sup>-</sup>/L

#### – Principle –

The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a manual reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is converted to cyanogen chloride by reactions with Chloramine-T that subsequently reacts with pyridine and barbituric acid to give a red-color complex. Absorbance of this at 570 nm complex is proportional to cyanide concentration in the sample.

#### – Interferences –

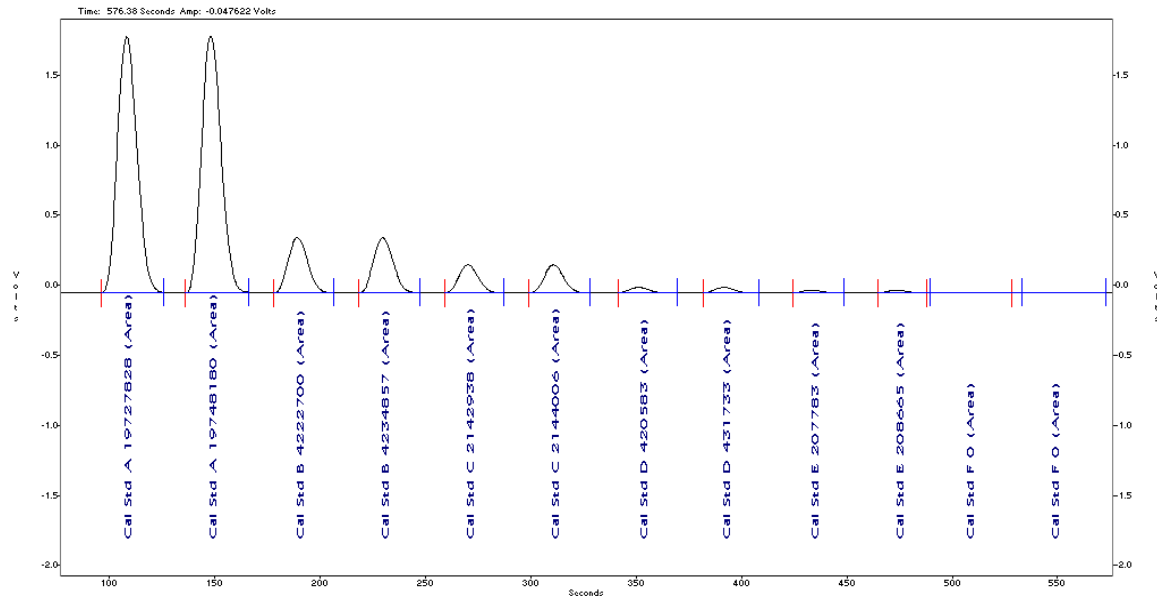
1. For strong acid dissociable and weak acid dissociable cyanide, non-volatile interferences are eliminated or minimized by the distillation procedure.
2. Some of the known interferences are aldehydes, nitrate-nitrite, and oxidizing agents, such as chlorine, thiocyanide, thiosulfate, and sulfide. Multiple interferences may require the analysis of a series of laboratory fortified sample matrices (LFM) to verify the suitability of the chosen treatment. See Standard Methods section 412A referenced in Section 16. for details of preliminary sample treatment to remove volatile interferences.

#### – Special Apparatus –

Please contact Lachat Technical Support for Ordering Information

1. Heater Module (Lachat Part No. A85100/A85200)
2. Macro Distillation setup

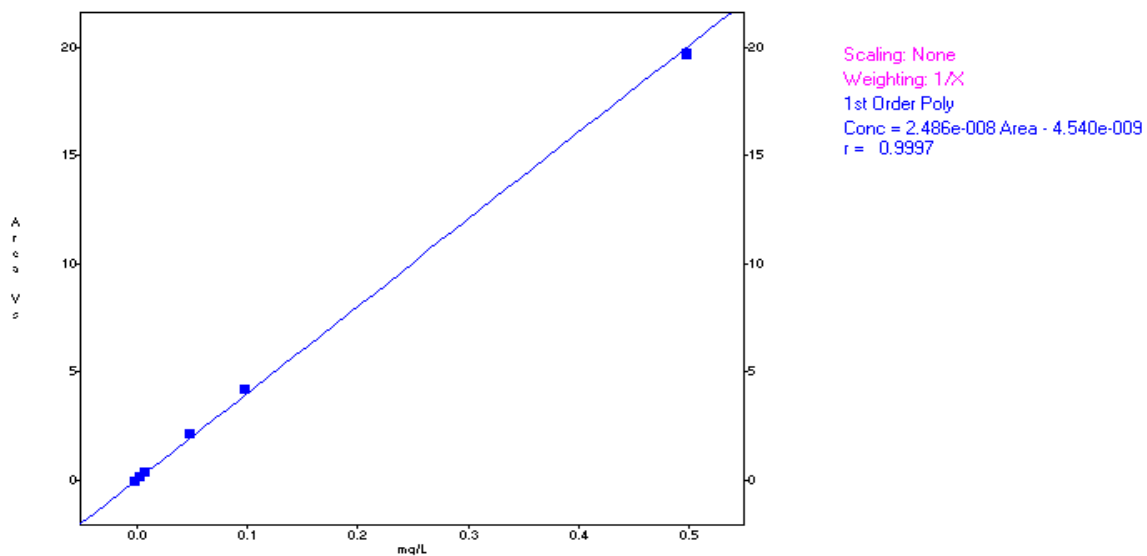
## Calibration Data for Cyanide



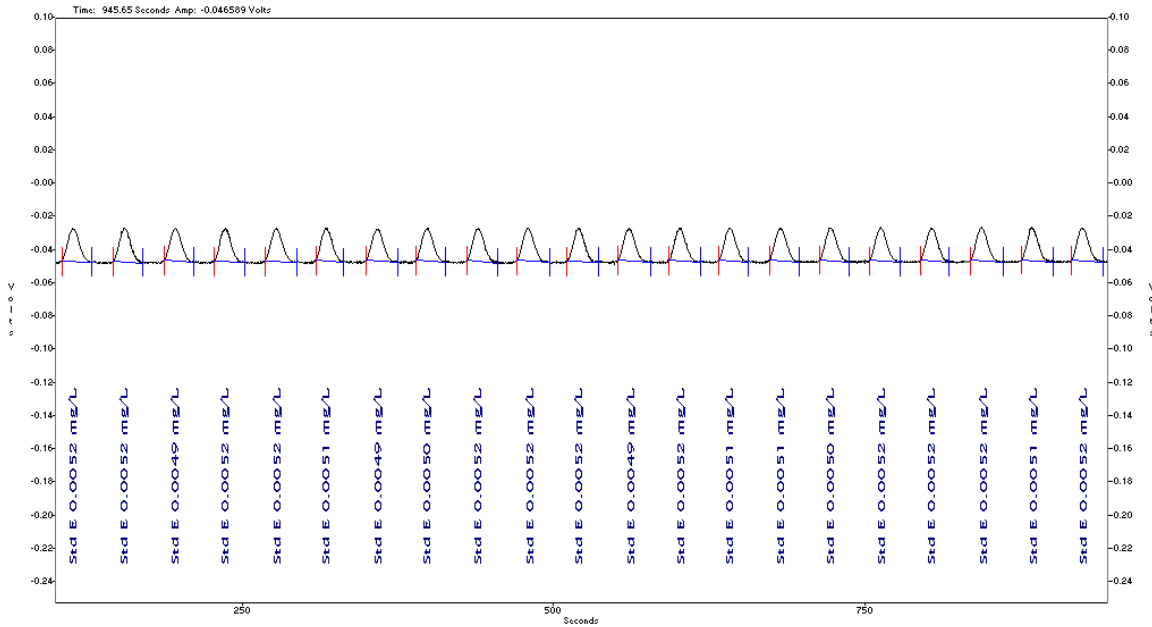
File Name: 010920c6.fdt  
Acq. Date: 20 September 2001

## Calibration Graph and Statistics

Level	Area	mg CN/L	Determined	Replicate %RSD	% residual
1	19738004	0.5	0.491	0.1	1.8
2	4228779	0.1	0.105	0.2	-5.1
3	2143472	0.05	0.053	0.0	-6.6
4	426158	0.01	0.011	1.9	-6.0
5	208224	0.005	0.005	0.3	-3.5
6	0	0	0	0.0	---



## Method Detection Limit claimed for Cyanide using Non Distilled 0.005 mg CN<sup>-</sup>/L Standards



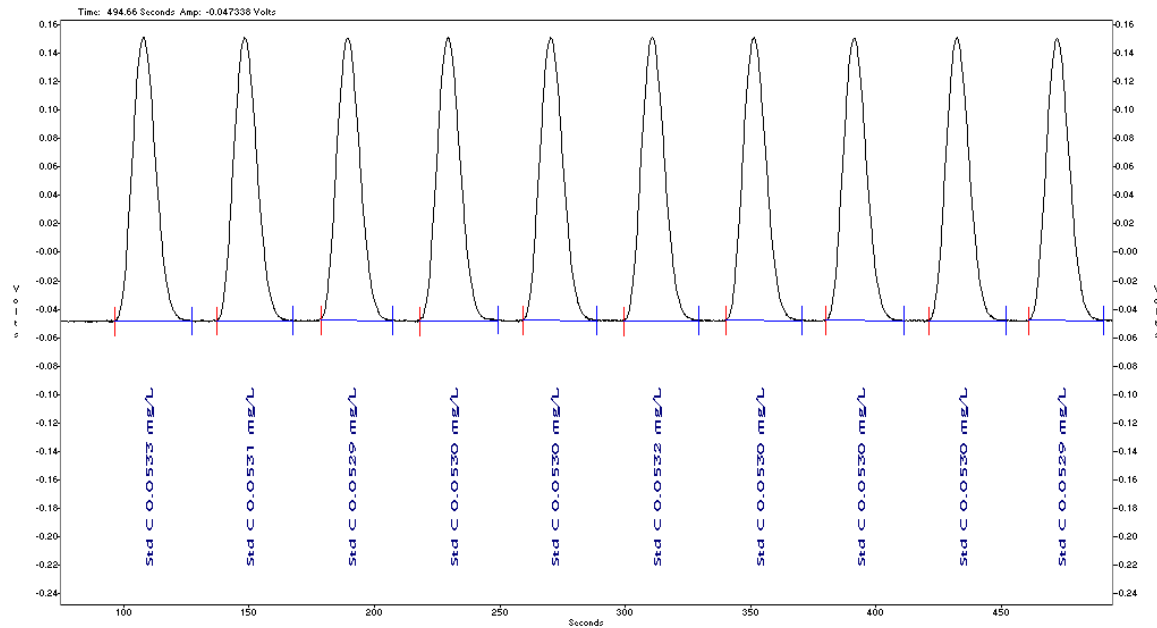
**MDL = 0.0005 mg CN<sup>-</sup>/L**

Standard Deviation (s) = 0.0001 mg CN<sup>-</sup>/L, Mean (x) = 0.0051 mg CN<sup>-</sup>/L, Known value = 0.005 mg CN<sup>-</sup>/L

File Name: 010920s2.fdt

Acq. Date: 20 September 2001

## Precision data for cyanide using 0.05 mg CN<sup>-</sup>/L standard



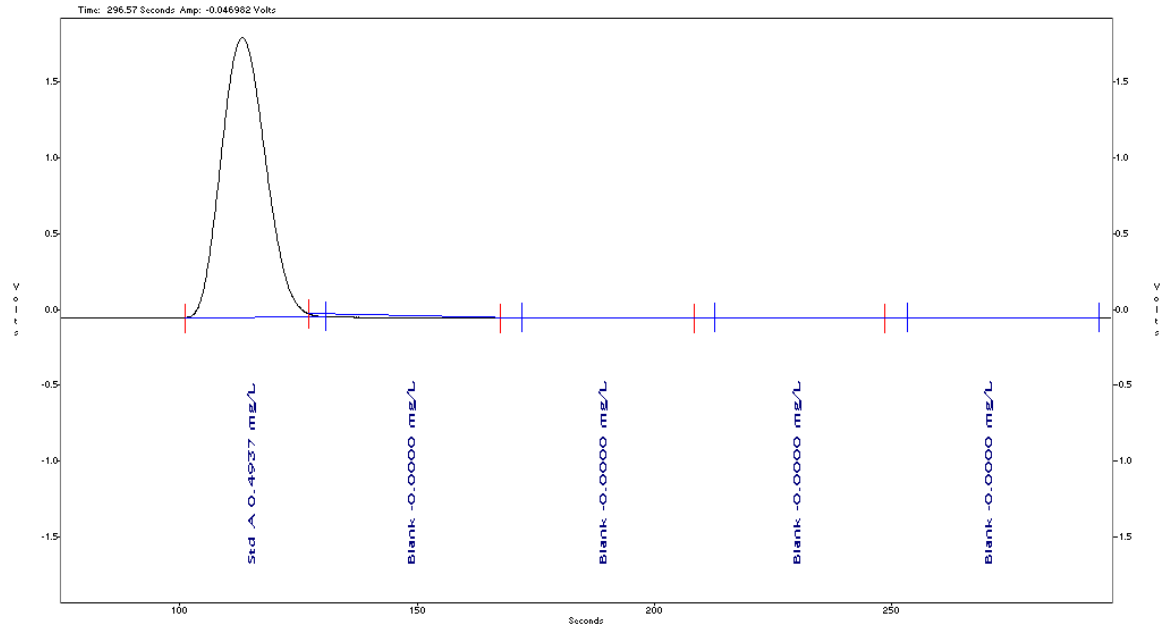
**% RSD = 0.255**

Standard Deviation (s) = 0.0001 mg CN<sup>-</sup>/L, Mean (x) = 0.053 mg CN<sup>-</sup>/L, Known value = 0.05 mg CN<sup>-</sup>/L

File Name: 010920s1.fdt

Acq. Date: 20 September 2001

## Carryover Study: 0.5 mg CN<sup>-</sup>/L standard followed by 4 blanks



**Carryover Passed**  
File Name: 010920s3.fdt  
Acq. Date: 20 September 2001

Method number 10-204-00-1-X (Total Cyanide after distillation using MicroDist) would also fall under this Standard Method. The Standard Methods states: Cyanides are digested and steam distilled from the sample as described in 4500 CN- I by using an apparatus described in 4500 CN- C or an equivalent Distillation Apparatus. In any case, the distillate should consist of cyanide in a 0.25M NaOH matrix.

Additionally, method number 10-204-00-1-X has been EPA approved for both NPDES and NPDWR monitoring.

# Chloride

**QuikChem<sup>®</sup> Method 10-117-07-1-A**  
SM 4500 Cl- G. Mercuric Thiocyanate Flow Injection Analysis

## Chloride in Waters

6.0 to 300.0 mg Cl/L

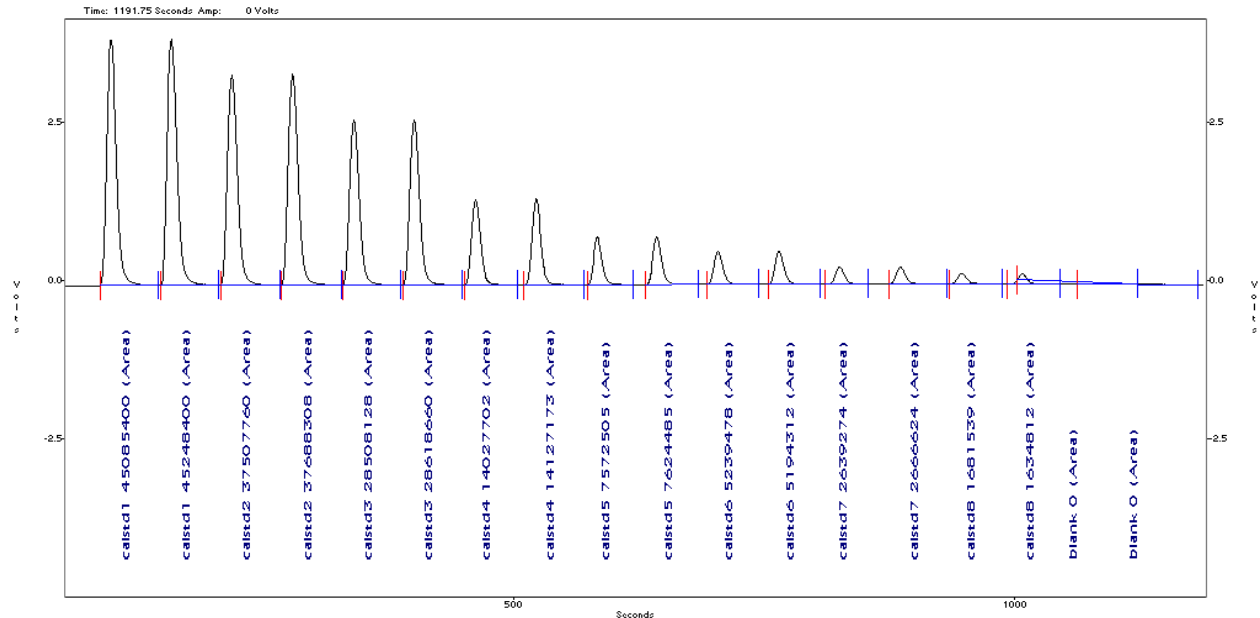
### – Principle –

Thiocyanate ion is liberated from mercuric thiocyanate by the formation of soluble mercuric chloride. In the presence of ferric ion, free thiocyanate ion forms the highly colored ferric thiocyanate, of which the absorbance is proportional to the chloride concentration. Ferric thiocyanate absorbs strongly at 480 nm. The calibration curve fits a second order polynomial.

### – Interferences –

1. Substances which reduce iron (III) to iron (II) and mercury (III) to mercury (II) (e.g. sulfite, thiosulfate).
2. Halides which also form strong complexes with mercuric ion (e.g. Br<sup>-</sup>, I<sup>-</sup>) give a positive interference.

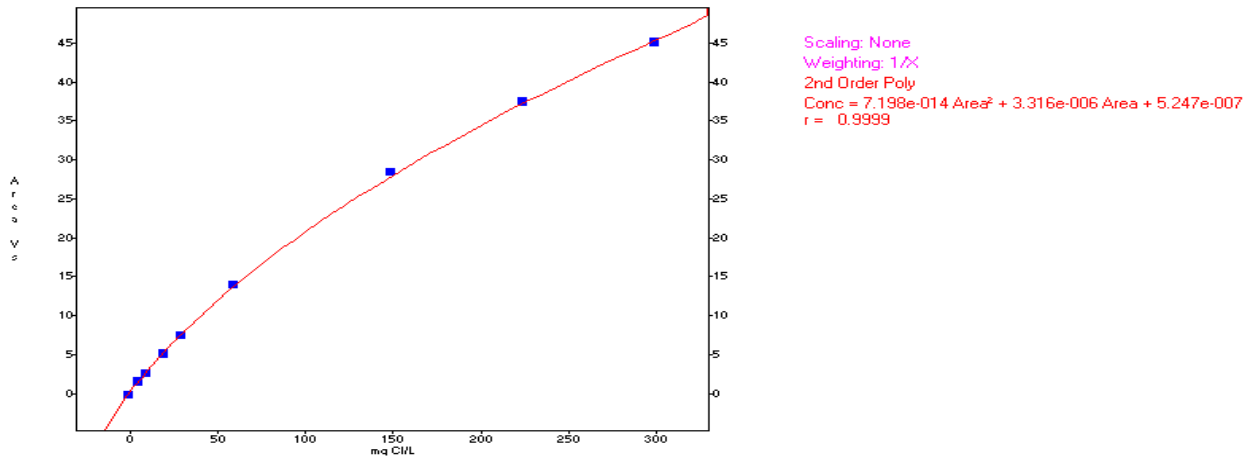
### Calibration Data for Chloride



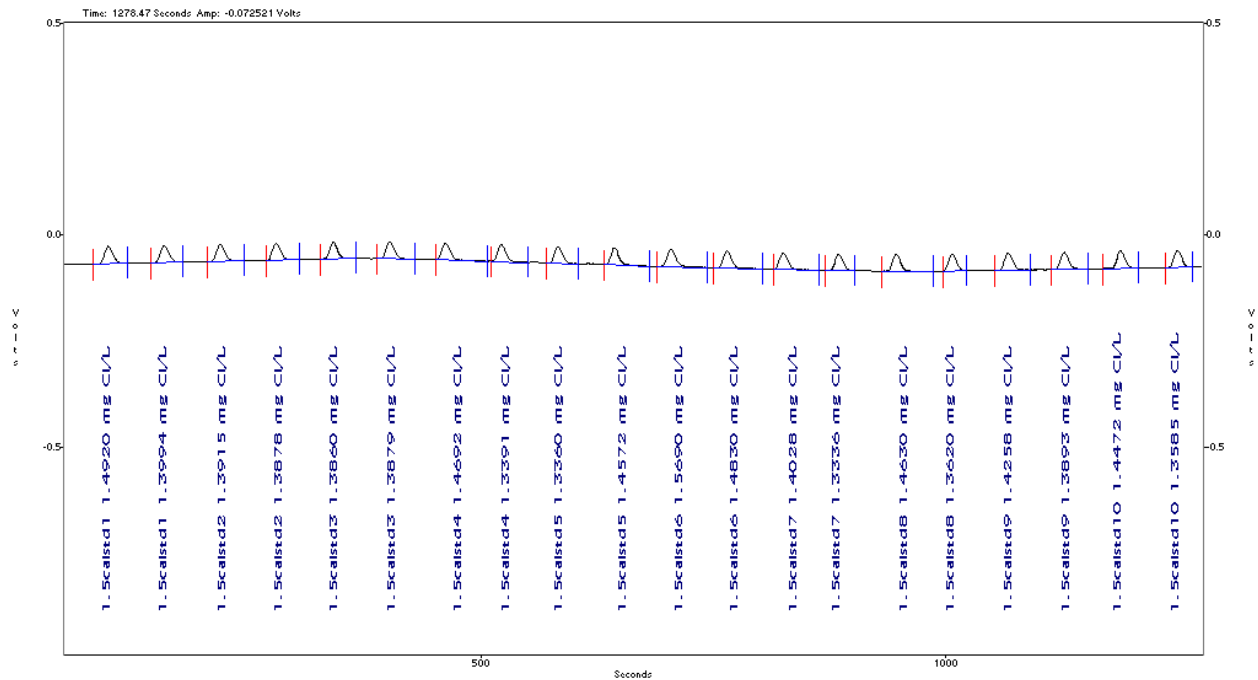
File Name: 1Clcal21.fdt  
Acq. Date: 21 August 2001

### Calibration Graph and Statistics

Level	Area	mg Cl/L	Determined	Replicate %RSD	% residual
1	45166900	300	296.7	0.3	1.1
2	37598032	225	226.4	0.3	-0.6
3	28563394	150	153.5	0.3	-2.3
4	14077438	60	60.96	0.5	-1.6
5	7598495	30	29.3	0.5	2.2
6	5216895	20	19.3	0.6	3.7
7	2652949	10	9.3	0.7	7.0
8	1658176	6	5.7	2.0	5.1
9	0	0	---	0.0	---

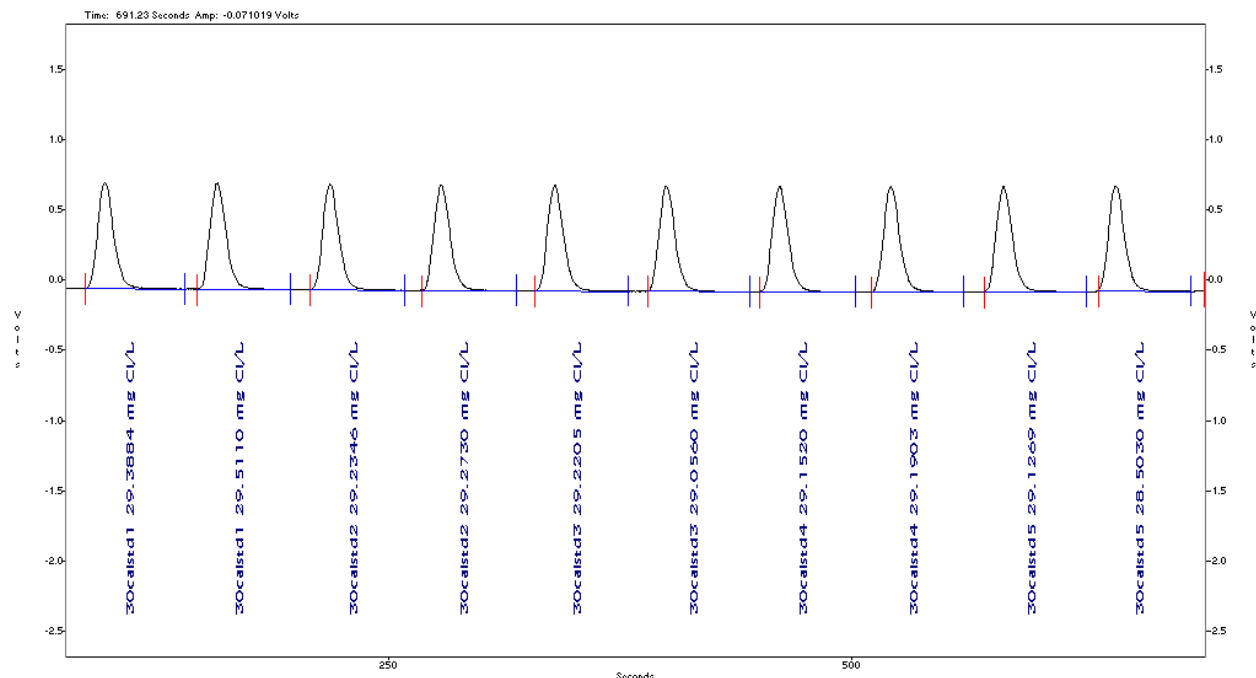


### Method Detection Limit for chloride using 1.5 mg Cl/L standard



**MDL= 0.15 mg Cl/L**  
Standard Deviation (s) = 0.05897 mg Cl/L, Mean (x) = 1.411 mg Cl/L, Known value = 1.5 mg Cl/L  
File Name: 1mdl21.fdt  
Acq. Date: 21 August 2001

