

Data Pack

Ultra Low Flow Methods

September 2009

Table of Contents

Introduction.....	3
Ammonia, Phenolate Method	6
Chloride, Mercuric Thiocyanate Method ..	14
Nitrate-Nitrite Cadmium Reduction Method ..	27
Nitrite	35
Orthophosphorus	41

Introduction

In developing Lachat's breakthrough Ultra High Throughput methods, Lachat Applications chemists modified traditional Flow Injection Analysis methods by increasing reagent and carrier stream flow rates. These modifications increased the methods' throughput while decreasing the reagent consumption per sample. As an end result, the Ultra High Throughput methods provided throughput of over 100 samples (per channel) per hour.

However, there are many labs that don't need this type of throughput but still desire the reduced reagent consumption with their FIA methods. For these labs, Lachat Application Chemists have developed the new **Ultra Low Flow methods** suite.

The Ultra Low Flow methods provide reagent conservation commonly seen with discrete analyzers, while maintaining the superior throughput, performance and support advantages of Lachat's QuikChem Flow Injection Analysis (FIA) analyzer. And unlike discrete analyzers, the Lachat ULF methods maintain low reagent consumption regardless of the number of reagents needed for a chemistry.

By slowing the speed of the reagent pump and optimizing the timing of the sample and reagents, significant reductions in the volume of reagents consumed per sample have been achieved. These reagent consumption savings have been realized without sacrificing the throughput or performance customers expect from FIA methodologies.

The ULF methods are compatible with the QC8000, QC8500 and QC8500 Series 2 FIA analyzers. No special equipment is needed, but the ULF methods all use a pump speed of 10 (versus 35 for "standard" Lachat methods), and so must be run together, or by themselves if other Lachat methodologies are also in use.

Contained in this Datapack are representative data from the initial suite of ULF methods developed. Additional ULF methods are being developed by the Lachat Applications team, so it is always best to consult Lachat Sales on the most current method portfolio.

The tables below provide a quick summary of the reagent usage between the new Ultra Low Flow methods and the traditional FIA methods.

Ammonia		
	Traditional method 10-107-06-1-J	ULF Methods 80-107-06-1-A, -B
	Cycle Period = 65 sec	Cycle Period = 60 sec
	Flow (mL per cycle period)	
Carrier	1.38	1.21
Buffer	1.02	0.27
Phenolate	1.02	0.27
Hypochlorite	0.82	0.22
Nitroprusside	0.82	0.22

Chloride		
	Traditional Method 10-117-07-1-A	ULF Method 80-117-07-1-A
	Cycle Period = 60 sec	Cycle Period = 60 sec
	Flow (mL per cycle period)	
Carrier (DI)	1.45	0.98
Color	2.47	0.50

Cyanide		
	Traditional Methods 10-204-00-1-A, -X	ULF Methods 80-204-00-1-A, -X
	Cycle Period = 45 sec	Cycle Period = 65 sec
	Flow (mL per cycle period)	
Carrier (0.25N NaOH)	2.21	0.77
Buffer	0.96	0.45
Chloramine T	0.96	0.15
Pyridine	1.85	0.40

Nitrate - Nitrite		
	Traditional Method 10-107-04-1-J	ULF Method 80-107-04-1-A
	Cycle Period = 60 sec	Cycle Period = 60 sec
	Flow (mL per cycle period)	
Sulfanilamide	0.76	0.42
NH4Cl buffer	1.45	0.42

Nitrite		
	Traditional Method 10-107-05-1-B	ULF Method 80-107-05-1-A
	Cycle Period = 45 sec	Cycle Period = 60 sec
	Flow (mL per cycle period)	
Sulfanilamide	0.57	0.42
NH4Cl buffer	1.09	0.42
Carrier (DI)	1.32	1.55

Orthophosphorus		
	Traditional Method 10-115-01-1-A	ULF Method 80-115-01-1-A
	Cycle Period = 60 sec	Cycle Period = 60 sec
	Flow (mL per cycle period)	
Molybdate	1.28	0.22
Ascorbic Acid	1.28	0.22
Carrier (DI)	2.95	1.55

Ammonia (Phenolate) Method

QuikChem Method 80-107-06-1-A

Ammonia (Phenolate) in Potable, Surface, and Wastewaters

(un-preserved samples)

0.25-20 mg N/L

0.1 to 5.0 mg N/L

0.01-1.0 mg N/L

– Principle –

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

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.

The ULF series of methods use a pump speed of 10, and so must be run alone or with other ULF methodologies.

– Interferences –

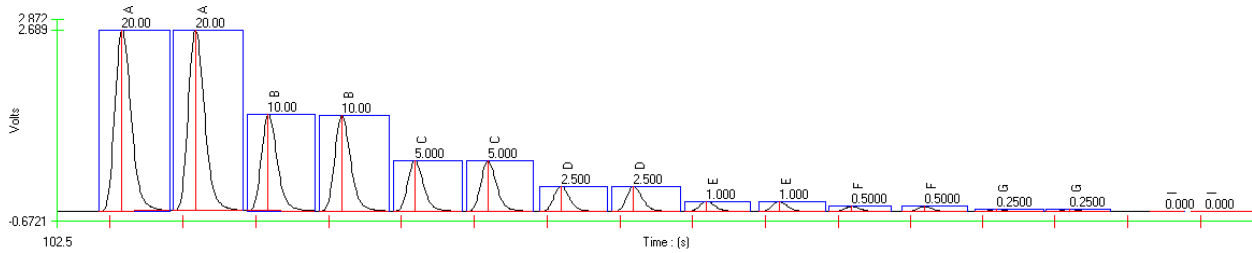
1. Calcium and magnesium ions may precipitate if present in sufficient concentration. EDTA is added to the sample in-line in order to prevent this problem.
2. Color, turbidity and certain organic species may interfere. Turbidity is removed by manual filtration. Sample color may be corrected for by running the samples through the manifold without color formation.
3. Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at the 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 samples.