### Precision data for chloride using 30 mg Cl/L standard



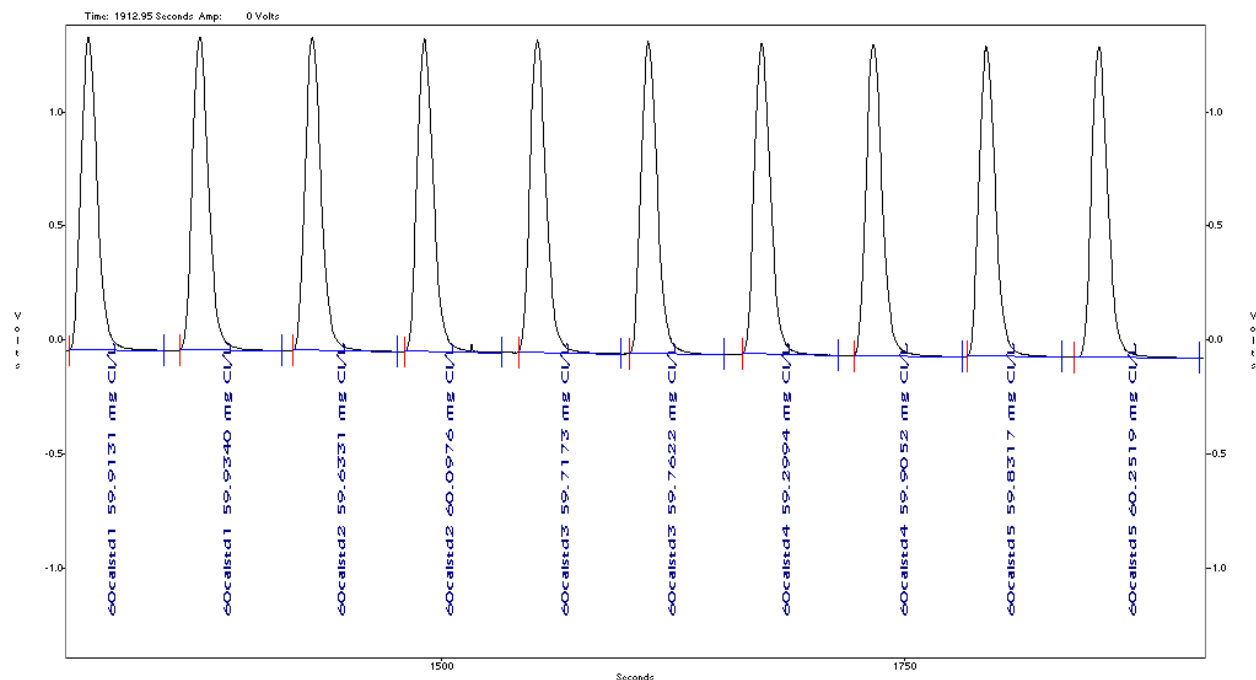
**% RSD = 0.915**

Standard Deviation (s) = 0.267 mg Cl/L, Mean (x) = 29.166 mg Cl/L, Known value = 30 mg Cl/L

File Name: Clprec21.fdt

Acq. Date: 21 August 2001

### Precision data for chloride using 60 mg Cl/L standard



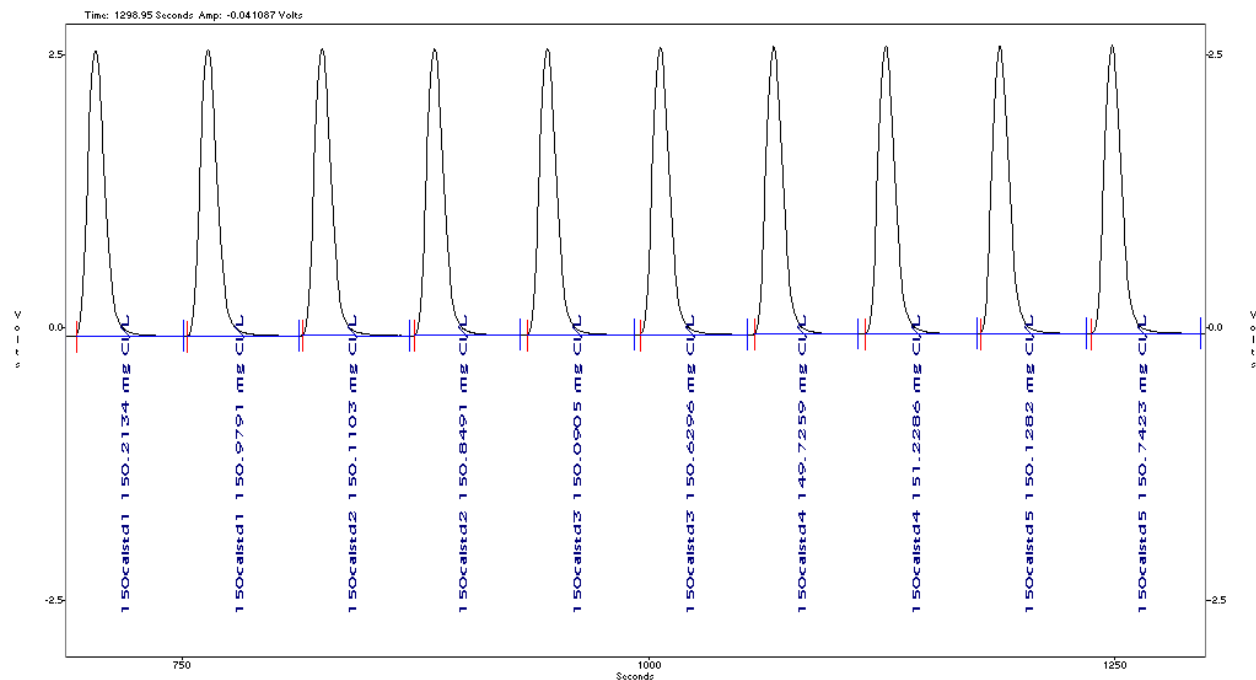
**% RSD = 0.435**

Standard Deviation (s) = 0.2604 mg Cl/L, Mean (x) = 59.835 mg Cl/L, Known value = 60 mg Cl/L

File Name: Clprec21.fdt

Acq. Date: 21 August 2001

### Precision data for chloride using 150 mg Cl/L standard



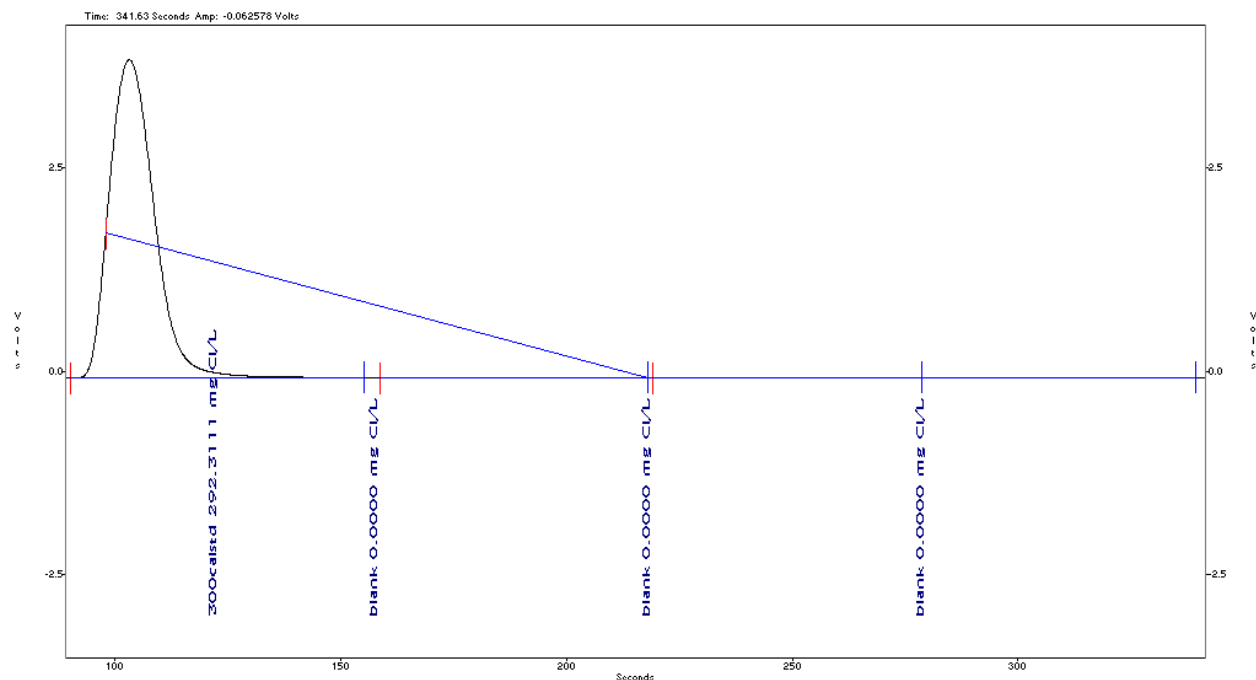
**% RSD = 0.3200**

Standard Deviation (s) = 0.48165 mg Cl/L, Mean (x) = 150.4697 mg Cl/L, Known value = 150 mg Cl/L

File Name: Clprec21.fdt

Acq. Date: 21 August 2001

### Carryover Study: 300 mg Cl/L standard followed by 3 blanks



**Carryover Passed**

File Name: cryover21.fdt

Acq. Date: 21 August 2001

# Fluoride

## QuikChem<sup>®</sup> Method 10-109-12-2-A

SM 4500 F<sup>-</sup> G. Ion Selective Electrode Flow Injection Analysis

### Fluoride in Water and Wastewater

0.1 to 5.0 mg F/L

#### – Principle –

Fluoride is determined potentiometrically using a combination fluoride electrode and the Lachat QuikChem Flow Injection Analyzer. The fluoride electrode consists of a lanthanum fluoride crystal across which a potential is developed by fluoride ions. The reference cell is a Ag/AgCl/Cl<sup>-</sup> cell. The reference junction is of the annular liquid-junction type and encloses the fluoride-sensitive crystal.

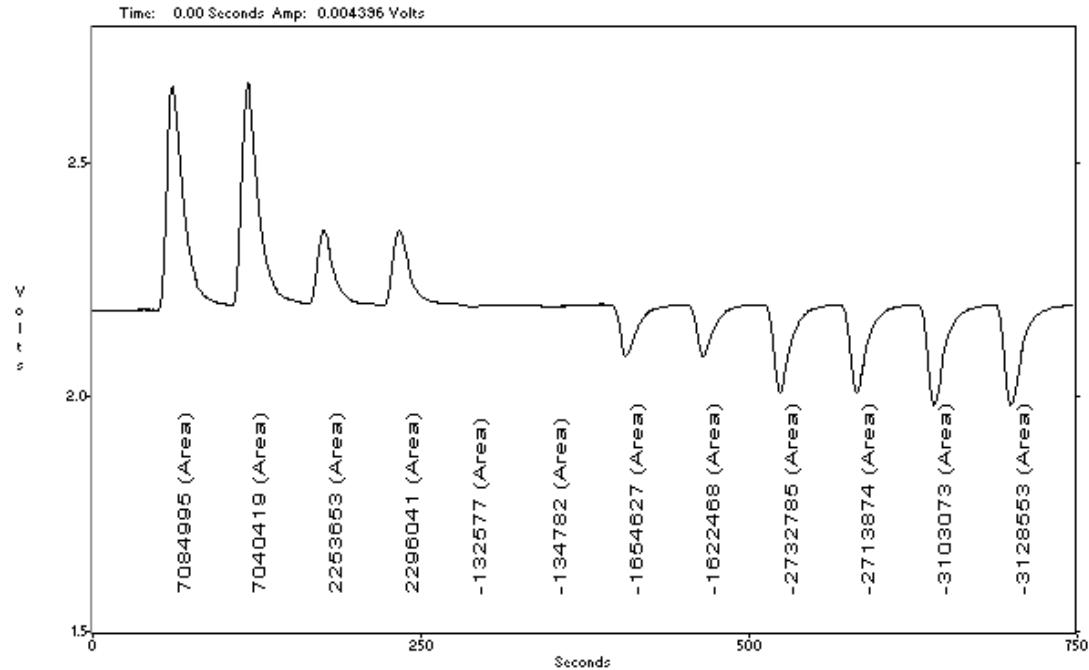
#### – Interferences –

1. The polyvalent cations, Si<sup>4+</sup>, Al<sup>3+</sup>, and Fe<sup>3+</sup>, interfere by forming complexes with fluoride. CDTA (1,2-cyclohexylene dinitrilotetracetic acid) is added to preferentially complex these cations and eliminate this interference when these concentrations do not exceed 3.0 mg Al<sup>3+</sup>/L and 20 mg Fe<sup>3+</sup>/L.
2. For US users determining Total or Total Dissolved Fluoride, the Bellack distillation (see Standard Methods in Sources below) is required for NPDES monitoring but is not required for SDWA monitoring.

#### – Special Apparatus –

Please contact Lachat Technical Support for Ordering Information

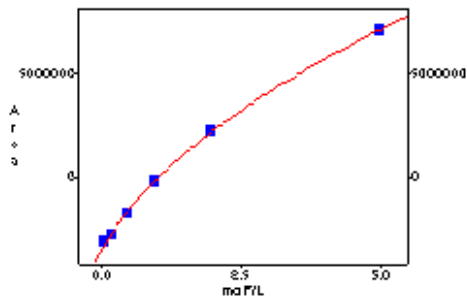
1. Lachat Fluoride Module (Lachat Part No. 84964)



File Name: msdcali.fdt

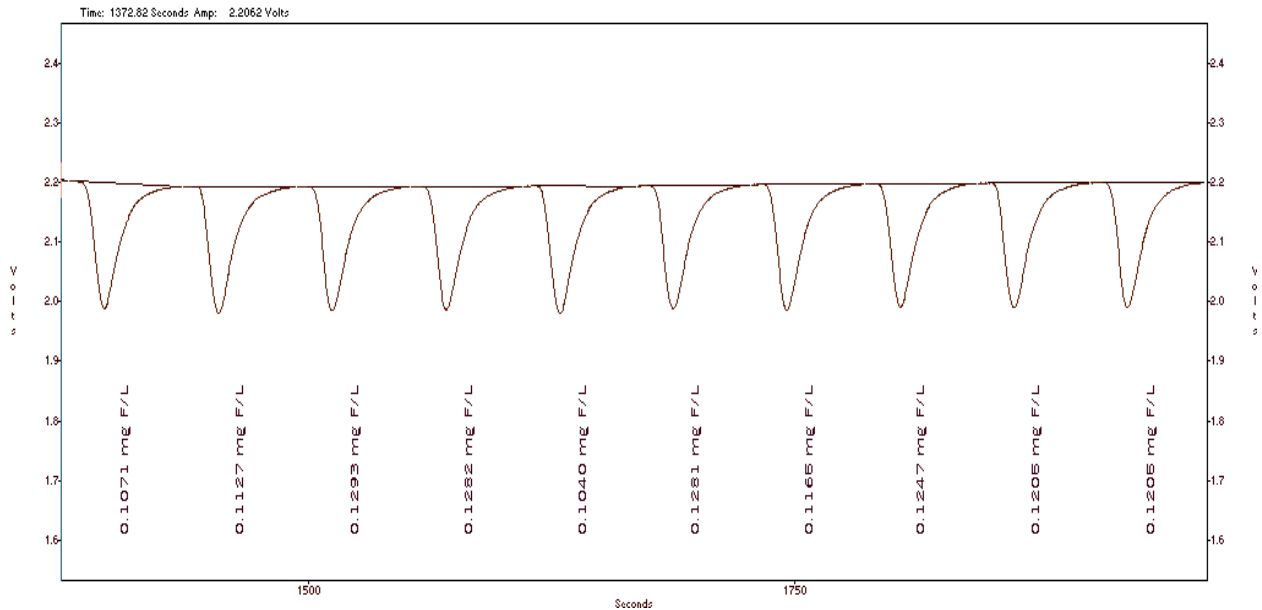
### Calibration Graph and Statistics

Level	Area	mg F/L	Determined	Replicate %RSD	% residual
1	7062707	5.00	5.00	0.4	0
2	2274847	2.00	2.01	1.3	-0.3
3	-133680	1.00	1.01	0	-0.6
4	-1638548	0.50	0.56	-1.4	4.9
5	-2723330	0.20	0.20	-0.5	-0.1
6	-3115813	0.10	0.11	-0.6	-13



Scaling: None  
 Weighting: None  
 2nd order poly  
 $\text{conc.} = 2.484\text{e-}014\text{Area}^2 + 3.740\text{e-}007\text{Area} + 1.039$   
 $R^2 = 0.9999$

### Method Detection Limit for Fluoride using 0.1 mg F/L standard

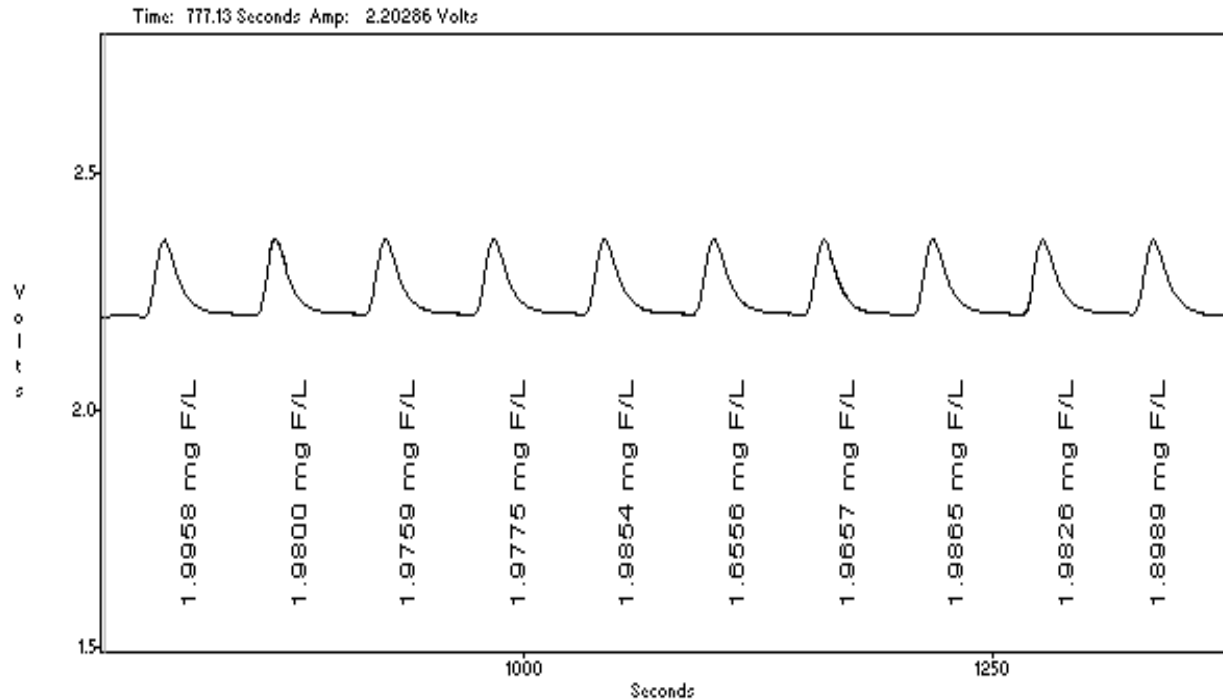


**MDL = 0.05 mg F/L claimed MDL due to carryover**

Standard Deviation (s) = 0.0089 mg F/L, Mean (x) = 0.119 mg F/L, Known value = 0.1 mg F/L

File Name: msdall.fdt

### Precision data for Fluoride using 2.0 mg F/L standard

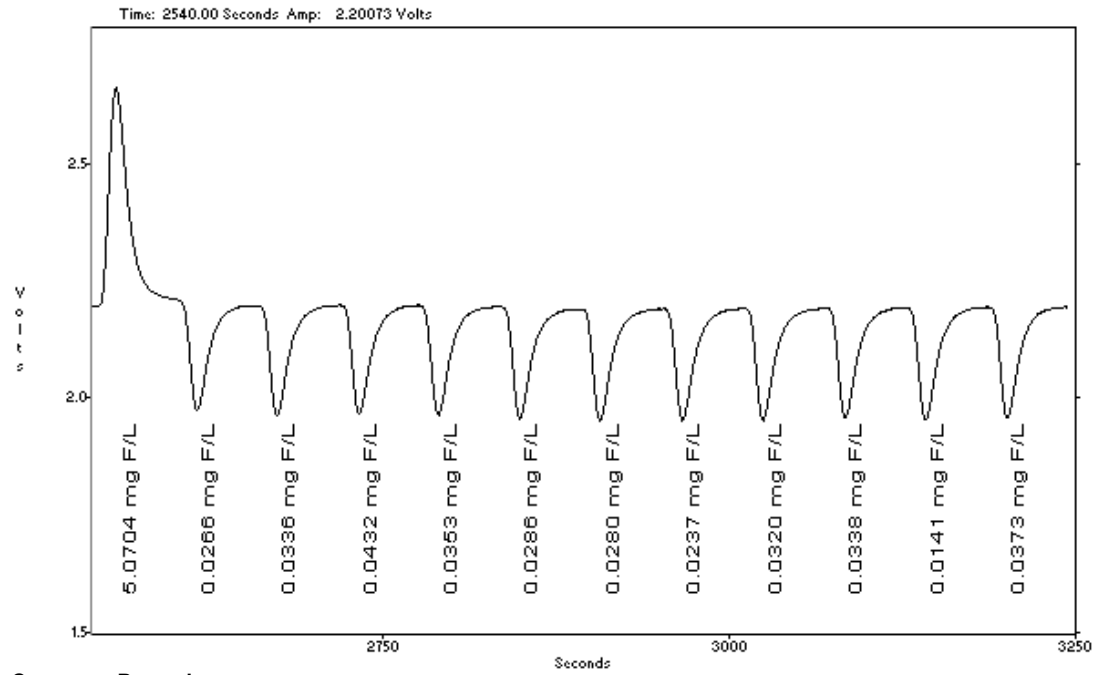


**% RSD = 0.5**

Standard Deviation (s) = 0.0103 mg F/L, Mean (x) = 1.94 mg F/L, Known value = 2.0 mg F/L

Acq. Date: msdall.fdt

### Carryover Study: 5.0 mg F/L standard followed by 11 blanks



**Carryover Passed**  
Acq. Date: msdall.fdt

# Total Nitrogen by In-Line UV/Persulfate Digestion and Oxidation with Flow Injection Analysis

**QuikChem<sup>®</sup> Method 10-107-04-3-P**  
SM 4500-N B.

## Total Nitrogen, In-line Persulfate Digestion

0.2 to 10.0 mg N/L

### – Principle –

Nitrogen compounds are oxidized in-line to nitrate using alkaline persulfate/UV digestion. Oxidation of nitrogen containing compounds to nitrate is achieved at 105°C with additional energy supplied by exposure to UV light. The digestion occurs prior to the injection valve.

Results for wastewater influent may be up to 30% low when compared with a rigorous TKN digestion because of sediment in the sample test tube. If effluent samples are preserved and filtered, in-line digestion results will match the manual off-line digestion. If samples are not filtered, in-line results will be 1-15% low compared with off-line digestion. Surface water samples may not require filtration but this should be verified with a sample containing high levels of solids.

After digestion 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 coupled with N-(1-naphthyl)ethylenediamine dihydrochloride. The resulting pink dye absorbs at 540 nm and is proportional to total nitrogen.

### – Interferences –

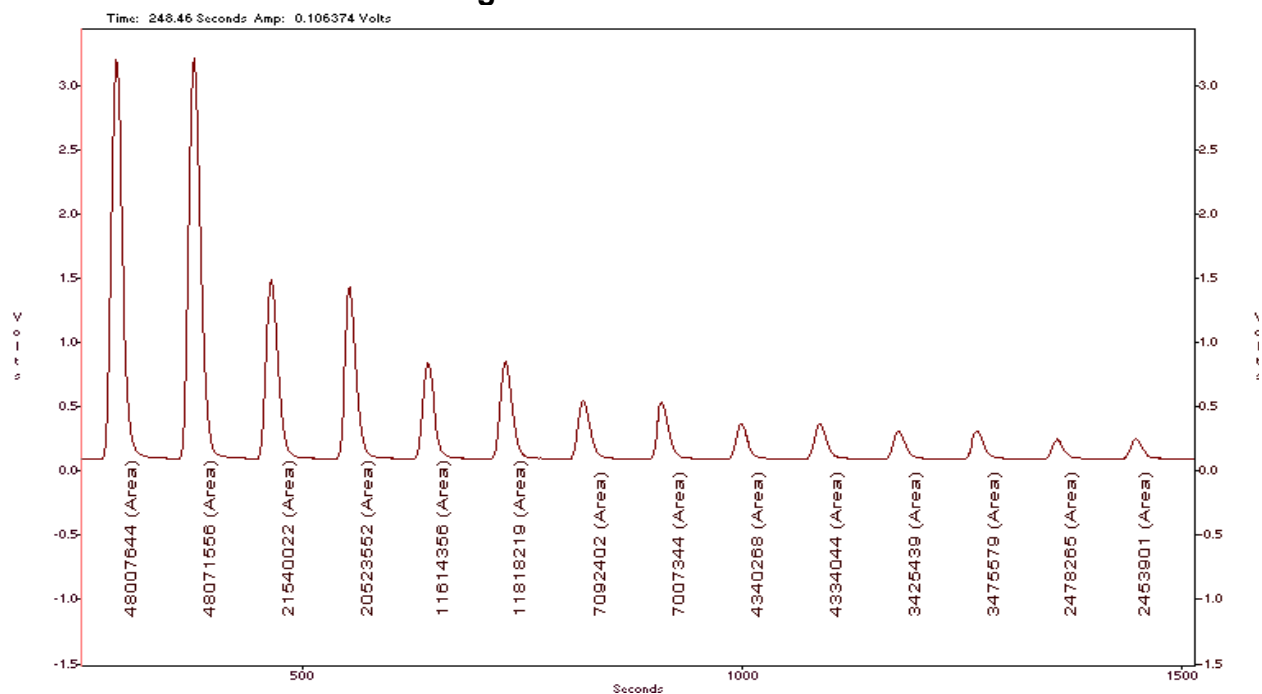
1. Chloride is a suspected interference. Seawater, when spiked at 5 mg N/L as ammonia, gave < 5% recovery.

### – Special Apparatus –

Please contact Lachat Technical Support for Ordering Information

1. Lachat Sample Preparation Module, A30X11 (X=1 for 110V, x=2 for 220V) with UV-254 lamp.
2. PVC PUMP TUBES MUST BE USED FOR THIS METHOD

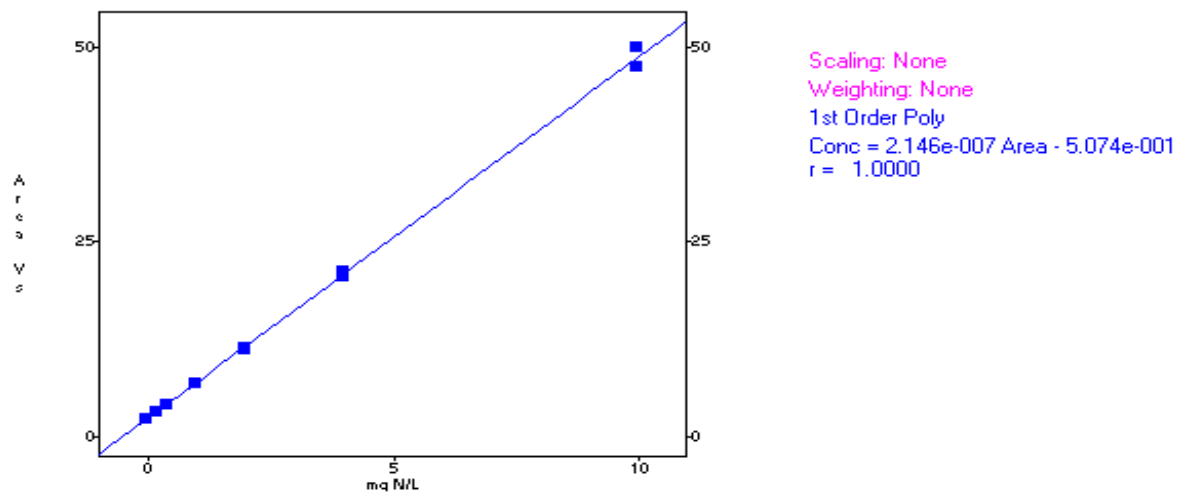
## Calibration Data for Total Nitrogen



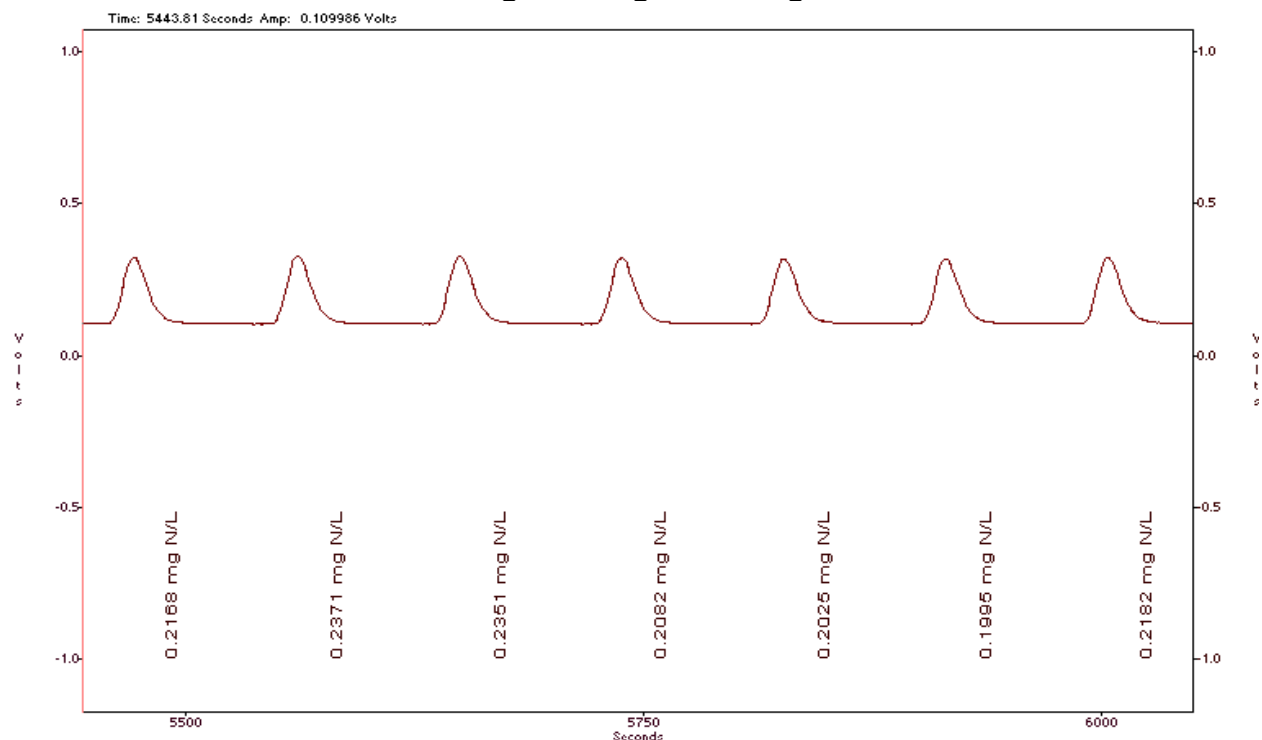
Method File Name: Inlnmsd3.fdt  
Acq. Date: 22 October 1998

## Calibration Graph and Statistics

Level	Area	mg N/L	Determined	Replicate %RSD	% residual
1	48980184	10.0	10.01	3.7	-0.1
2	21048072	4.00	4.01	2.5	-0.3
3	11499288	2.00	1.96	1.1	2.0
4	6922639	1.00	0.98	0.6	2.2
5	4219699	0.40	0.39	2.1	0.4
6	3378857	0.20	0.22	0.8	-8.9
7	2501036	0.00	---	0.9	---



### Method Detection Limit for Nitrogen using a 0.20 mg N/L standard

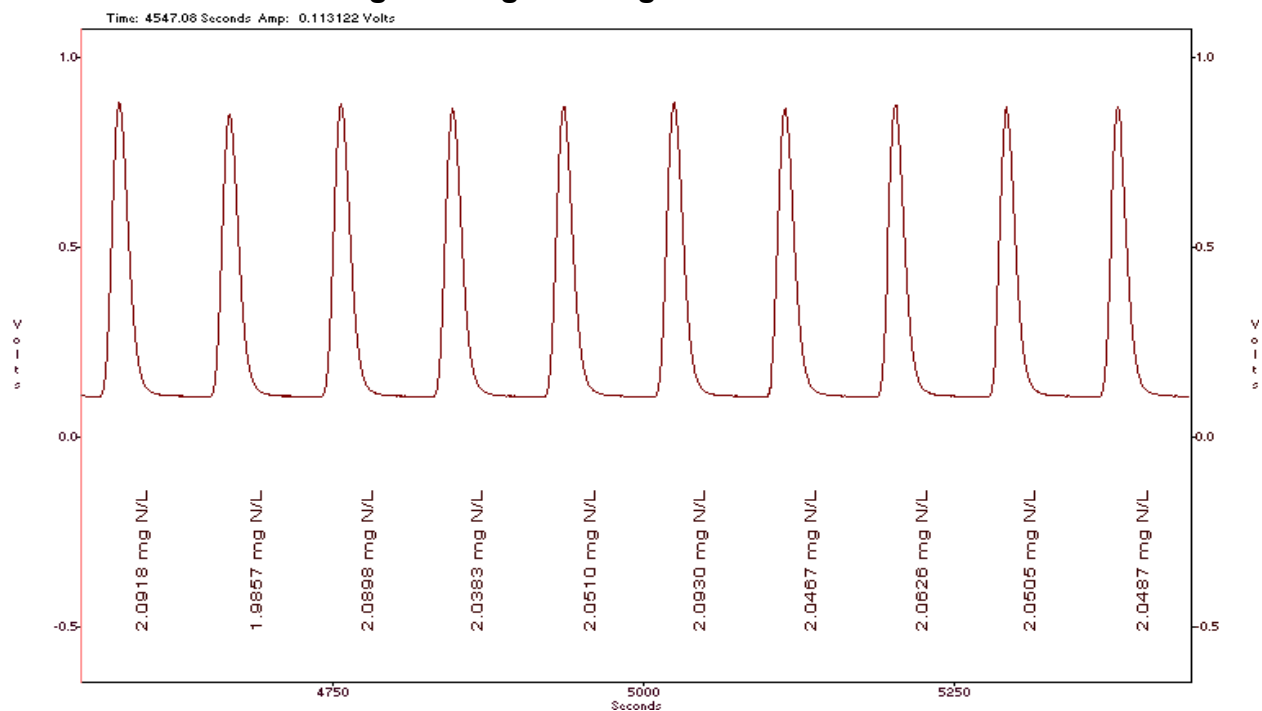


**MDL = 0.046 mg N/L**

Standard Deviation (s) = 0.015 mg N/L, Mean (x) = 0.217 mg N/L, Known Value = 0.20 mg N/L

Acq. Date: 22 October 1998

### Precision Data for Nitrogen using a 2.0 mg N/L standard

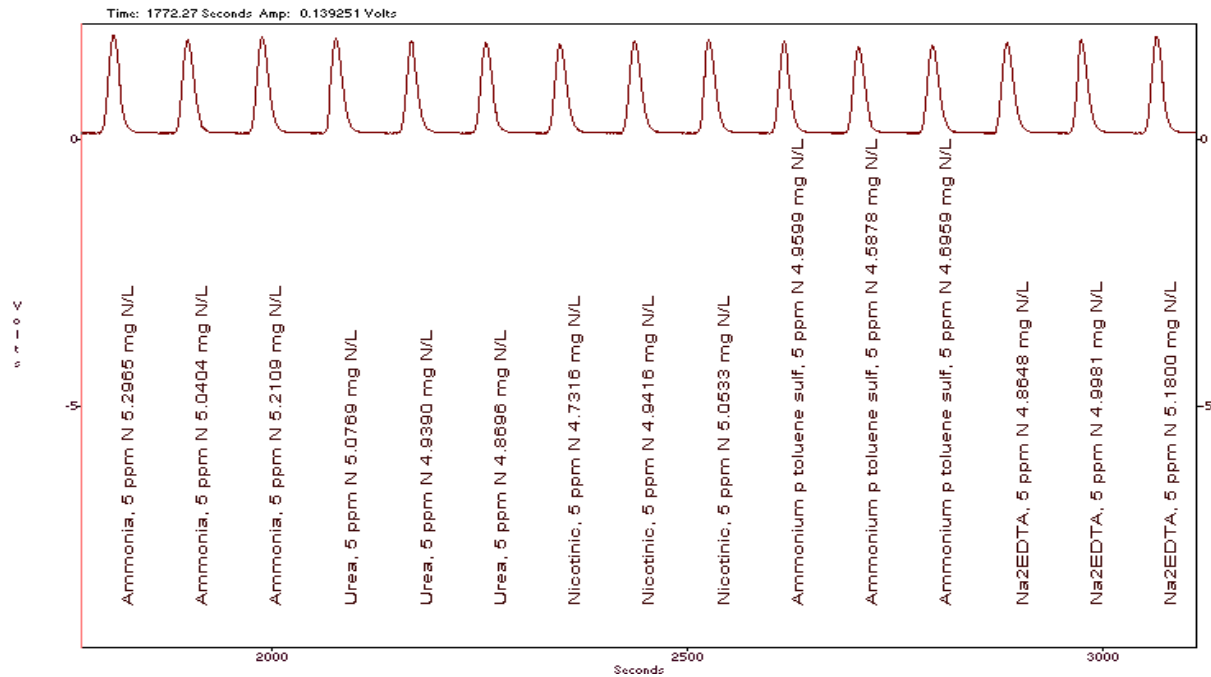


**% RSD = 1.56**

Standard Deviation (s) = 0.032 mg N/L, Mean (x) = 2.055 mg N/L, Known Value = 2.0 mg N/L

Acq. Date: 22 October 1998

### Digestion Efficiency for Nitrogen containing compounds at 5 mg N/L

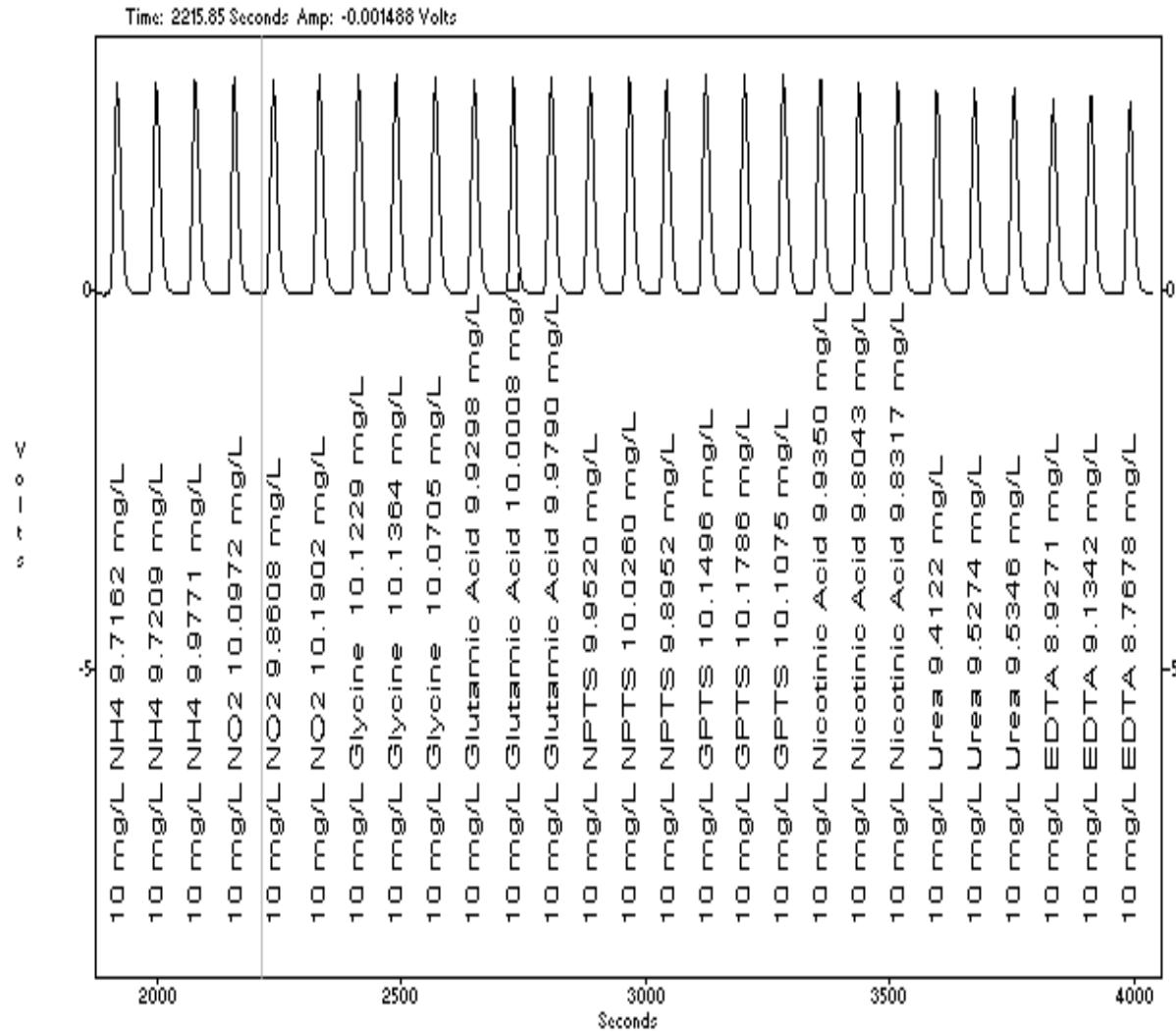


Acq. Date: 22 October 1998

Nitrogen Form	Mean Result (mg N/L)	%Recovery
Ammonia	5.18	103.7
Urea	4.96	99.24
Nicotinic Acid	4.90	98.18
NPTS*	4.75	94.96
EDTA	5.01	100.3

\* Ammonium p-toluenesulfonate

### Digestion Efficiency for Nitrogen containing compounds at 10 mg N/L



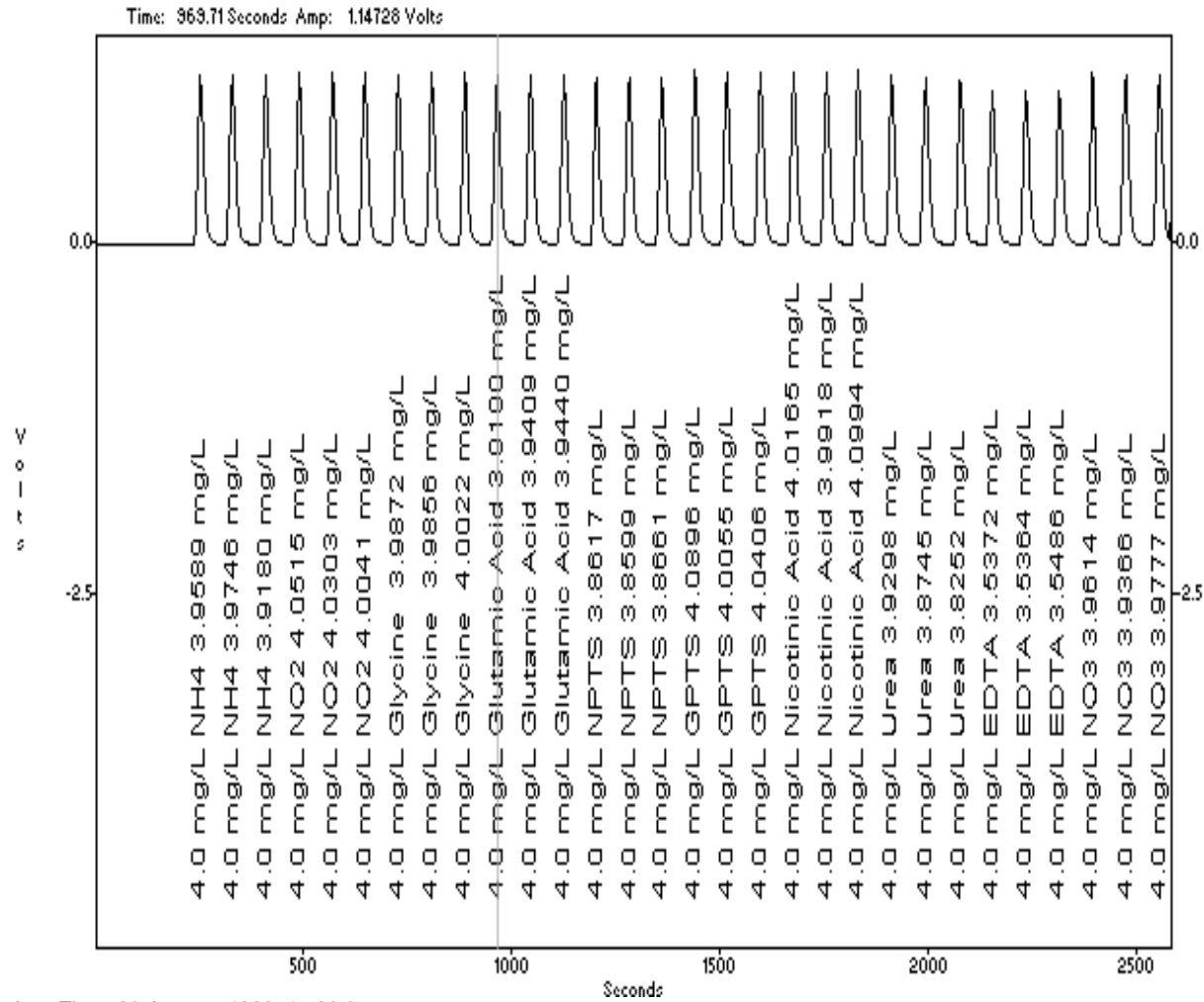
Acq. Time: 30 January 1996, 13:59:36

Nitrogen Form	Mean Result (mg N/L)	%Recovery
Ammonia	9.81	98.1
Nitrite	10.05	100.5
Glycine	10.11	101.0
Glutamic Acid	9.97	99.7
NPTS*	9.96	99.6
GPTS**	10.14	101.4
Nicotinic Acid	9.86	98.6
Urea	9.49	94.9
EDTA	8.94	89.4

\* Ammonium p-toluenesulfonate

\*\* Glycine p-toluenesulfonate

### Digestion Efficiency for Nitrogen containing compounds at 4.00 mg N/L

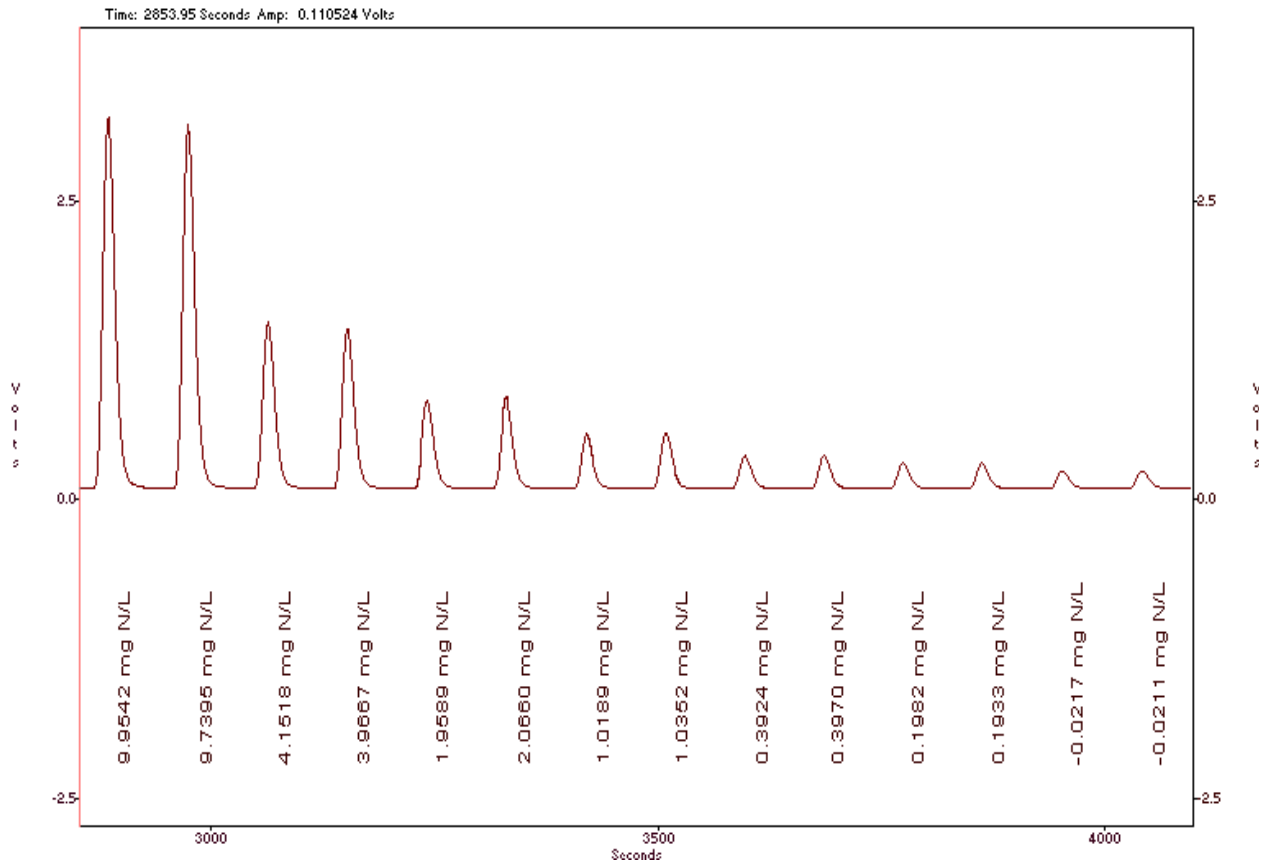


Nitrogen Form	Mean Result (mg N/L)	%Recovery
Ammonia	3.95	99.7
Nitrite	4.03	101.8
Glycine	3.99	100.8
Glutamic Acid	3.93	99.2
*NPTS	3.86	97.4
**GPTS	4.05	102.3
Nicotinic Acid	4.04	102.0
Urea	3.88	98.0
EDTA	3.54	89.4

\* Ammonium p-toluenesulfonate

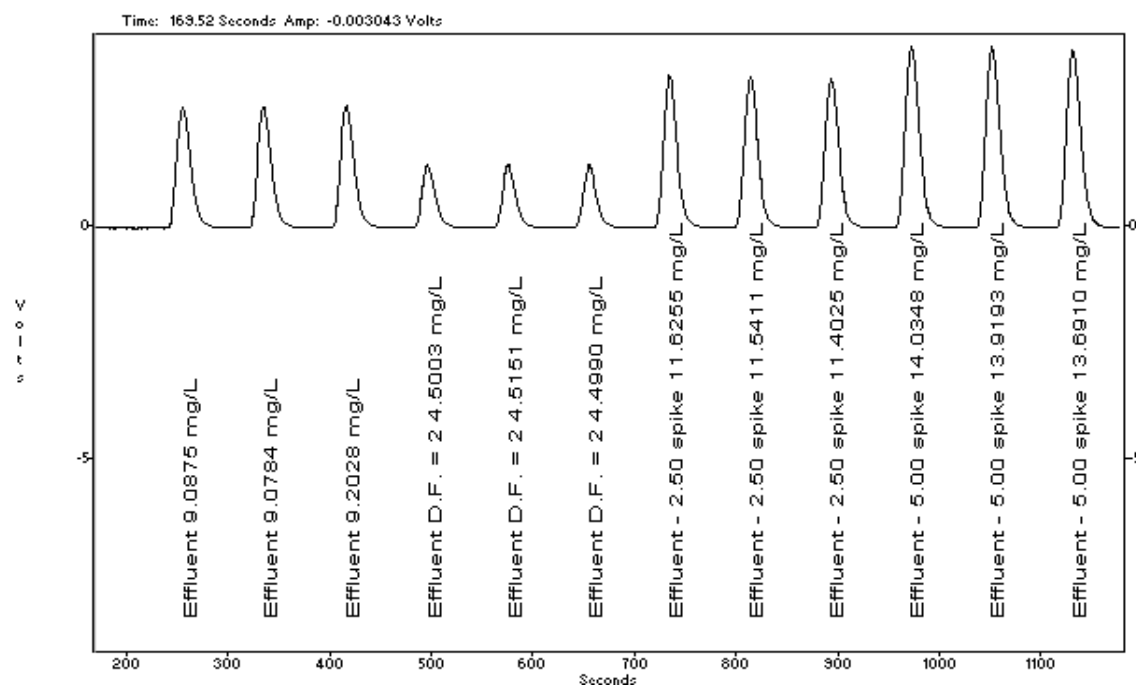
\*\* Glycine p-toluenesulfonate

### Accuracy for calibration standards run as unknown samples



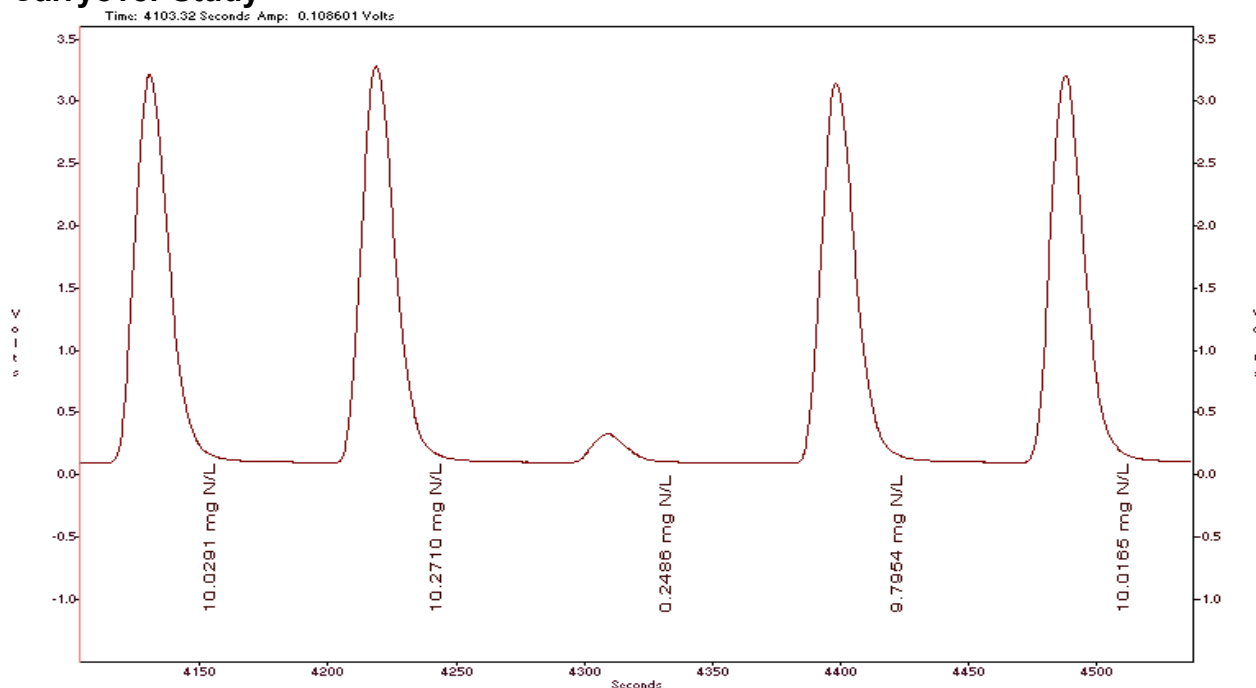
Known Concentration mg N/L	Average mg N/L	Standard Deviation mg N/L	% Residuals	%RSD
10.0	9.84	0.15	-1.6	1.54
4.0	4.05	0.13	1.25	3.22
2.0	2.01	0.07	0.5	3.76
1.0	1.02	0.01	2.0	1.12
0.4	0.39	0.003	-2.5	0.82
0.2	0.19	0.003	-5.0	1.78
0.0	-0.02	0.0004	---	---

### Wastewater Study: Dilution and Spike of Milwaukee Effluent



	Effluent	Effluent Dilution (DF=2)	Effluent Spike at 2.50 mg N/L	Effluent Spike at 5.00 mg N/L
Mean (mg/L)	9.12	4.51	11.52	13.88
% Recovery	--	98.9	96.0	95.2

### Carryover Study



Two 10.0 mg N/L standards followed by 0.2 mg N/L standard followed by two 10 mg N/L standards

**Carryover Passed**

Acq. Date: 22 October 1998

# Nitrate/Nitrite by Cadmium Reduction in Flow Injection Analysis

**QuikChem<sup>®</sup> Method 10-107-04-1-C**  
SM 4500-NO<sub>3</sub>- I.

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

High Throughput Method

0.01 to 2.0 mg N/L as NO<sub>3</sub><sup>-</sup> + NO<sub>2</sub><sup>-</sup>  
or NO<sub>2</sub><sup>-</sup>

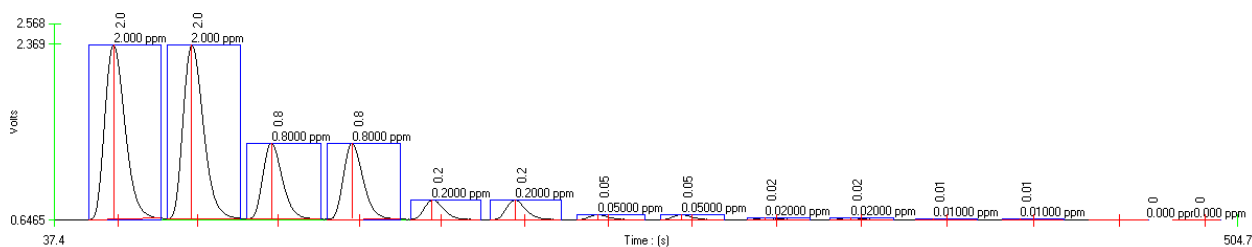
### – Principle –

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. Nitrate may then determined by subtraction.

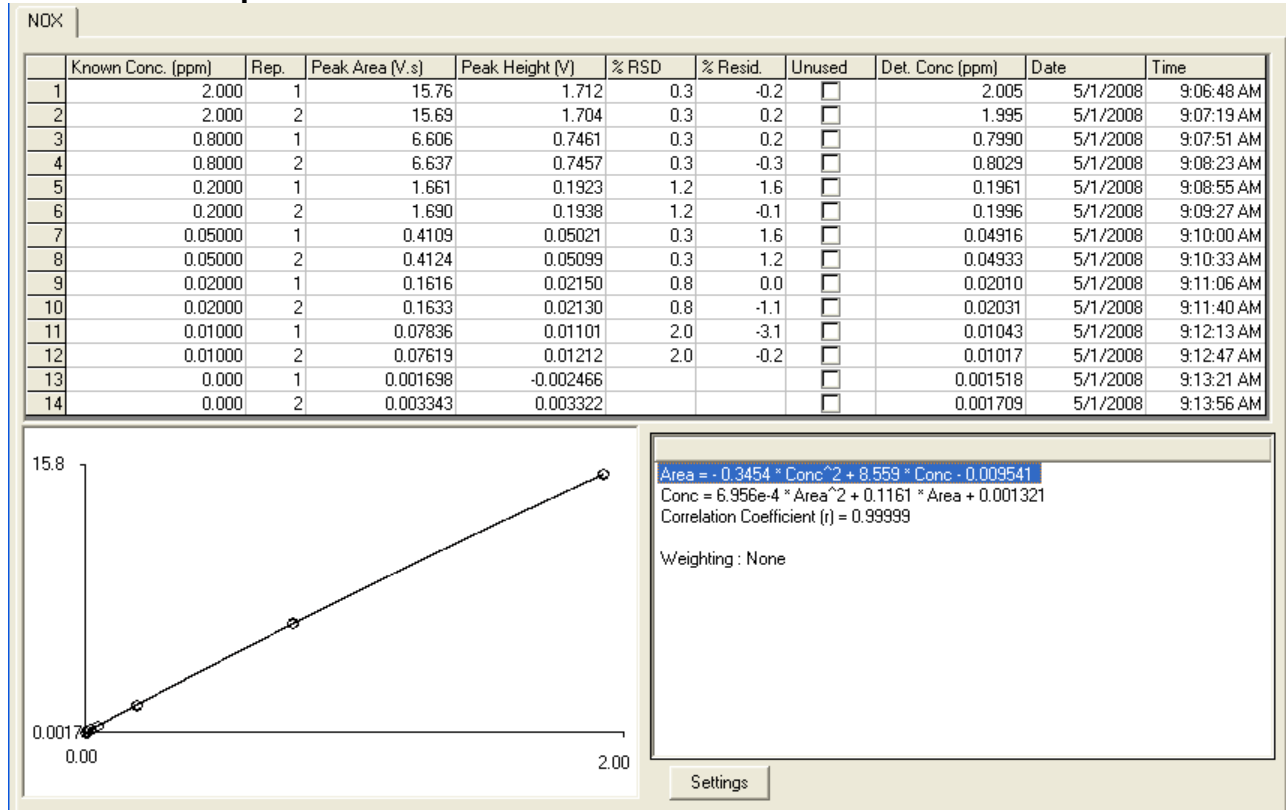
### – Interferences –

1. Residual chlorine can interfere by oxidizing the cadmium column.
2. Low results could 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.

### Calibration Data for Nitrate/Nitrite

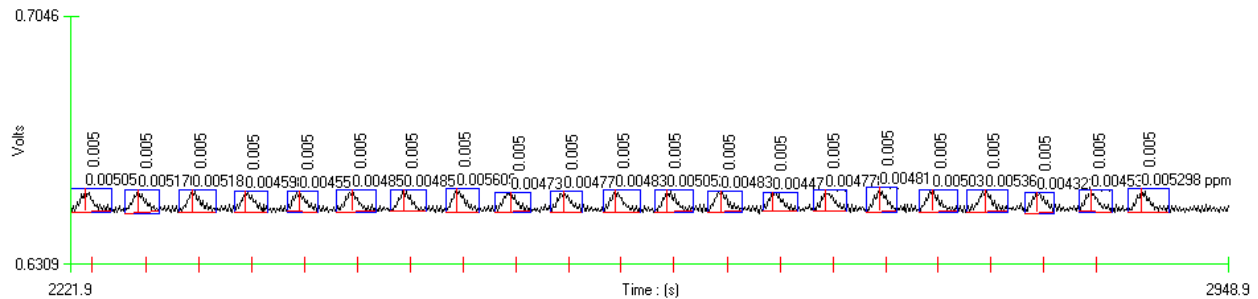


### Calibration Graph and Statistics



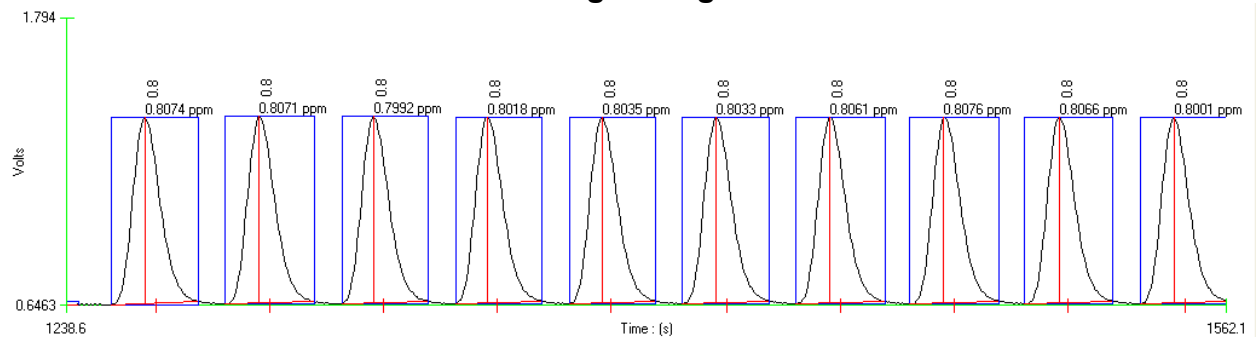
File Name: OM\_5-01-2008\_09-06-12AM.OMN  
 Acq Date: 01 May 2007

### Method Detection Limit for nitrate + nitrite using 0.005 mg/L standard



**MDL = 0.001 mg N/L**  
 Standard Deviation (s) = 0.00032 mg N/L, Mean (x) = 0.004892 mgN/L, Known value = 0.005 mg N/L  
 File Name: OM\_5-01-2008\_11-09-07AM.OMN  
 Acq Date: 01 May 2007

### Precision data for nitrate + nitrite using 0.8 mg N/L standard



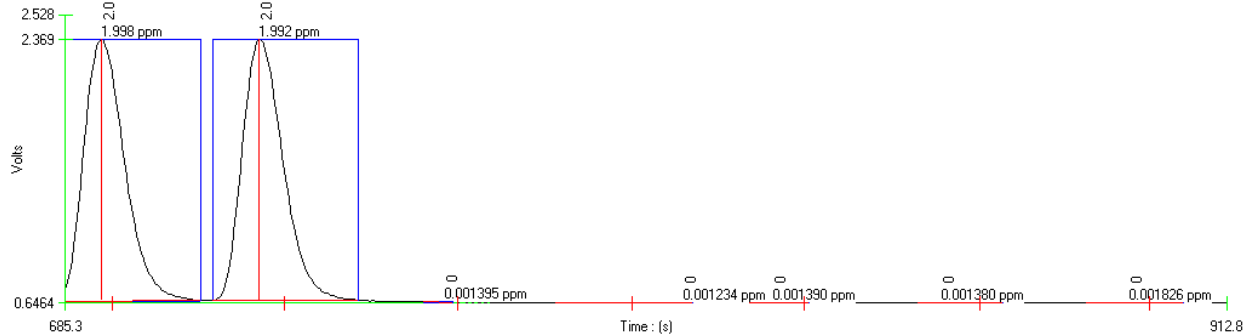
**% RSD = 0.39**

Standard Deviation (s) = 0.0031 mg N/L, Mean (x) = 0.8043 mg N/L, Known value = 0.8 mg N/L

File Name: OM\_5-01-2008\_09-06-12AM.OMN

Acq Date: 01 May 2007

### Carryover Study: 2.0 mg N/L standard followed by 5 blanks

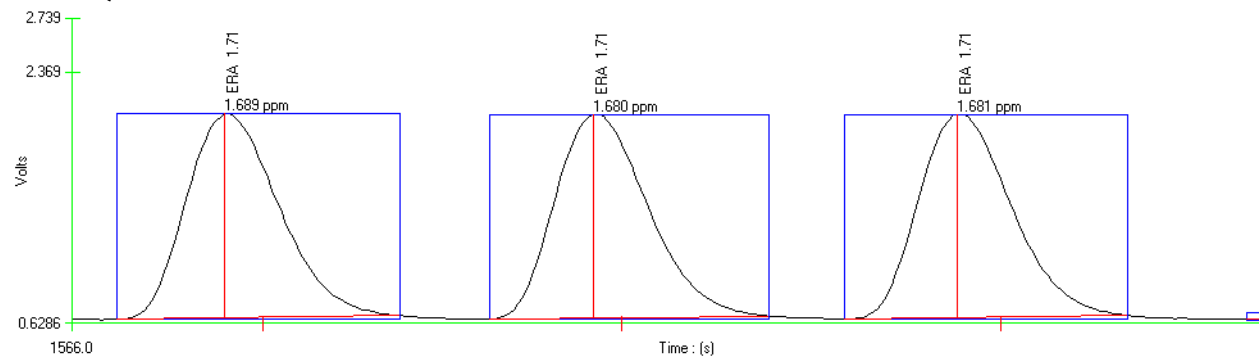


**Carryover Passed**

File Name: OM\_5-01-2008\_09-06-12AM.OMN

Acq Date: 01 May 2007

### ERA QC Standard



File Name: OM\_5-01-2008\_09-06-12AM.OMN

Acq Date: 01 May 2007

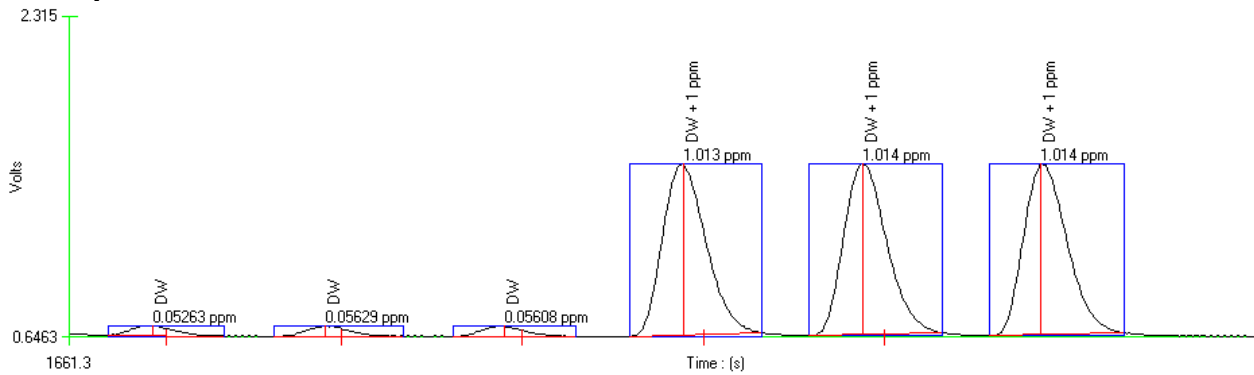
ERA QC Standard WasteWatR Simple Nutrients, Catalog  
no. 505, Lot No. P148505  
ERA, Arvada, Colorado, US Phone: 303-431-8454  
Mean Determined Concentration: **1.683 mg N/L**

**Known Concentration:** 1.71 mg N/L  
Interlaboratory Acceptance Range: **1.54 –1.95** mg N/L



**ENVIRONMENTAL  
RESOURCE  
ASSOCIATES**  
The Industry Standard™

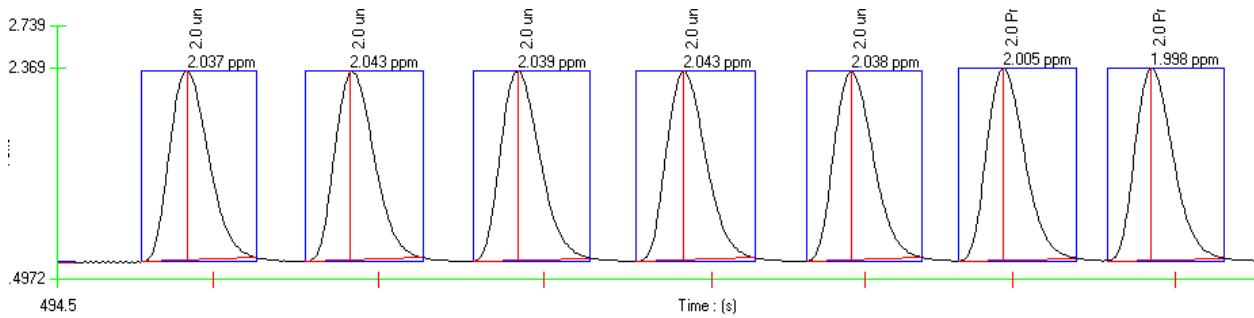
### DW Spike



Sample	Unspiked	Spiked	%Recovery
Loveland Tap	0.0563 mg N/L	1.0137 mg N/L	<b>95.97%</b>

File Name: OM\_5-01-2008\_09-06-12AM.OMN  
Acq Date: 01 May 2007

### Preserved versus un-preserved standard at 2.0 mg N/L

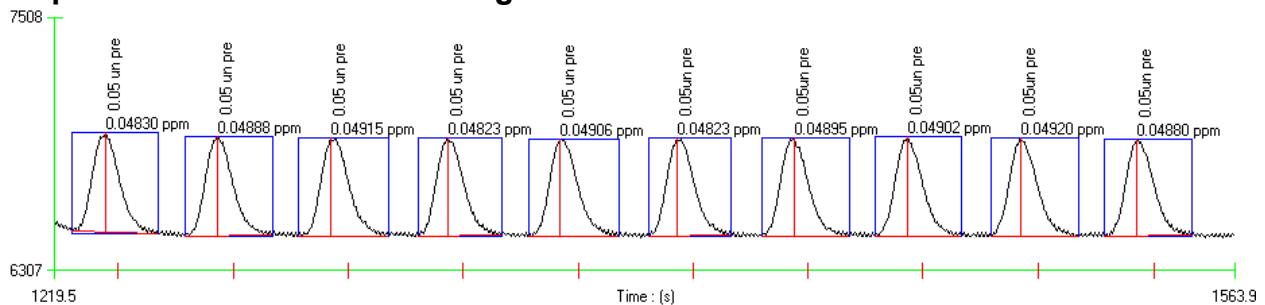


Acq Date: 01 May 2007

Preserved 2.0 mg N/L	Unpreserved 2.0 mg N/L	% Difference
2.0015	2.04	<b>+1.92%</b>

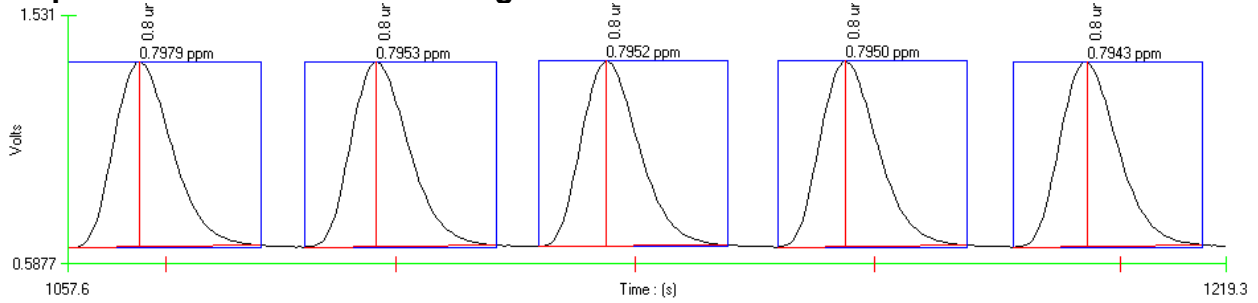
**Conclusion:** High buffer flow can accommodate preserved or un-preserved samples using this method.  
File Name: OM\_5-01-2008\_09-06-12AM.OMN

### Unpreserved standard at 0.05 mg N/L



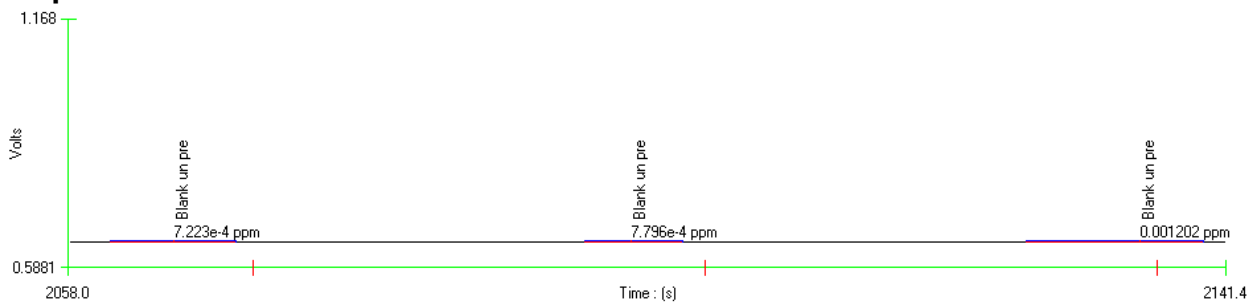
Average: 0.04873 mg N/L SD: 0.00044 mg N/L Known: 0.05 mg N/L %Difference: **-2.5%**  
File Name: OM\_5-01-2008\_11-09-07AM.OMN  
Acq Date: 01 May 2007

### Unpreserved standard at 0.80 mg N/L



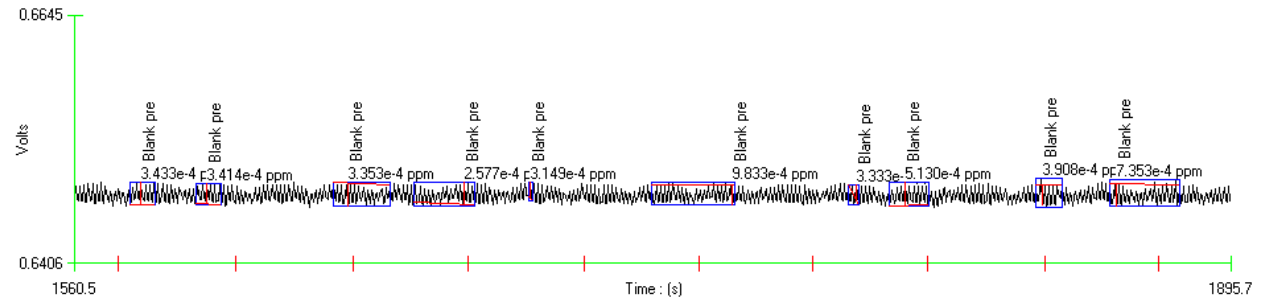
Average: 0.7955 mg N/L SD: 0.0014 mg N/L Known: 0.8 mg N/L %Difference: **-0.563%**  
 File Name: OM\_5-01-2008\_11-09-07AM.OMN  
 Acq Date: 01 May 2007

### Unpreserved blank



Average: 0.0009 mg N/L SD: 0.0 mg N/L Known: 0.0 mg N/L  
 File Name: OM\_5-01-2008\_11-09-07AM.OMN  
 Acq Date: 01 May 2007

### DIN Blanks



Average: 0.00455 mg N/L SD = 0.000231 mg N/L. Calculated DIN Limits: Detection Limit = 0.0007 mg N/L a, Decision Limit = 0.001 mg N/L, Determination Limit = 0.0063 mg

# Kjeldahl Nitrogen by Block Digestion and Flow Injection Analysis

**QuikChem<sup>®</sup> Method 10-107-06-2-H**  
SM 4500 N<sub>org</sub> D.

**Total Kjeldahl Nitrogen in Waters**  
Copper Catalyst/Block Digester Method

0.1 to 5.0 mg N/L

## – Principle –

This method covers the determination of Kjeldahl nitrogen using copper sulfate as the digestion catalyst. The method determines nitrogen in drinking water, ground water, and surface waters, as well as domestic and industrial wastes. The digestion procedure converts nitrogen compounds of biological origin such as amino acids, proteins and peptides to ammonia. Nitrogenous compounds of some industrial wastes such as amines, nitro compounds, hydrazones, oximes, semicarbazones and some refractory tertiary amines may not be recovered. Nitrate is not recovered.

The colorimetric method is based on the formation of chloramine, followed by a coupling reaction which forms indophenol blue at pH >10.5. Nitroprusside is added as a catalyst.

## – Interferences –

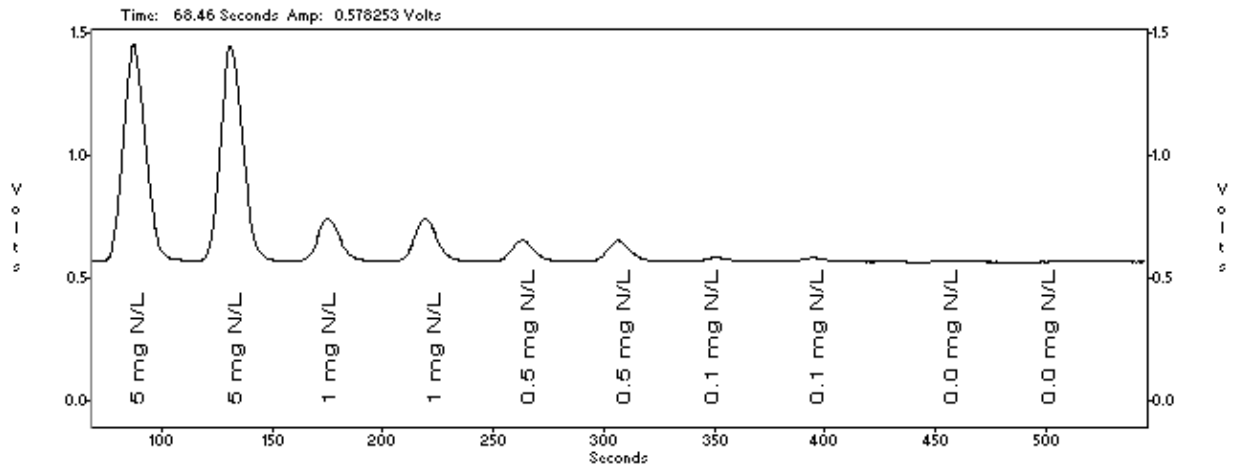
1. Samples must not consume more than 10% of the sulfuric acid during the digestion. The buffer will accommodate a small range of H<sub>2</sub>SO<sub>4</sub> concentrations in the digested, diluted samples with no change in signal intensity.

## – Special Apparatus –

Please contact Lachat Technical Support or Ordering Information

1. Heating Unit, Lachat Part No. A85X00 (X=1 for 110V, X=2 for 220V)
2. Block Digester/75 mL/BD46 Lachat Part No. A18206
3. PVC PUMP TUBES MUST BE USED FOR THIS METHOD

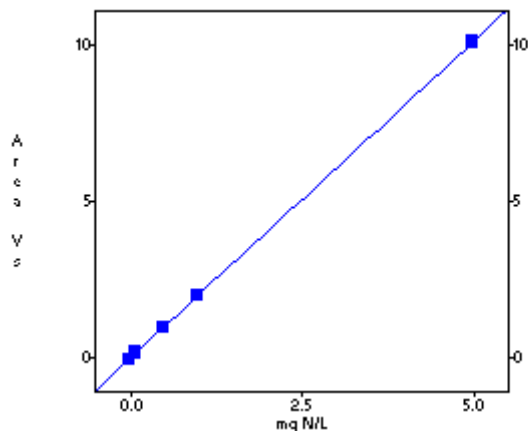
### Calibration Data for TKN



Method file name: 060695C1  
Acq. time: 06 June 1995 14:03:17

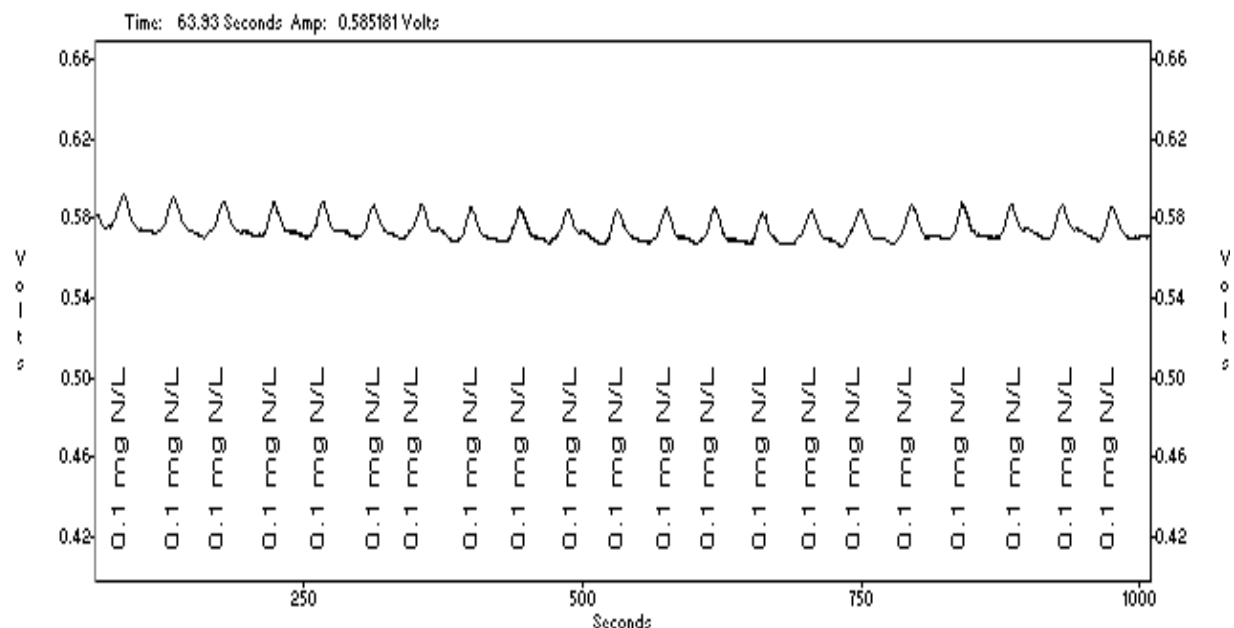
### Calibration Graph and Statistics

Level	Rep 1	Rep 2	mg N/L	Determined	Replicate %RSD	% residual
1	10182655	10171608	5.0	5.0	0.2	0.0
2	2046616	2030567	1.0	0.997	0.6	-0.3
3	1018479	1018834	0.5	0.4975	0.0	-0.5
4	213191	156723	0.1	0.0932	21.6	6.8
5	0	0	0.0	0.0	0.0	0.0



Scaling: None  
Weighting: None  
1st Order Poly  
Conc = 4.907e-007 Area + 2.405e-003  
 $R^2 = 1.0000$

### Method Detection Limit for TKN using a 0.1 mg N/L Standard

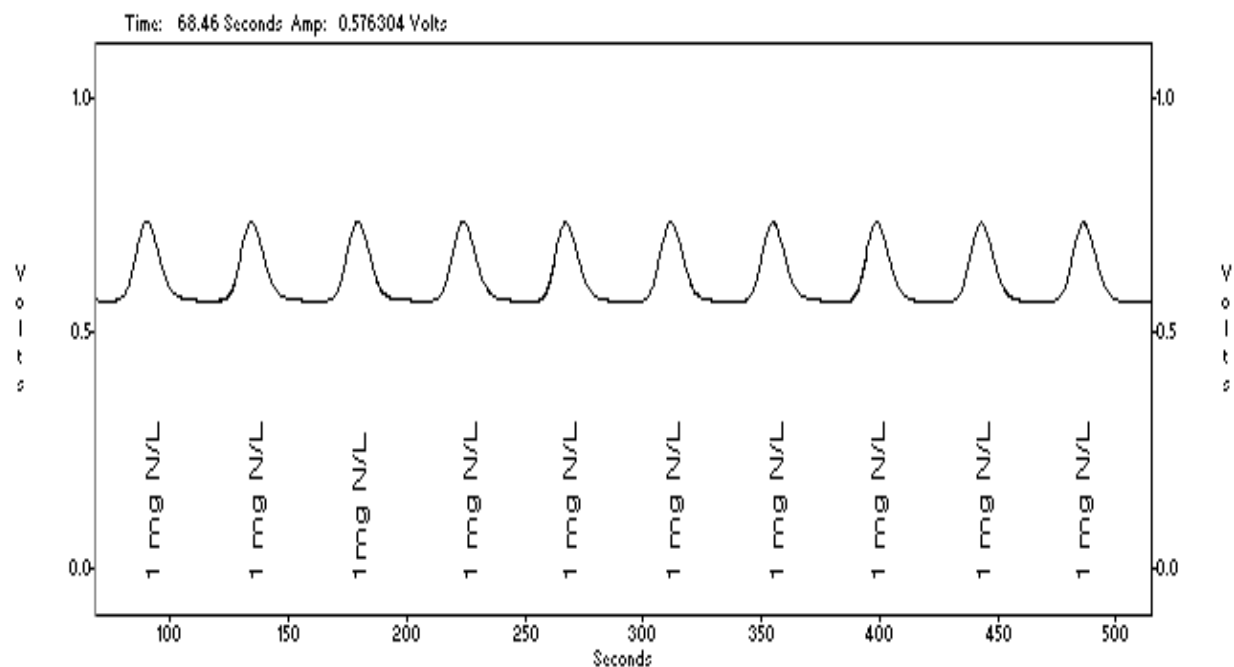


**MDL = 0.034 mg N/L**

Standard Deviation (s) = 0.0137 mg N/L, Mean (X) = 0.1029 mg N/L, Known Value = 0.100 mg N/L

ACQ. Time: 06 June 1995, 14:22:47

### Precision data for Total Kjeldahl Nitrogen, using a 1.00 mg N/L standard

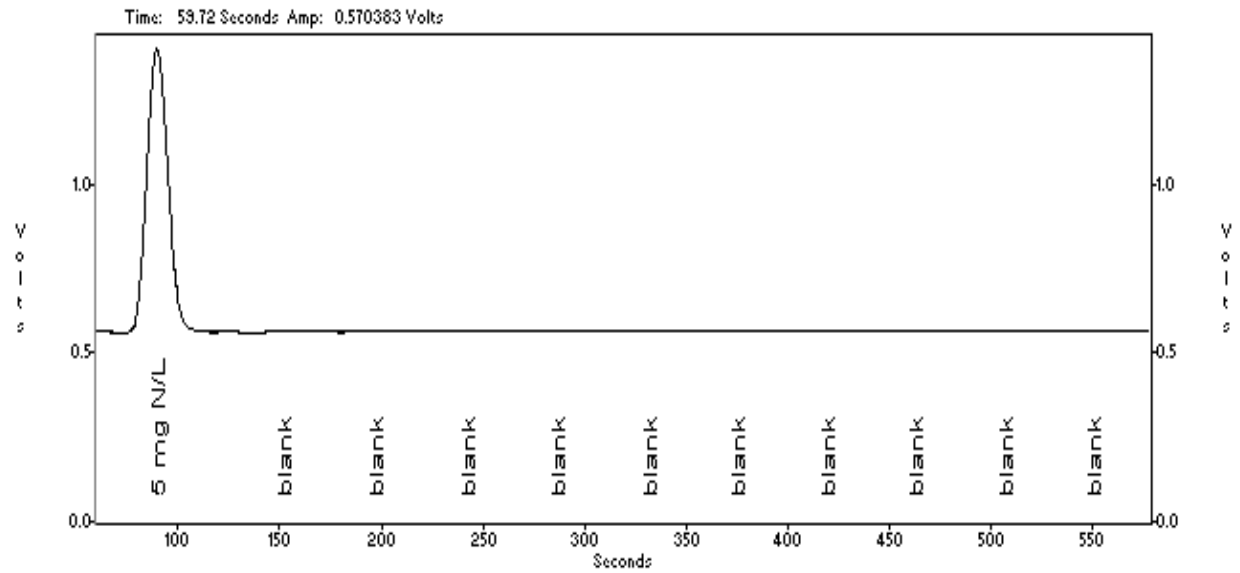


**%RSD = 1.49%**

Standard Deviation (s) = 0.014928 mg N/L, Mean (X) = 0.9995 mg N/L, Known Value = 1.00 mg N/L

ACQ. Time: 06 June 1995, 14:42:43

### Carryover Study: 5.0 mg N/L Standard followed by 10 blanks



**Carryover passed**  
ACQ Time: 06 June 1995, 14:56:23

- Method 10-107-06-2-l covers the range of 0.5-20 mg N/L in copper digests

# Orthophosphate

## QuikChem<sup>®</sup> Method 10-115-01-1-A SM 4500-P G.

### Orthophosphate in Waters

0.01 to 2.00 mg P/L

#### – 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 –

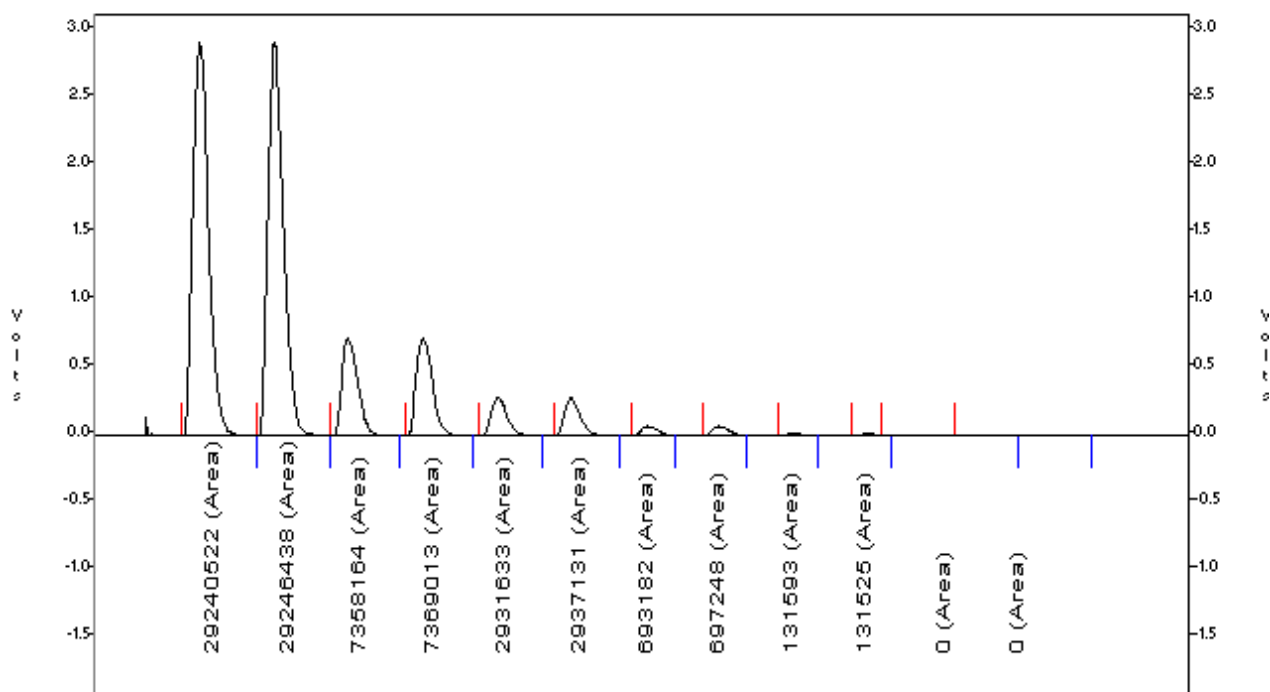
1. Silica forms a pale blue complex which also absorbs at 880 nm. This interference is generally insignificant as a silicate concentration of approximately 30 mg  $\text{SiO}_2/\text{L}$  would be required to produce a 0.005 mg P/L positive error in orthophosphate.
2. Concentrations of ferric iron ( $\text{Fe}^{3-}$ ) 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 Technical Support for Ordering Information

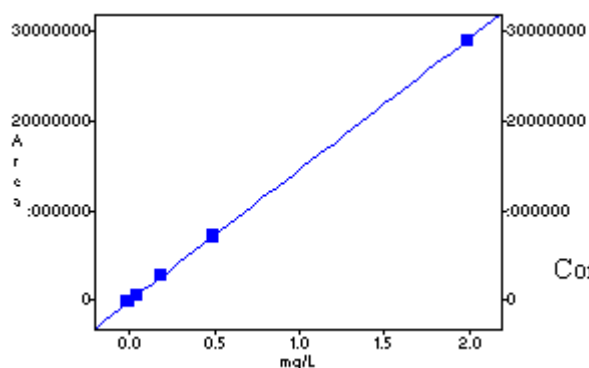
1. Heating Unit, Lachat Part No. A85X00 (X=1 for 110V, X=2 for 220V)

### Calibration Data for Orthophosphate



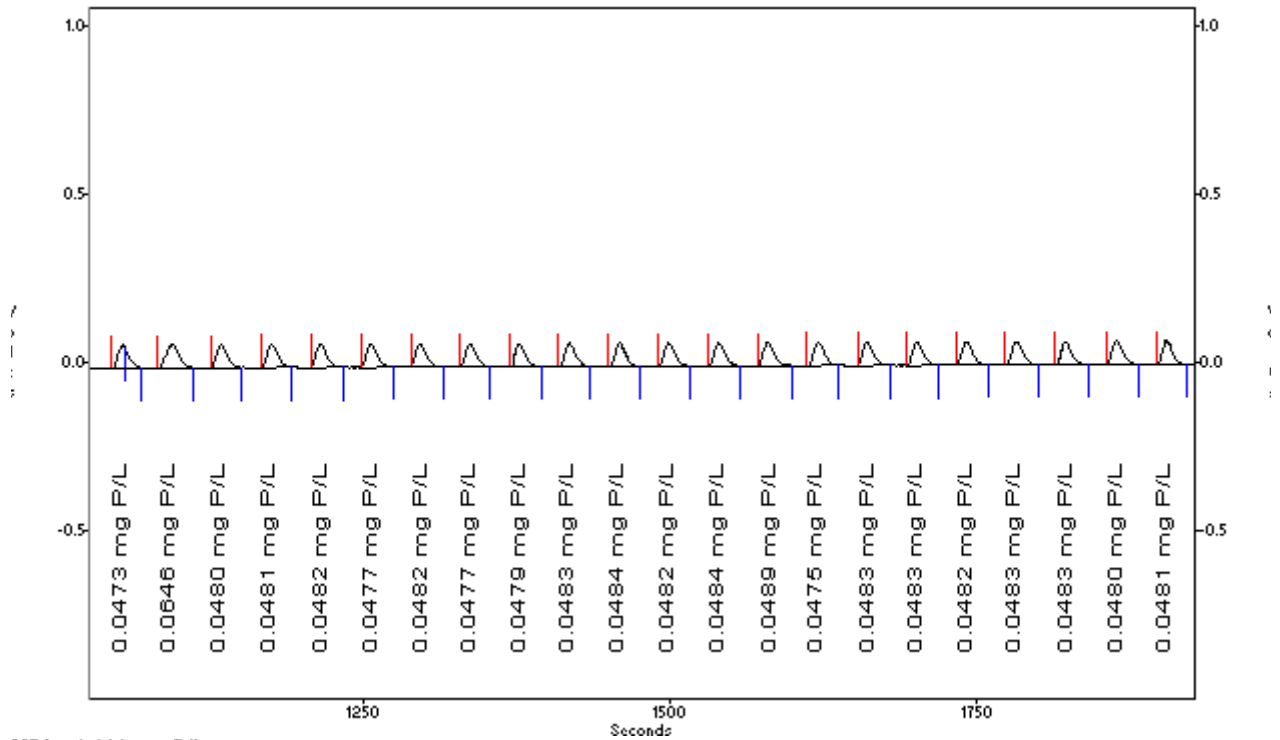
### Calibration Graph and Statistics

Standard (mg P/L)	Determined	RSD%	Residual%	Area 1	Area2
2.00	2.00	<0.1	0.0	29246438	29240522
0.50	0.50	0.1	-0.7	7369013	7358164
0.20	0.21	0.1	-0.3	2931633	2937131
0.05	0.05	0.4	4.8	697248	693182
0.01	0.01	<0.1	9.3	131525	131593
0.00	0.00	--	--	0	0



Scaling: none  
 Weighting: none  
 1st Order Poly  
 Conc =  $6.84e-8 \text{Area} + 8.02e-5$   
 $R^2 = 1.0000$

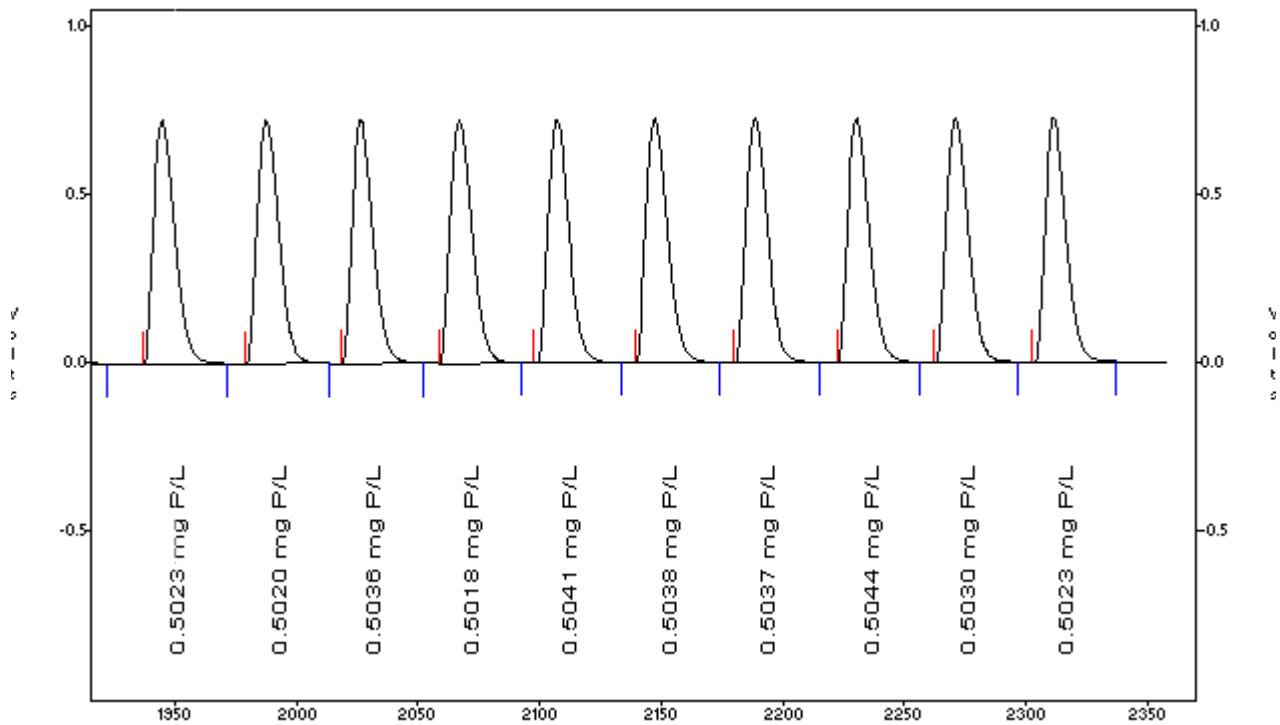
### Method Detection Limit for Orthophosphate using 0.05 mg P/L standard



**MDL = 0.009 mg P/L**

Standard Deviation (s) = 0.0036 units mg P/L, Mean (x) = 0.0489 mg P/L, Known value = 0.05 mg P/L

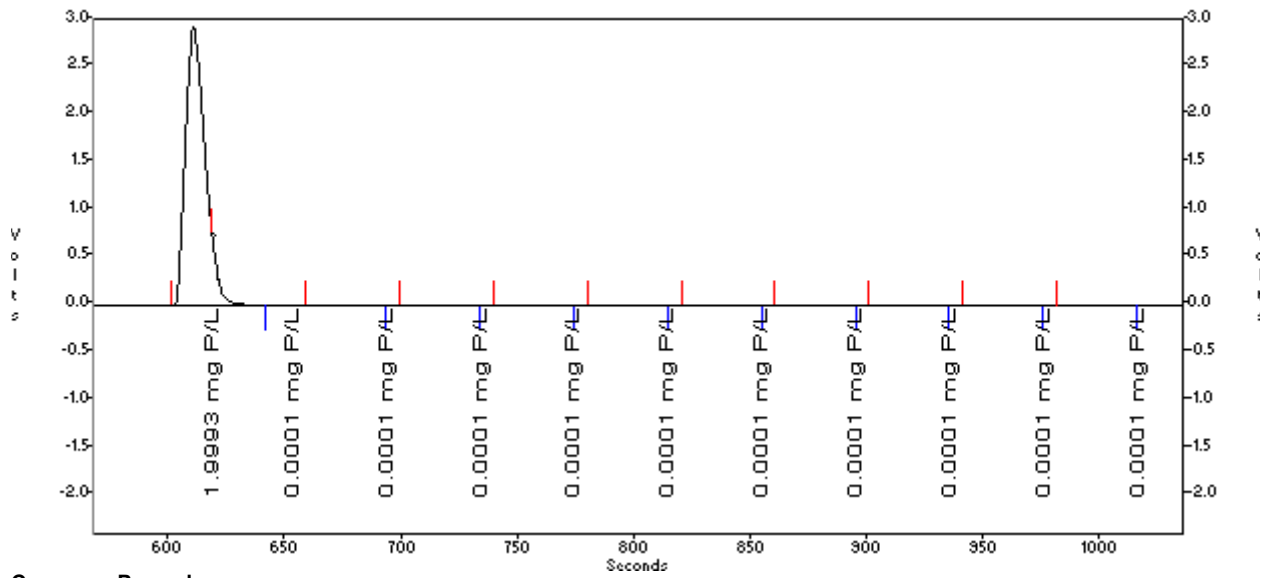
### Precision data for Orthophosphate using 0.5 mg P/L standard



**% RSD = 0.186**

Standard Deviation (s) = 0.001 mg P/L, Mean (x) = 0.503 mg P/L, Known value = 0.5 mg P/L

**Carryover Study: 2 mg P/L standard followed by 10 blanks**



**Carryover Passed**

- Other ranges are available

# Total Phosphorus by In-line UV/Persulfate Digestion and Flow Injection Analysis

QuikChem<sup>®</sup> Method 10-115-01-3-A  
SM 4500-P I.

## Total Phosphorous (In-Line Persulfate Digests)

0.10 to 10.0 mg P/L

### – Principle –

The method is based on the digestion of various phosphorous forms and conversion to phosphate by peroxodisulfate with an in-line UV digestion. Organic phosphorus is converted to orthophosphate by UV catalyzed persulfate digestion. Polyphosphates are converted to orthophosphate by sulfuric acid digestion. The digestion process occur prior to the sample valve. A portion of the digested sample is then injected and phosphate is determined by FIA.

Wastewater samples are acid preserved and filtered. When this is the case, in-line digestion results match the manual off-line digestion. If samples are not filtered, in-line results will be 1-15% low compared with off-line digestion. Surface water samples may not require filtration but this should be verified with a sample containing high levels of solids.

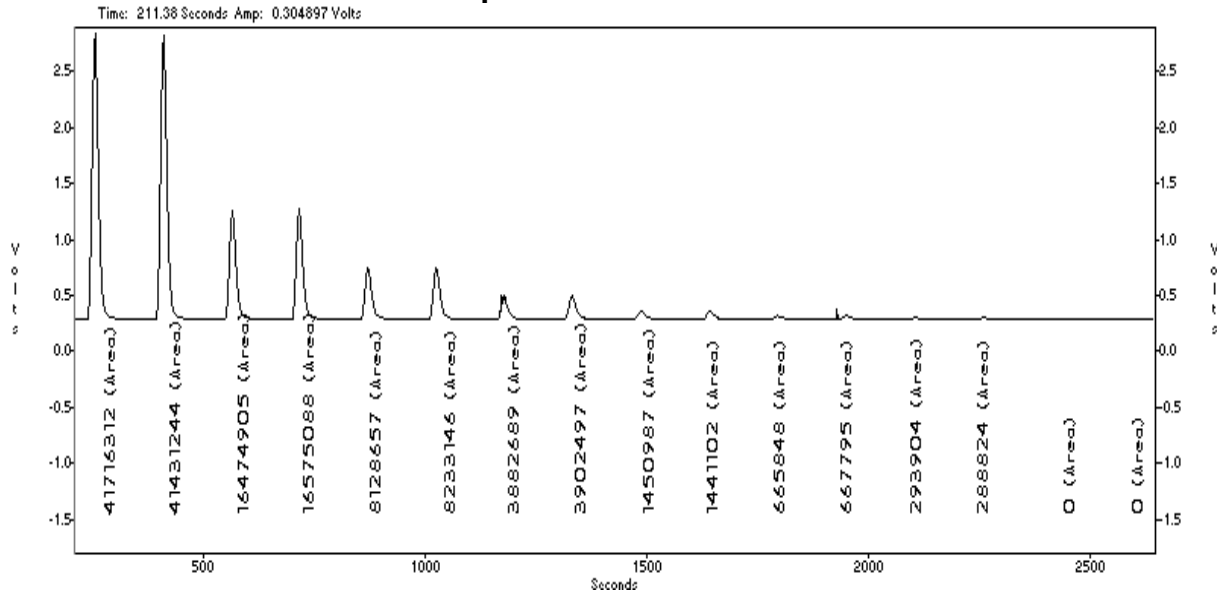
After digestion the orthophosphate ion ( $\text{PO}_4^{3-}$ ) reacts with ammonium molybdate and antimony potassium tartrate to form a phosphomolybdate complex. This complex is reduced with ascorbic acid to form a blue complex which absorbs light at 880 nm. The absorbance is proportional to the concentration of orthophosphate in the sample.

### – Special Apparatus –

Please contact Lachat Technical Support for Ordering Information

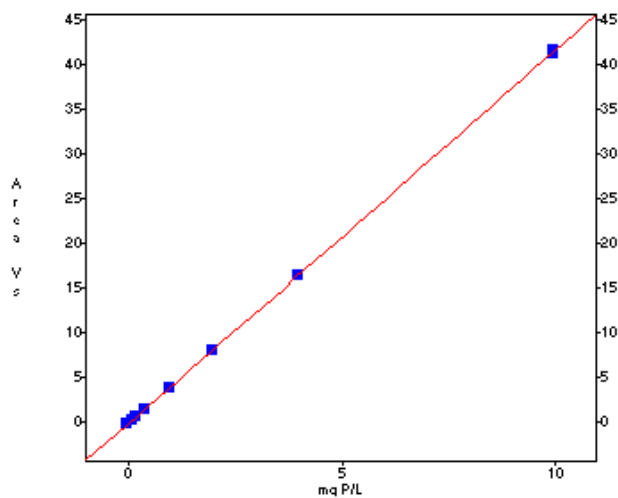
1. Lachat sample preparation module, A30X11 (x=1 for 110V, x=2 for 220V) with UV-254 nm lamp.
2. PVC PUMP TUBES MUST BE USED FOR THIS METHOD.

### Calibration Data for Total Phosphorus



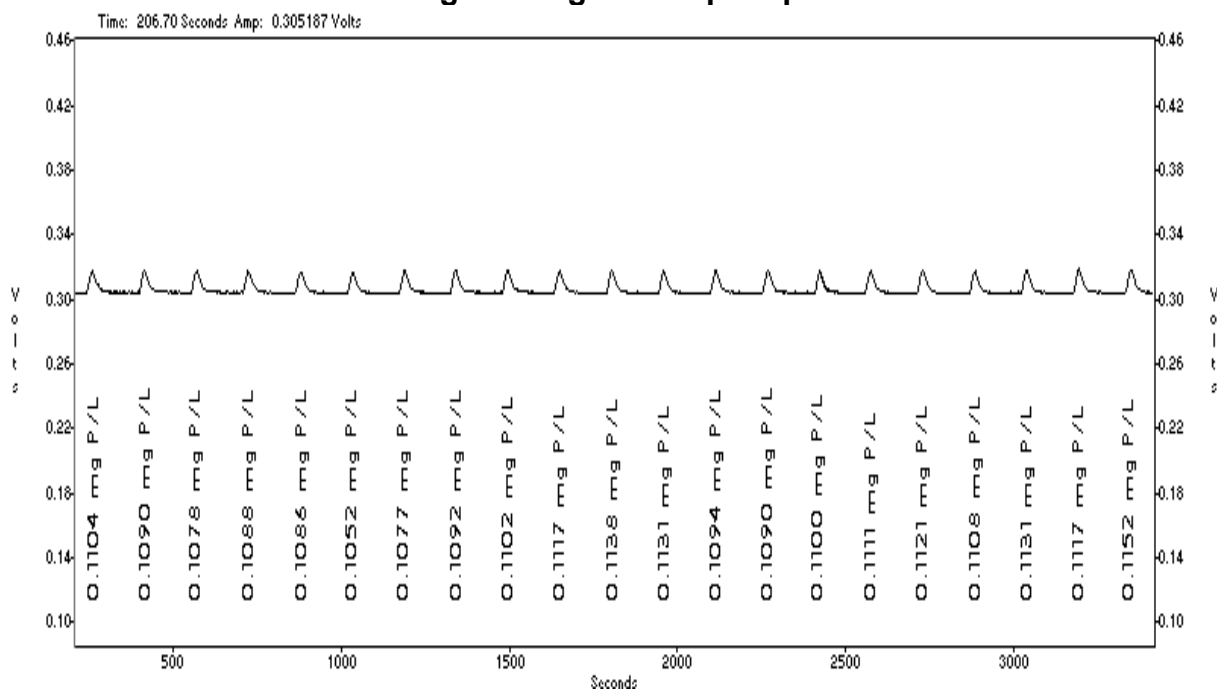
### Calibration Graph and Statistics

Level	Area (V-s)	mg P/L	Determined	Rep %RSD	% residual
1	41573776	10.0	10.00	0.5	0.0
2	16524996	4.00	4.01	0.4	-0.2
3	8180901	2.00	2.00	0.9	-0.2
4	3892593	1.00	0.97	0.4	2.8
5	1446045	0.40	0.38	0.5	4.4
6	666822	0.20	0.19	0.2	2.7
7	291364	0.10	0.10	1.2	-4.1
8	0	0.00	0.00	0.0	---



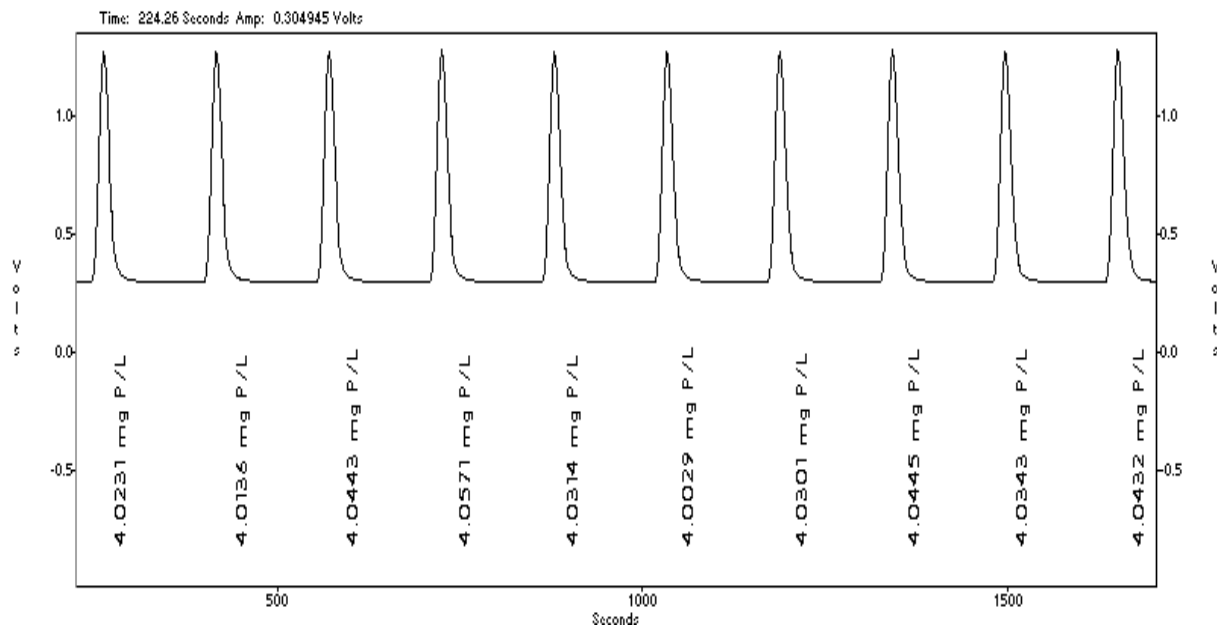
Scaling: None  
 Weighting: None  
 2nd Order Poly  
 Conc =  $-3.592e-017 \text{ Area}^2 + 2.412e-007 \text{ Area} + 3.386e-002$   
 $R^2 = 1.0000$

### Method Detection Limit using 0.10 mg/L orthophosphate



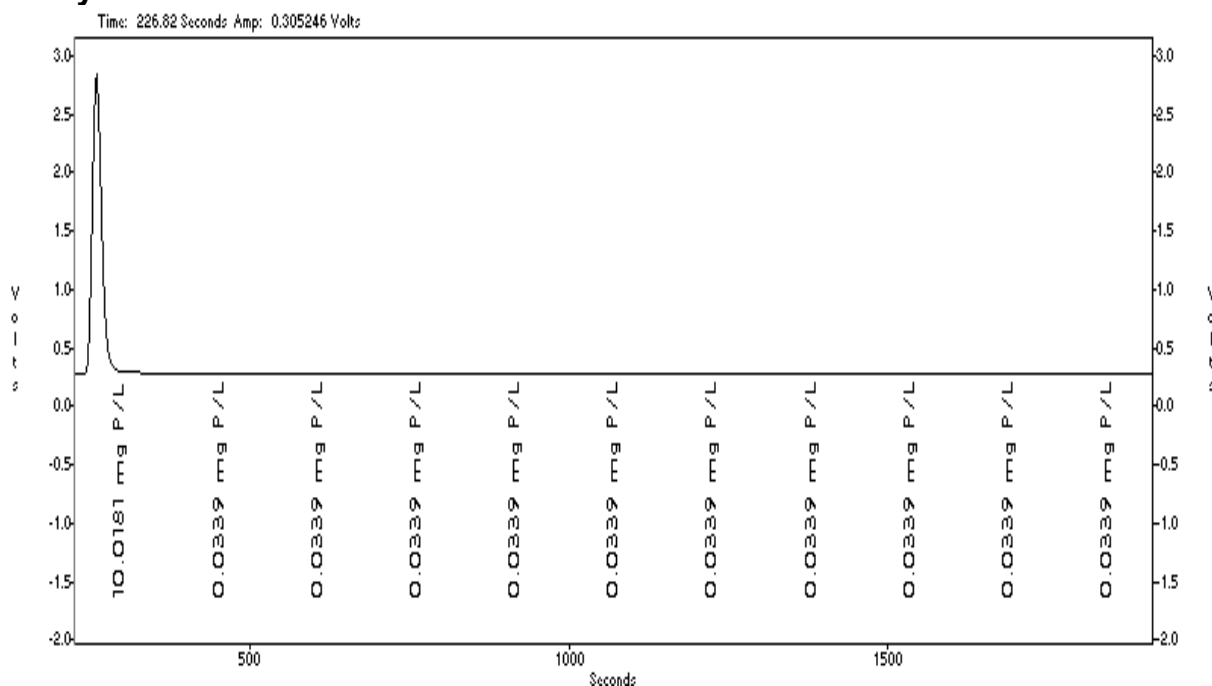
**MDL = 0.007 mg P/L**  
 Mean = 0.104 mg P/L, Std. Dev. = 0.0027  
 Data Filename: 072296m1.fdt  
 Acq Time: 22 July 1996, 14:02:38

### Precision data for phosphorus using 4 ppm trimethyl phosphate



**%RSD = 0.40**  
 Mean = 4.032 mg P/L, Std. Dev. = 0.0161, known value = 4.0 mg P/L  
 Data Filename: 071096p1.fdt  
 Acq Time: 10 July 1996, 12:24:33

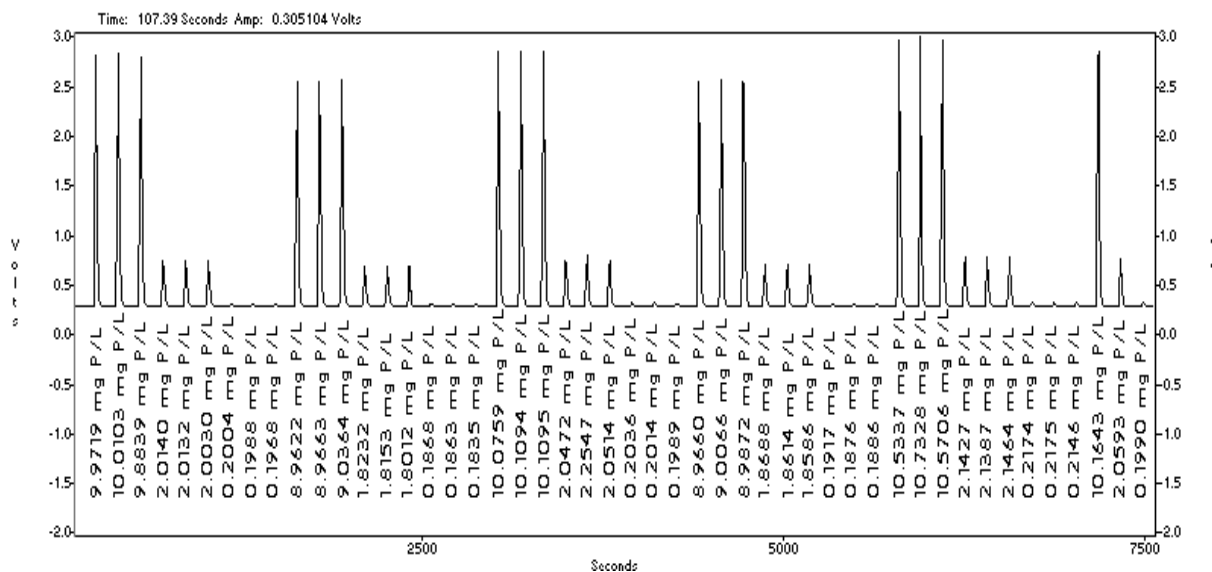
### Carryover



**Carryover passed**

Data Filename: 072296c1.fdt

Acq Time: 22 July 1996, 15:53:50

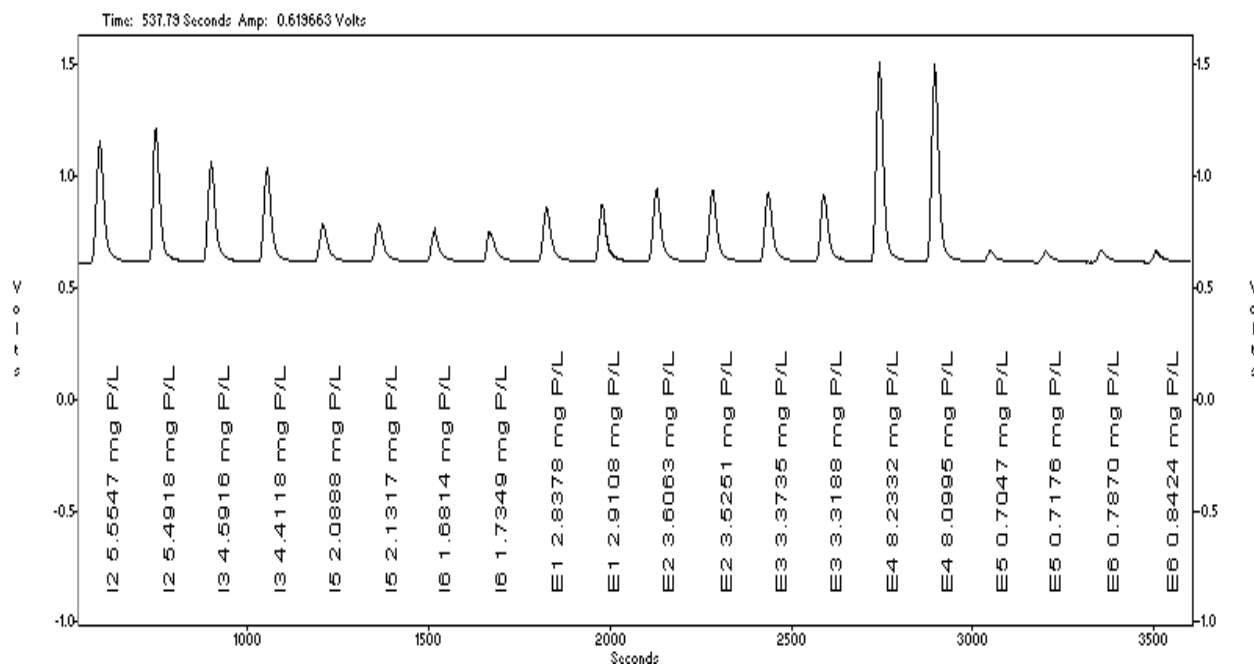


Compounds	Mean concentration (mg P/L)	Standard Deviation	% Recovery based from KH <sub>2</sub> PO <sub>4</sub> Recovery
<b>KH<sub>2</sub>PO<sub>4</sub></b>			
10 mg P/L	9.95	0.065	---
2 mg P/L	2.01	0.006	---
0.2 mg P/L	0.20	0.002	---
<b>Na Pyrophosphate</b>			
10 mg P/L	8.99	0.042	90.3
2 mg P/L	1.81	0.011	90.2
0.2 mg P/L	0.19	0.002	93.4
<b>Phenyl phosphate</b>			

10 mg P/L	10.10	0.019	101.5
2 mg P/L	2.12	0.119	105.0
0.2 mg P/L	0.20	0.002	101.3
<b>Trimethyl phosphate</b>			
10 mg P/L	8.99	0.020	90.3
2 mg P/L	1.86	0.005	92.7
0.2 mg P/L	0.18	0.002	95.3
<b>Sodium Tripolyphosphate</b>			
10 mg P/L	10.61	0.106	106.7
2 mg P/L	2.14	0.004	106.6
0.2 mg P/L	0.22	0.002	108.9

## Summary

All the the recoveries were calculated from the  $\text{KH}_2\text{PO}_4$  standards run as samples. The digestion recovers organic phosphorus very well with average recoveries of 98% for phenyl phosphate and 92% for trimethyl phosphate. The average poly phosphate recoveries were 88% for sodium pyrophosphate and 98% for sodium tripoly phosphate.



## Sample evaluation, wastewater samples determined in duplicate

Samples (ID)	Average Concentration (mg P/L)
Influent (I2)	5.52
Influent (I3)	4.50
Influent (I5)	2.11
Influent (I6)	1.70
Effluent (E1)	2.87
Effluent (E2)	3.57
Effluent (E3)	3.34
Effluent (E4)	8.16
Effluent (E5)	0.71
Effluent (E6)	0.82

# Silicate by Flow Injection Analysis

**QuikChem<sup>®</sup> Method 10-114-27-1-B**

SM 4500 SiO<sub>2</sub> F.

## **Molybdate-Reactive Silicate in Waters**

5.0 to 100 µg SiO<sub>2</sub>/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 –**

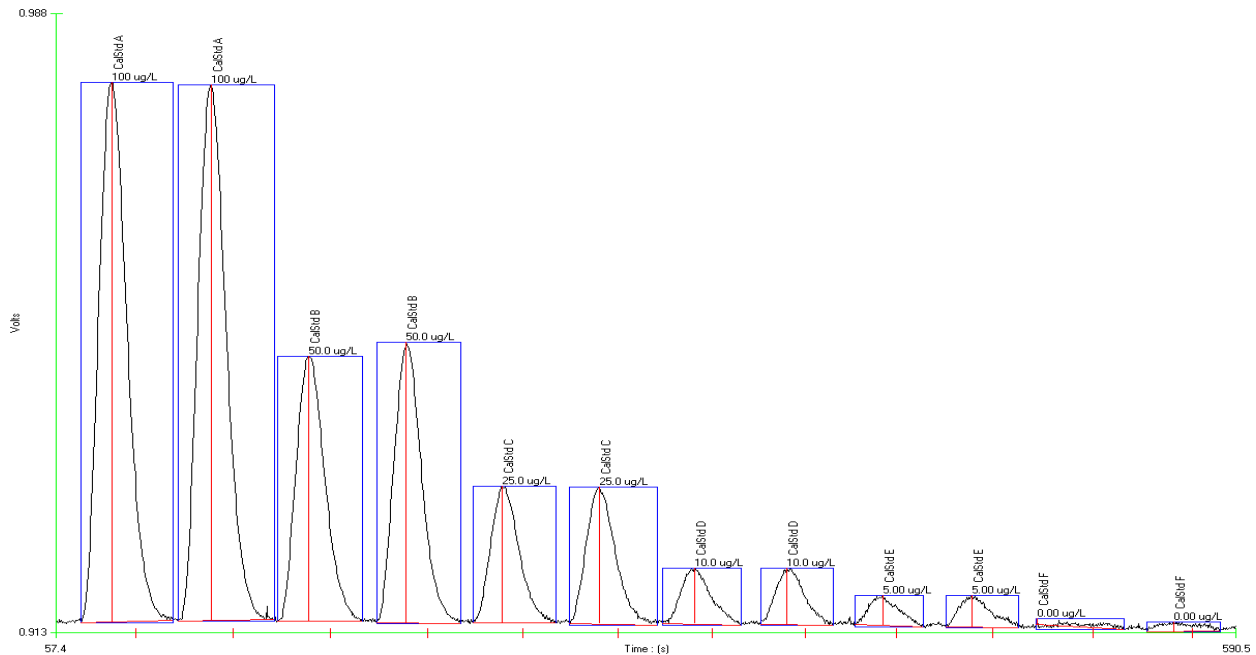
1. The interference due to phosphate is reduced by the addition of oxalic acid. A solution of 1000 µg P/L was determined as 20 µg SiO<sub>2</sub>/L. The extent of phosphate interference should be verified by determining a solution of phosphate at the highest concentration that is expected to be encountered. 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 Technical Support 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.

### Calibration Data for Silicate



File name: OM\_10-30-2007\_08\_14\_38PM.OMN  
Acq. Date: 30 October 2007

### Calibration Graph and Statistics

**Calibration Results - Channel 3**

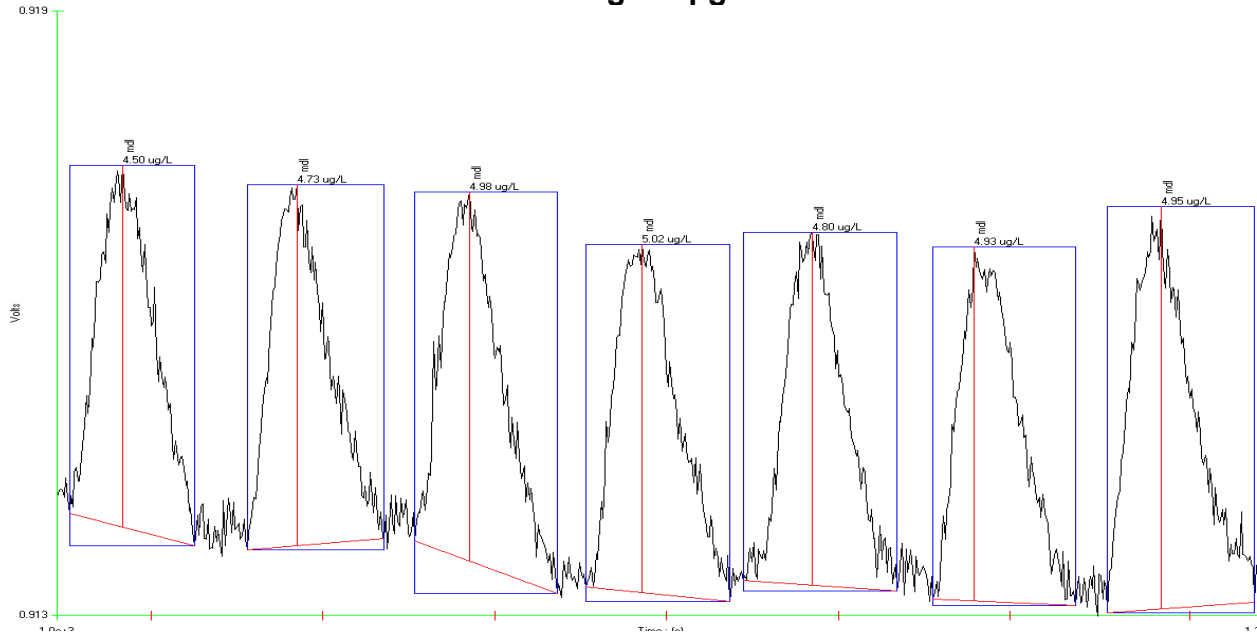
SiO2

	Known Conc. (ug/L)	Rep.	Peak Area (V.s)	Peak Height (V)	% RSD	% Resid.	Unused	Det. Conc (ug/L)	Date	Time
1	100	1	0.942	0.0647	0.2	-0.3	<input type="checkbox"/>	100	10/30/2007	8:15:29 AM
2	100	2	0.940	0.0643	0.2	-0.1	<input type="checkbox"/>	100	10/30/2007	8:16:14 AM
3	50.0	1	0.471	0.0318	0.5	5.0e-3	<input type="checkbox"/>	50.0	10/30/2007	8:16:59 AM
4	50.0	2	0.468	0.0337	0.5	0.7	<input type="checkbox"/>	49.7	10/30/2007	8:17:42 AM
5	25.0	1	0.235	0.0163	0.6	1.2	<input type="checkbox"/>	24.7	10/30/2007	8:18:26 AM
6	25.0	2	0.233	0.0164	0.6	2.0	<input type="checkbox"/>	24.5	10/30/2007	8:19:10 AM
7	10.0	1	0.0963	6.72e-3	2.4	1.2	<input type="checkbox"/>	9.88	10/30/2007	8:19:53 AM
8	10.0	2	0.0931	6.76e-3	2.4	4.5	<input type="checkbox"/>	9.53	10/30/2007	8:20:35 AM
9	5.00	1	0.0496	3.61e-3	3.3	2.3	<input type="checkbox"/>	4.88	10/30/2007	8:21:17 AM
10	5.00	2	0.0520	3.88e-3	3.3	-2.4	<input type="checkbox"/>	5.14	10/30/2007	8:21:59 AM
11	0.00	1	7.40e-3	7.38e-4			<input type="checkbox"/>	0.371	10/30/2007	8:22:42 AM
12	0.00	2	0.0127	1.17e-3			<input type="checkbox"/>	0.939	10/30/2007	8:23:28 AM

Area = 9.35e-3 \* Conc + 3.97e-3  
 Conc = 107 \* Area - 0.421  
 Correlation Coefficient (r) = 0.99994  
 Weighting : None

Settings

### Method Detection Limit for silicate using 5.0 µg SiO<sub>2</sub>/L standard



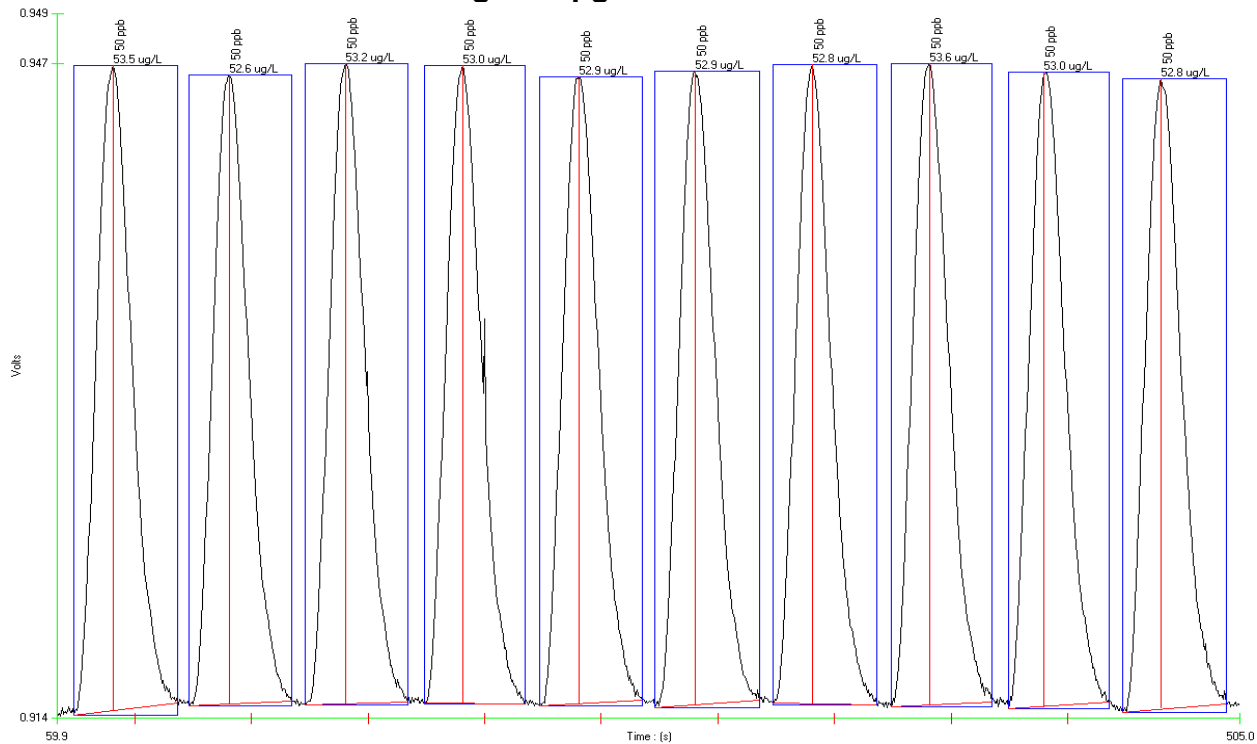
**MDL = 0.58 µg SiO<sub>2</sub>/L**

Standard Deviation (s) = 0.183 µg SiO<sub>2</sub>/L, Mean (x) = 4.84 µg SiO<sub>2</sub>/L, Known value = 5.0 µg SiO<sub>2</sub>/L

File name: OM\_10-29-2007\_02\_39\_12PM.OMN

Acq. Date: 29 October 2007

### Precision data for silicate using 50.0 µg SiO<sub>2</sub>/L standard



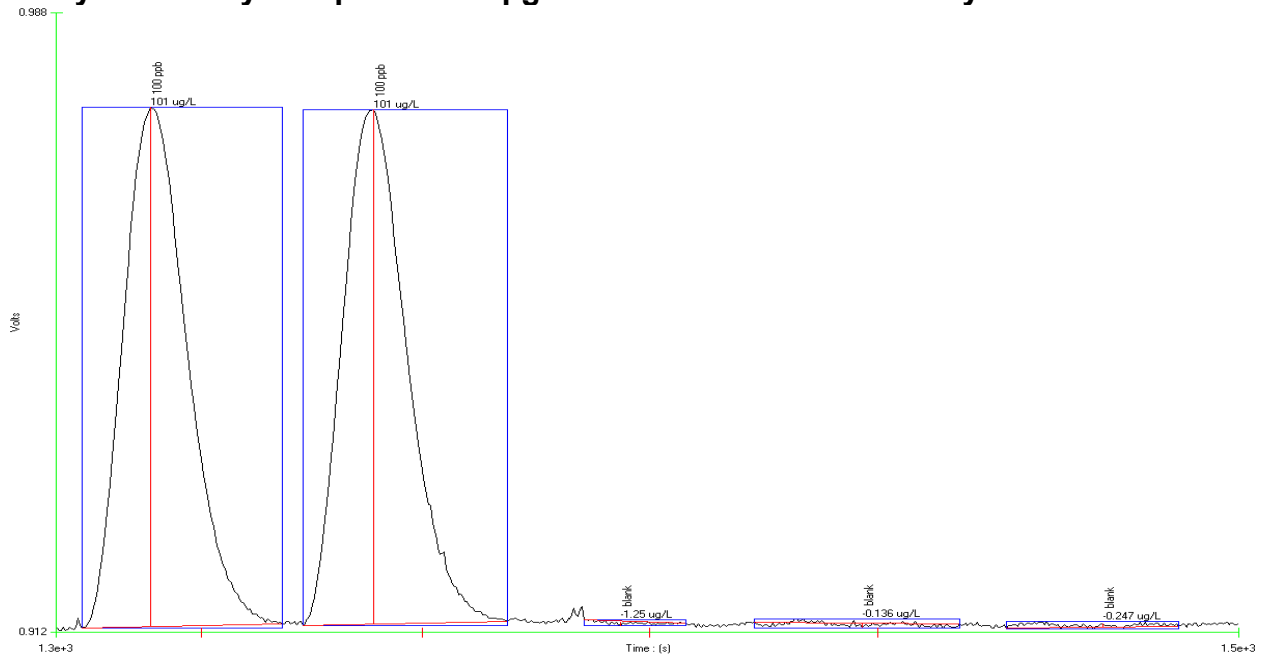
**% RSD = 0.60%**

Standard Deviation (s) = 0.316 µg SiO<sub>2</sub>/L, Mean (x) = 53.03 µg SiO<sub>2</sub>/L, Known value = 50.0 µg SiO<sub>2</sub>/L

File name: OM\_10-30-2007\_10\_50\_16AM.OMN

Acq. Date: 30 October 2007

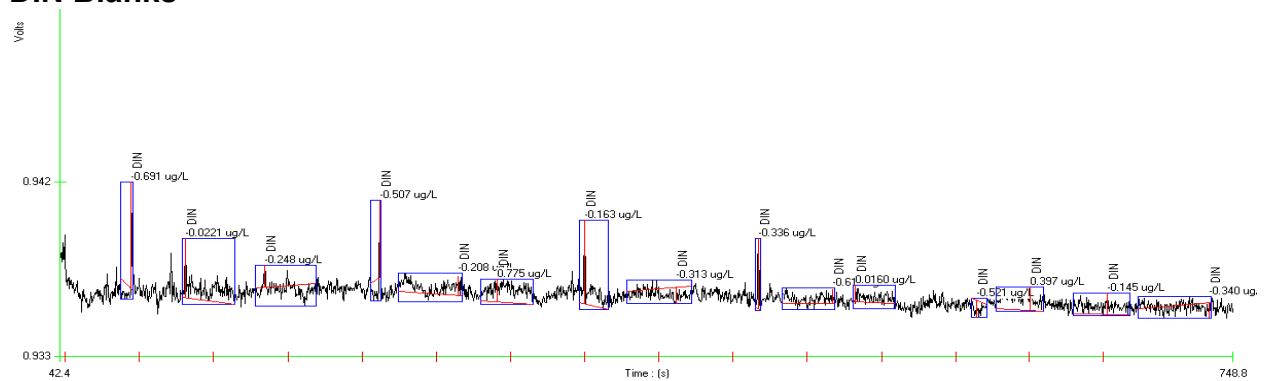
**Carryover Study: 2 replicates of  $\mu\text{g SiO}_2/\text{L}$  standard followed by 3 blanks**



**Carryover Passed**

File name: OM\_10-30-2007\_08\_14\_38AM.OMN  
Acq. Date: 30 October 2007

**DIN Blanks**



Average:  $-0.195 \mu\text{g SiO}_2/\text{L}$ , SD =  $0.383 \mu\text{g SiO}_2/\text{L}$ . Calculated DIN Limits: Detection Limit =  $1.15 \mu\text{g SiO}_2/\text{L}$ , Decision Limit =  $2.30 \mu\text{g SiO}_2/\text{L}$ , Determination Limit =  $3.45 \mu\text{g SiO}_2/\text{L}$ ;  
File name: OM\_10-30-2007\_01\_51-54PM.OMN  
Acq. Date: 30 October 2007

# Sulfide by MicroDIST Methylene Blue Flow Injection Analysis Method

QuikChem<sup>®</sup> Method 10-116-29-1-A  
(SM 4500-S<sup>2-</sup> I.)

## Sulfide by MICRODIST<sup>®</sup>

0.02 to 2.0 mg S/L

### – Principle –

Water and wastewater samples are distilled into a sodium hydroxide trapping solution and the distillate is then sampled. Hydrogen sulfide (H<sub>2</sub>S) reacts in acid media and in the presence of ferric chloride with two molecules of N,N-dimethyl-p-phenylenediamine to form methylene blue. The resulting color is read at 660 nm.

### – Interferences –

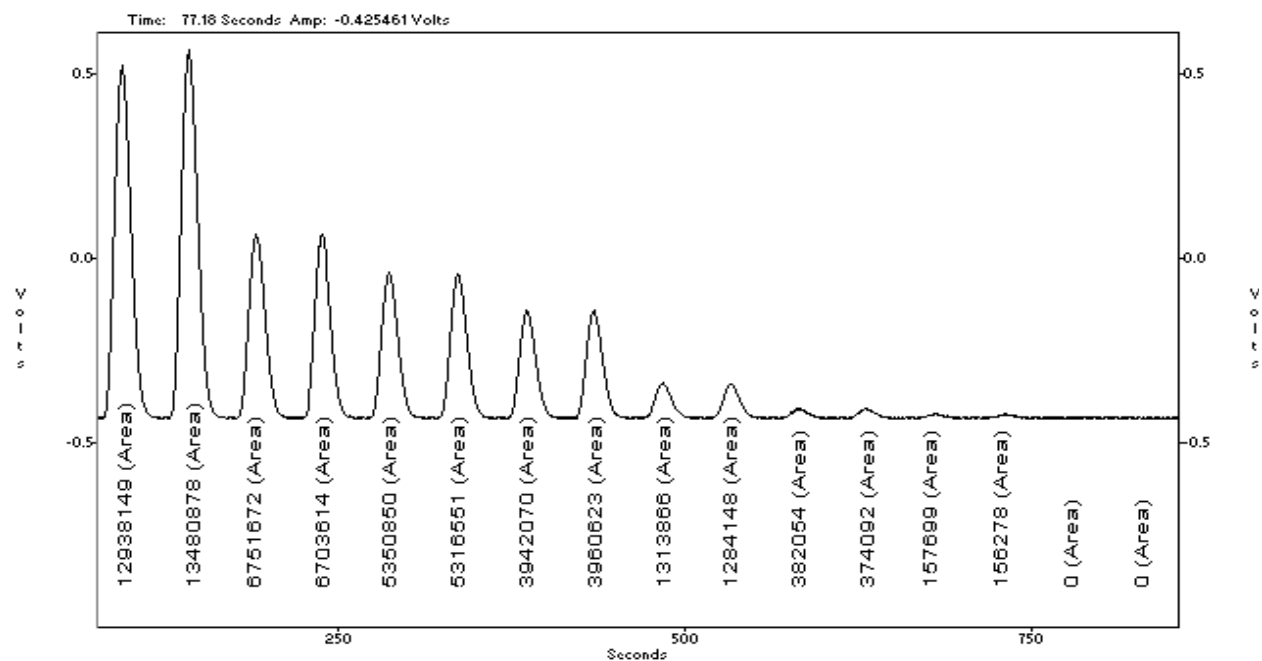
1. Strong reducing agents inhibit color formation at levels of several hundred ppm.
2. Iodide interferes at levels greater than 2 mg/L.

### – Special Apparatus –

Please contact Lachat Technical Support for Ordering Information

1. Heating Unit Lachat Part No. A85X00 (X=1 for 110V, X=2 for 220V)
2. MICRO DIST<sup>®</sup> (Lachat Part No. A17102) and MICRO DIST<sup>®</sup> tubes (Lachat Part No. A17009)

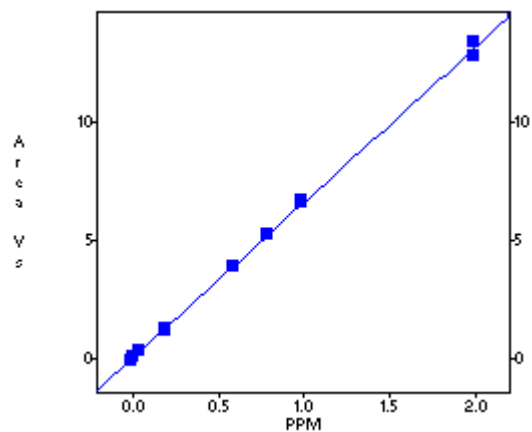
### Calibration Data for Sulfide



File Name: 960208c1.fdt  
Acq. Time: February 8, 1996, 14:10:00

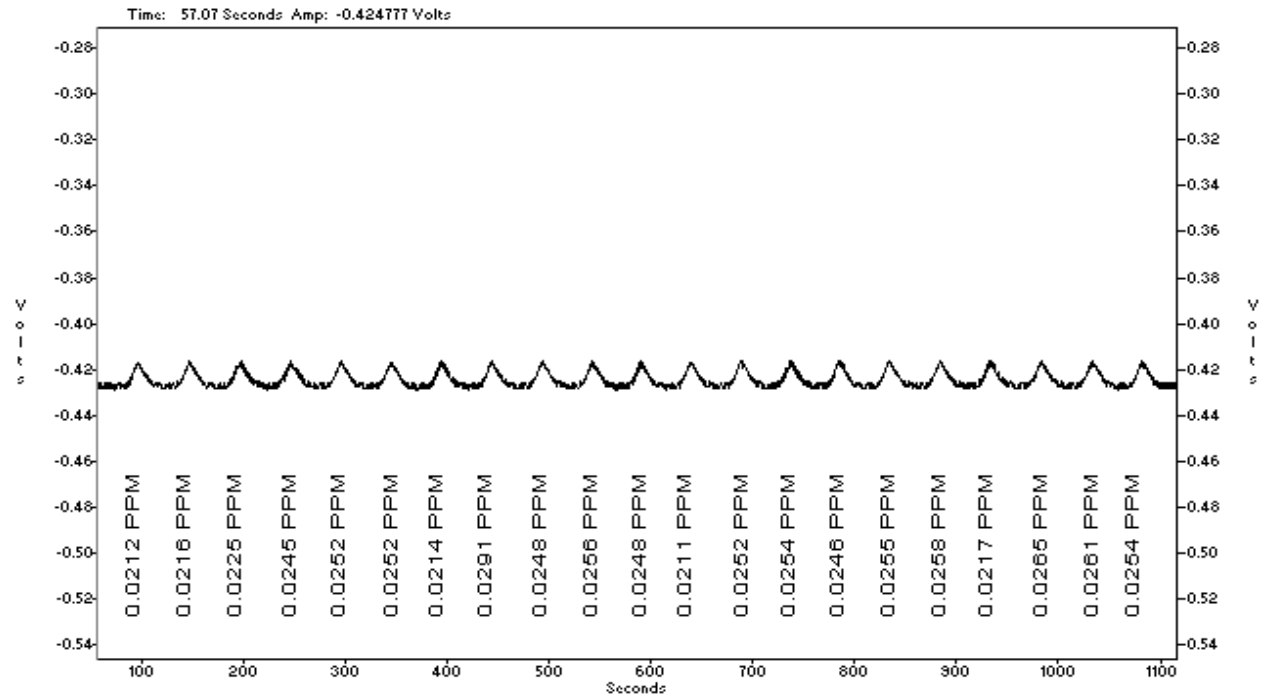
### Calibration Graph and Statistics

Level	Area (V-s)	mg S/L	Determined	Replicate %RSD	% residual
1	13209514	2.00	1.99	2.9	0.3
2	6727643	1.00	1.01	0.5	-1.4
3	5333700	0.80	0.80	0.5	-0.4
4	3951346	0.60	0.59	0.3	1.0
5	1299007	0.20	0.19	1.6	3.4
6	378073	0.05	0.05	1.5	-8.0
7	156988	0.02	0.02	0.6	-3.0
8	0	0.0	0.0	0.0	0.0



Scaling: None  
Weighting: None  
1st Order Poly  
Conc = 1.512e-007 Area - 3.132e-003  
R<sup>2</sup> = 0.9999

### Method Detection Limit for sulfide using a 0.02 mg/L standard

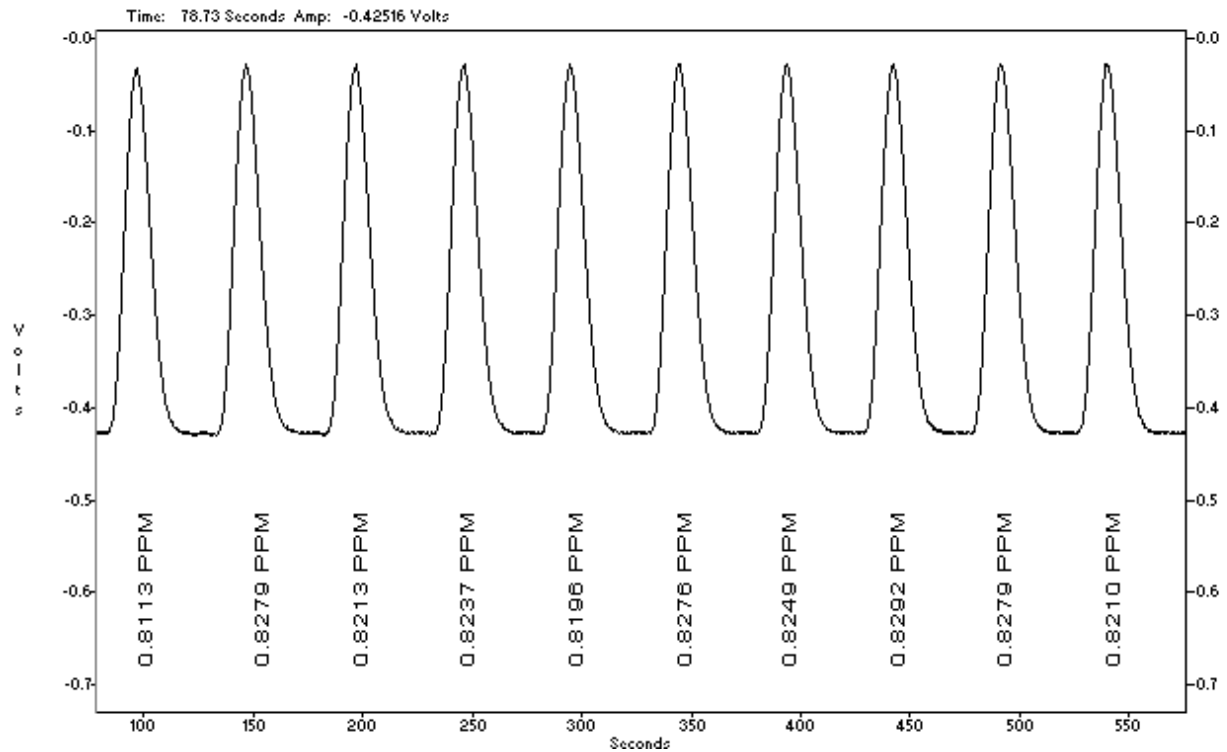


**MDL = 0.005 mg/L**

Standard Deviation (s) = 0.0021 mg/L, Mean (x) = 0.0244 mg/L, Known Value = 0.02 mg/L

Acq. Date: February 8, 1996

### Precision Data for sulfide using a 0.8 mg/L standard

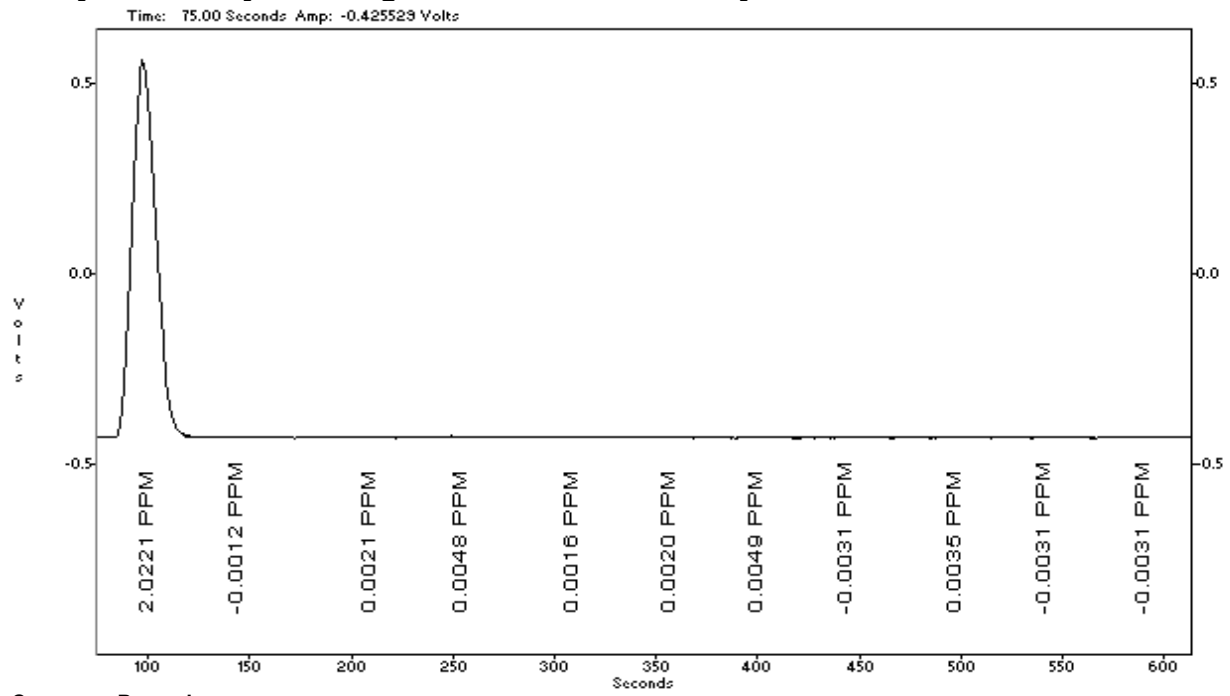


**% RSD = 0.66**

Standard Deviation (s) = 0.0054 mg/L, Mean (x) = 0.8234 mg/L, Known Value = 0.8 mg/L

Acq. Date: February 8, 1996

### Carryover Study: 2.00 mg/L standard followed by 10 blanks



**Carryover Passed**  
Acq. February 8, 1996

# Sulfate

## Methylthymol Blue Flow Injection Analysis

**QuikChem<sup>®</sup> Method 10-116-10-2-A**  
SM 4500-SO<sub>4</sub><sup>2-</sup> G.

### Sulfate (Methylthymol Blue) in Waters

5 to 100 mg SO<sub>4</sub><sup>2-</sup>/L

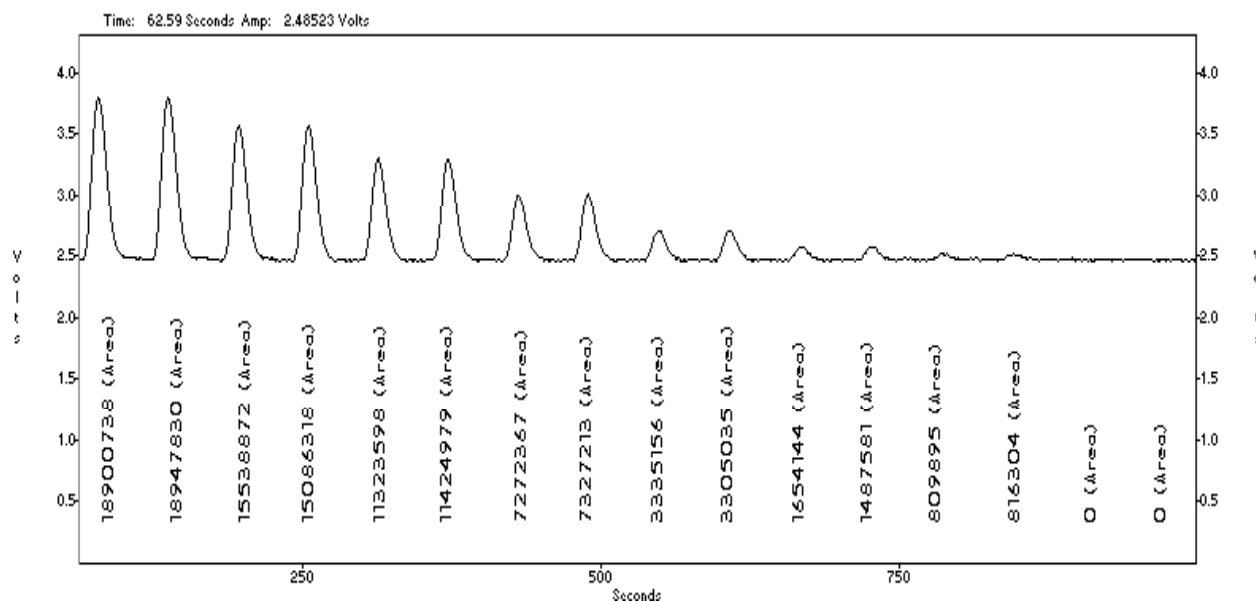
#### – Principle –

At pH 13.0 barium forms a blue complex with methylthymol blue (MTB). This gives a dark blue baseline. The sample is injected into a low, but known, concentration of sulfate. The sulfate from the sample then reacts with the ethanolic barium-MTB solution and displaces the MTB from the barium to give barium sulfate and uncomplexed MTB. Uncomplexed MTB has a gray color. The pH is raised and the gray color of uncomplexed MTB is measured at 460 nm.

#### – Interferences –

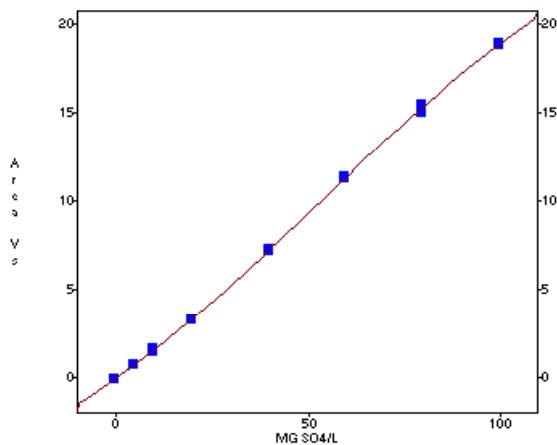
1. Multivalent cations: A cation exchange column removes multivalent cations such as Ca<sup>2+</sup> and Mg<sup>2+</sup>. A midrange sulfate standard containing a typical level of hardness as CaCO<sub>3</sub> can be run periodically to check the performance of the column. Any decrease in peak height from that of a sulfate standard without added CaCO<sub>3</sub> indicates the need to regenerate or replace the resin.
2. Strongly acidic samples: Samples with pH less than 2 must be neutralized. High acid concentrations can displace multivalent cations from the column.
3. Turbidity: Turbid samples may be filtered or centrifuged.
4. Orthophosphate: Orthophosphate forms a precipitate with barium at high pH. If samples are known to be high in orthophosphate, a recovery study, using added amounts of sulfate, should be done, or a sample blank containing only orthophosphate should be run.

### Calibration Data for Sulfate



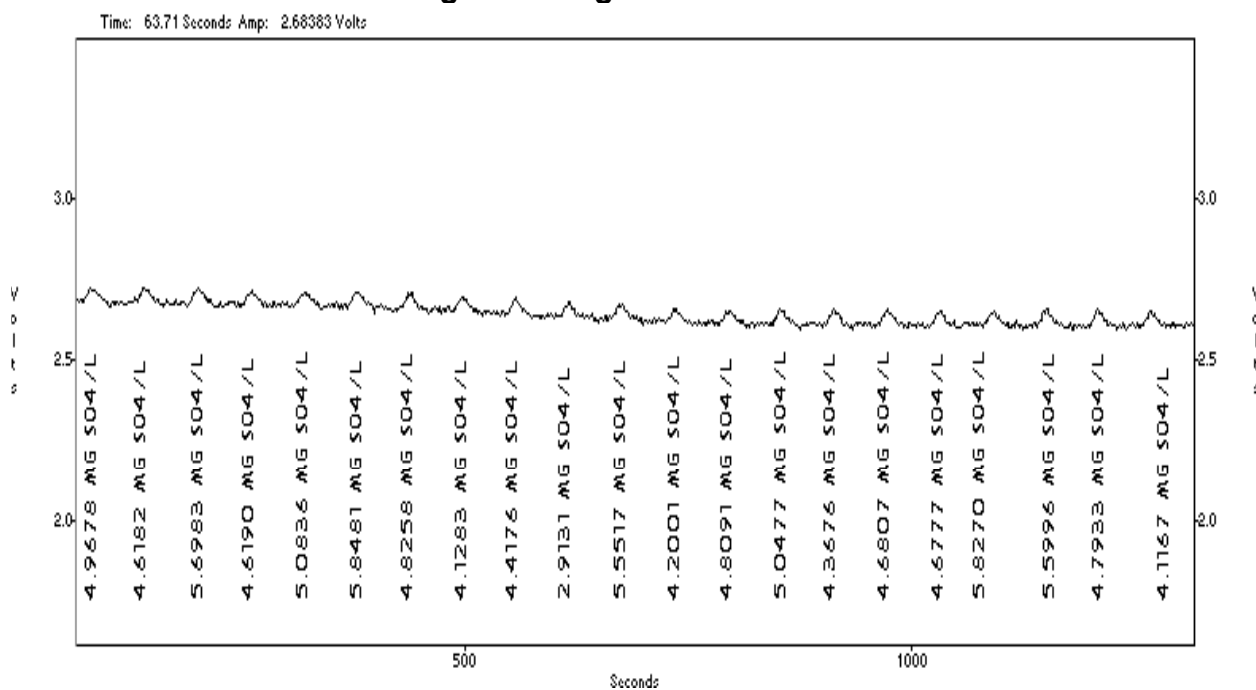
### Calibration Graph and Statistics

Level	Area	mg SO <sub>4</sub> /L	Determined	Replic %RSD	%residual
1	18924284	100	100.1	0.2	-0.1
2	15312595	80	79.8	2.1	0.3
3	11374288	60	60.1	0.6	-0.1
4	7299790	40	40.3	0.5	-0.8
5	3320096	20	19.7	0.6	1.4
6	1570863	10	9.8	7.5	2.5
7	813100	5.0	5.2	0.6	-3.8
8	0	0.0	0.1	0.0	---



Scaling: None  
 Weighting: None  
 3rd Order Poly  
 Conc = 5.173e-021 Area<sup>3</sup> - 1.549e-013 Area<sup>2</sup> + 6.362e-006 Area + 1.144e-001  
 R<sup>2</sup> = 1.0000

### Method Detection Limit using a 5.00 mg SO<sub>4</sub>/L standard



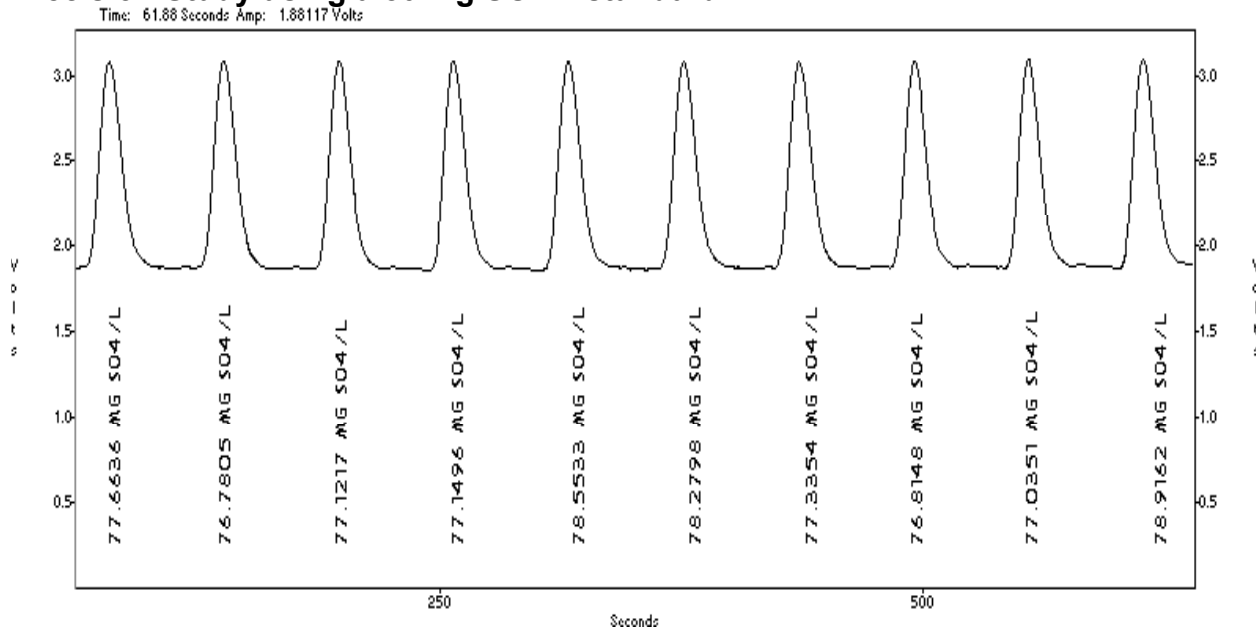
**MDL = 1.8 mg/L**

Mean (X) = 4.80 mg/L, standard deviation = 0.693, known value = 5.00 mg/L

File Name: 061196M5.fdt

Acq. time: 11 June 1996, 14:48:59

### Precision Study using a 80 mg SO<sub>4</sub>/L standard



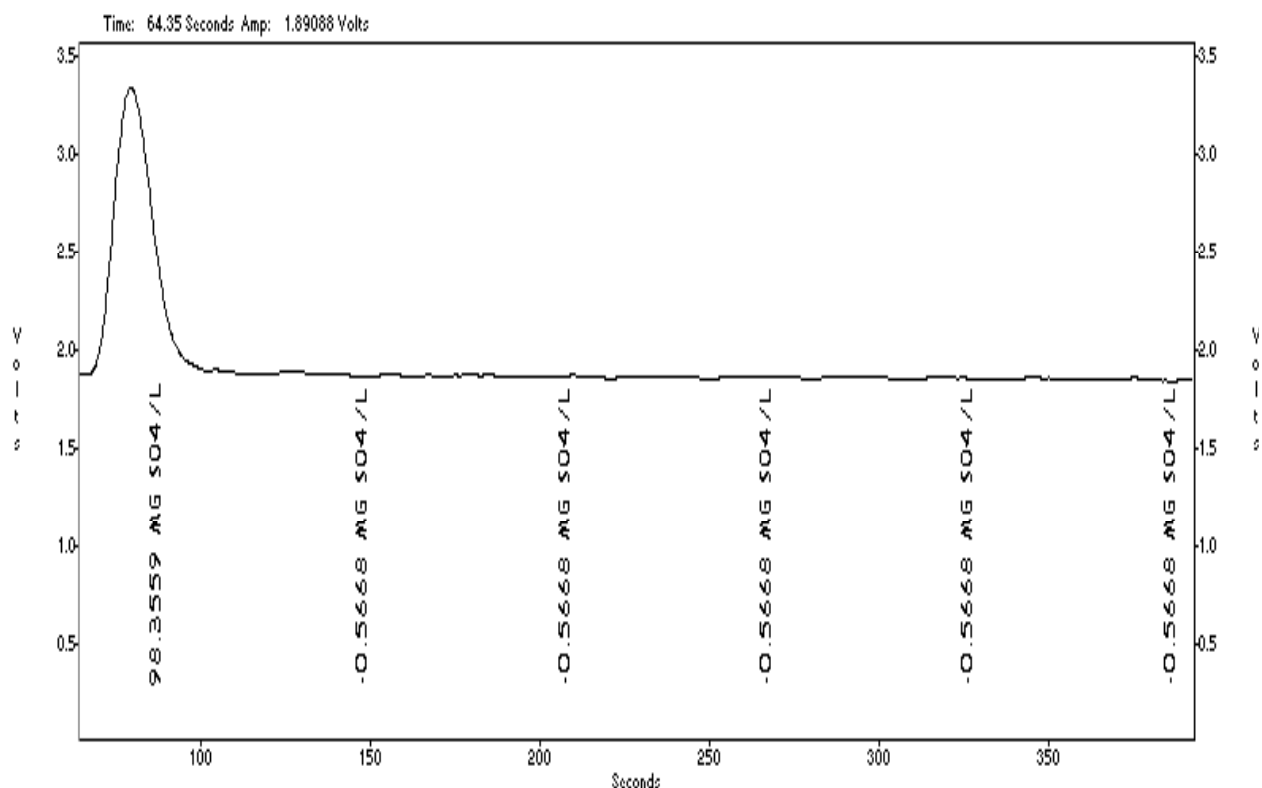
**%RSD = 0.98**

Mean (X) = 77.6 mg/L, standard deviation = 0.761, known value = 80 mg SO<sub>4</sub>/L

Filename: 061296.fdt

Acq. Time: 12 June 1996, 17:49:13

### Carryover Study: 100 mg/L standard followed by 5 blanks



**Carryover passed**

Acq Time: 12 June 1996, 18:01:26

### Cation Exchange Column Performance:

150 mg Ca/L and 20 mg SO<sub>4</sub><sup>2-</sup>/L standard

Cup #	Sample ID	mg SO <sub>4</sub> /L	Cup #	Sample ID	mg SO <sub>4</sub> /L	Cup #	Sample ID	mg SO <sub>4</sub> /L
1	20 mg SO <sub>4</sub> /L	19.6125	53	20 mg SO <sub>4</sub> /L	20.0607	105	20mg SO <sub>4</sub> /L	20.6004
2	20 mg SO <sub>4</sub> /L	19.5882	54	20 mg SO <sub>4</sub> /L	19.6217	106	20mg SO <sub>4</sub> /L	21.9944
3	20 mg SO <sub>4</sub> /L	20.2303	55	20 mg SO <sub>4</sub> /L	20.3163	107	20mg SO <sub>4</sub> /L	21.6175
4	20 mg SO <sub>4</sub> /L	19.7691	56	20 mg SO <sub>4</sub> /L	19.7155	108	20mg SO <sub>4</sub> /L	22.5170
5	20 mg SO <sub>4</sub> /L	19.5719	57	20 mg SO <sub>4</sub> /L	19.7221	109	20mg SO <sub>4</sub> /L	21.7381
6	20 mg SO <sub>4</sub> /L	19.1665	58	20 mg SO <sub>4</sub> /L	20.2055	110	20mg SO <sub>4</sub> /L	21.3391
7	20 mg SO <sub>4</sub> /L	19.6855	59	20 mg SO <sub>4</sub> /L	19.3597	111	20mg SO <sub>4</sub> /L	21.6503
8	20 mg SO <sub>4</sub> /L	20.4929	60	20 mg SO <sub>4</sub> /L	20.5937	112	20mg SO <sub>4</sub> /L	21.4289
9	20 mg SO <sub>4</sub> /L	19.7813	61	20 mg SO <sub>4</sub> /L	20.1458	113	20mg SO <sub>4</sub> /L	21.7629
10	20 mg SO <sub>4</sub> /L	19.7734	62	20 mg SO <sub>4</sub> /L	20.1491	114	20mg SO <sub>4</sub> /L	22.0275
11	20 mg SO <sub>4</sub> /L	19.8616	63	20 mg SO <sub>4</sub> /L	20.5181	115	20mg SO <sub>4</sub> /L	21.0549
12	20 mg SO <sub>4</sub> /L	20.0599	64	20 mg SO <sub>4</sub> /L	20.4262	116	20mg SO <sub>4</sub> /L	22.0417
13	20 mg SO <sub>4</sub> /L	20.1057	65	20 mg SO <sub>4</sub> /L	19.9017	117	20mg	22.3390

							SO <sub>4</sub> /L	
14	20 mg SO <sub>4</sub> /L	20.5716	66	20 mg SO <sub>4</sub> /L	19.8725	118	20mg SO <sub>4</sub> /L	22.2342
15	20 mg SO <sub>4</sub> /L	19.6498	67	20 mg SO <sub>4</sub> /L	19.4742	119	20mg SO <sub>4</sub> /L	20.8131
16	20 mg SO <sub>4</sub> /L	19.6782	68	20 mg SO <sub>4</sub> /L	20.1980	120	20mg SO <sub>4</sub> /L	20.3641
17	20 mg SO <sub>4</sub> /L	19.6913	69	20 mg SO <sub>4</sub> /L	19.9658	121	20mg SO <sub>4</sub> /L	20.6705
18	20 mg SO <sub>4</sub> /L	19.7174	70	20 mg SO <sub>4</sub> /L	20.0692	122	20mg SO <sub>4</sub> /L	19.3238
19	20 mg SO <sub>4</sub> /L	20.1944	71	20 mg SO <sub>4</sub> /L	20.1548	123	20mg SO <sub>4</sub> /L	19.4882
20	20 mg SO <sub>4</sub> /L	21.0604	72	20 mg SO <sub>4</sub> /L	20.9164	124	20mg SO <sub>4</sub> /L	18.4319
21	20 mg SO <sub>4</sub> /L	21.0787	73	20 mg SO <sub>4</sub> /L	20.7728	125	20mg SO <sub>4</sub> /L	18.2703
22	20 mg SO <sub>4</sub> /L	19.9720	74	20 mg SO <sub>4</sub> /L	20.3322			
23	20 mg SO <sub>4</sub> /L	20.0594	75	20 mg SO <sub>4</sub> /L	20.0129			
24	20 mg SO <sub>4</sub> /L	19.9015	76	20 mg SO <sub>4</sub> /L	20.4394			
25	20 mg SO <sub>4</sub> /L	19.9902	77	20 mg SO <sub>4</sub> /L	20.2556			
26	20 mg SO <sub>4</sub> /L	20.3749	78	20 mg SO <sub>4</sub> /L	20.1694			
27	20 mg SO <sub>4</sub> /L	20.6618	79	20 mg SO <sub>4</sub> /L	20.1115			
28	20 mg SO <sub>4</sub> /L	19.9542	80	20 mg SO <sub>4</sub> /L	19.3258			
29	20 mg SO <sub>4</sub> /L	19.8591	81	20 mg SO <sub>4</sub> /L	20.1737			
30	20 mg SO <sub>4</sub> /L	20.2615	82	20 mg SO <sub>4</sub> /L	19.7565			
31	20 mg SO <sub>4</sub> /L	19.8476	83	20 mg SO <sub>4</sub> /L	20.9117			
32	20 mg SO <sub>4</sub> /L	20.4042	84	20 mg SO <sub>4</sub> /L	20.1948			
33	20 mg SO <sub>4</sub> /L	19.7422	85	20 mg SO <sub>4</sub> /L	20.0011			
34	20 mg SO <sub>4</sub> /L	19.2882	86	20 mg SO <sub>4</sub> /L	20.1331			
35	20 mg SO <sub>4</sub> /L	19.7433	87	20 mg SO <sub>4</sub> /L	19.6930			
36	20 mg SO <sub>4</sub> /L	19.4740	88	20 mg SO <sub>4</sub> /L	18.9998			
37	20 mg SO <sub>4</sub> /L	19.9046	89	20 mg SO <sub>4</sub> /L	19.0762			
38	20 mg SO <sub>4</sub> /L	20.1726	90	20 mg SO <sub>4</sub> /L	19.0929			
39	20 mg SO <sub>4</sub> /L	19.8188	91	20 mg SO <sub>4</sub> /L	19.6422			
40	20 mg SO <sub>4</sub> /L	19.6012	92	20 mg SO <sub>4</sub> /L	19.9463			
41	20 mg SO <sub>4</sub> /L	20.2396	93	20 mg SO <sub>4</sub> /L	19.7878			
42	20 mg SO <sub>4</sub> /L	19.7031	94	20 mg SO <sub>4</sub> /L	19.3705			
43	20 mg SO <sub>4</sub> /L	19.7780	95	20 mg SO <sub>4</sub> /L	19.8718			
44	20 mg SO <sub>4</sub> /L	19.8120	96	20 mg SO <sub>4</sub> /L	19.5606			
45	20 mg SO <sub>4</sub> /L	20.0930	97	20 mg SO <sub>4</sub> /L	19.5971			
46	20 mg SO <sub>4</sub> /L	19.6724	98	20 mg SO <sub>4</sub> /L	19.0914			
47	20 mg SO <sub>4</sub> /L	19.7872	99	20 mg SO <sub>4</sub> /L	19.9078			
48	20 mg SO <sub>4</sub> /L	19.6778	100	20 mg SO <sub>4</sub> /L	19.5671			
49	20 mg SO <sub>4</sub> /L	19.3390	101	20 mg SO <sub>4</sub> /L	18.9494			
50	20 mg SO <sub>4</sub> /L	19.3043	102	20 mg SO <sub>4</sub> /L	19.5392			
51	20 mg SO <sub>4</sub> /L	20.7353	103	20 mg SO <sub>4</sub> /L	18.8889			
52	20 mg SO <sub>4</sub> /L	19.8902	104	20 mg SO <sub>4</sub> /L	22.7349			