– 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. PVC PUMP TUBES MUST BE USED FOR THIS METHOD.

Calibration Data for Ammonia Range 1



File Name: supp hi 7 28 09.omn
Acq. Date: 28 July 2009

Calibration Graph and Statistics

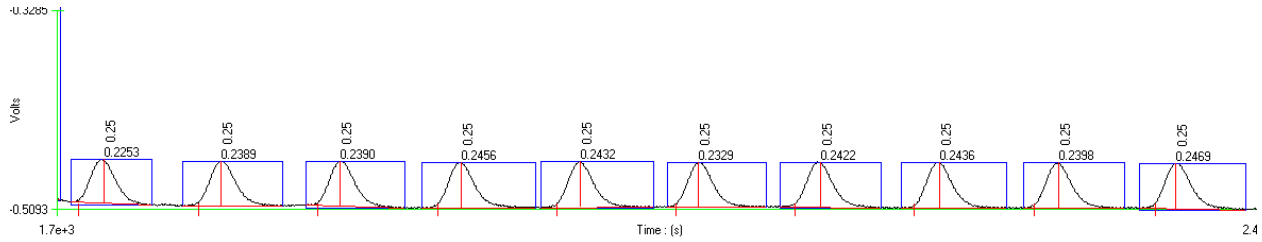
Ammonia N

Known Conc.	Rep.	Peak Area (V.s)	Peak Height (V)	% RSD	% Resid.	Unused	Det. Conc	Date	Time
20.00	1	55.40	3.186	0.3	-0.4	<input type="checkbox"/>	20.08	7/28/2009	1:55:49 PM
20.00	2	55.14	3.176	0.3	0.1	<input type="checkbox"/>	19.97	7/28/2009	1:56:57 PM
10.00	1	28.51	1.686	0.2	1.0	<input type="checkbox"/>	9.910	7/28/2009	1:58:04 PM
10.00	2	28.58	1.689	0.2	0.7	<input type="checkbox"/>	9.937	7/28/2009	1:59:12 PM
5.000	1	14.88	0.8877	0.4	-1.3	<input type="checkbox"/>	5.065	7/28/2009	2:00:23 PM
5.000	2	14.81	0.8835	0.4	-0.8	<input type="checkbox"/>	5.039	7/28/2009	2:01:26 PM
2.500	1	7.428	0.4443	0.3	-0.3	<input type="checkbox"/>	2.503	7/28/2009	2:02:33 PM
2.500	2	7.398	0.4395	0.3	0.1	<input type="checkbox"/>	2.493	7/28/2009	2:03:40 PM
1.000	1	2.954	0.1758	0.3	0.3	<input type="checkbox"/>	0.9944	7/28/2009	2:04:47 PM
1.000	2	2.967	0.1778	0.3	-0.1	<input type="checkbox"/>	0.9987	7/28/2009	2:05:54 PM
0.5000	1	1.506	0.08983	0.7	-2.3	<input type="checkbox"/>	0.5106	7/28/2009	2:07:00 PM
0.5000	2	1.521	0.08976	0.7	-3.4	<input type="checkbox"/>	0.5158	7/28/2009	2:08:07 PM
0.2500	1	0.6555	0.04095	6.2	9.3	<input type="checkbox"/>	0.2279	7/28/2009	2:09:14 PM
0.2500	2	0.7159	0.04217	6.2	0.9	<input type="checkbox"/>	0.2479	7/28/2009	2:10:20 PM
0.000	1	0.01825	1.841e-3			<input type="checkbox"/>	0.01645	7/28/2009	2:11:27 PM
0.000	2	-0.04723	-1.586e-3			<input type="checkbox"/>	-5.247e-3	7/28/2009	2:12:33 PM

Area = - 0.01216 * Conc^2 + 3.004 * Conc - 0.02773
 Conc = 5.558e-4 * Area^2 + 0.3314 * Area + 0.01040
 Correlation Coefficient (r) = 0.99999

Weighting : 1/x

File Name: supp hi 7 28 09.omn
Acq. Date: 28 July 2009



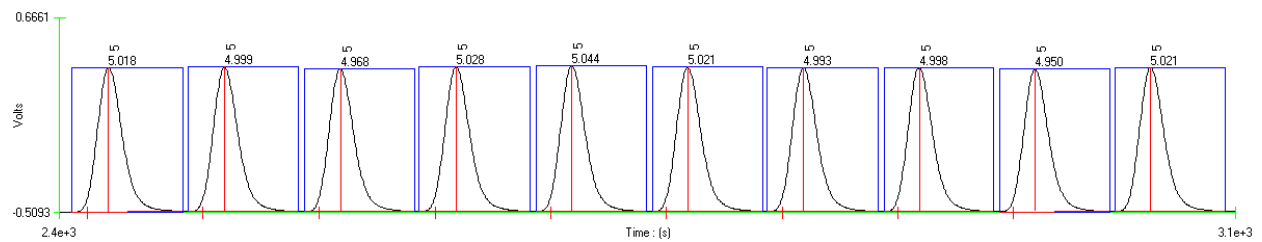
Method Detection Limit for ammonia using 0.25 mg N/L standard

MDL= 0.05 mg N/L

Standard Deviation (s) = 0.0065 mg N/L, Mean (x) = 0.24 mg N/L, Known value = 0.25 mg N/L

File Name: supp hi 7 28 09.omn

Acq. Date: 28 July 2009



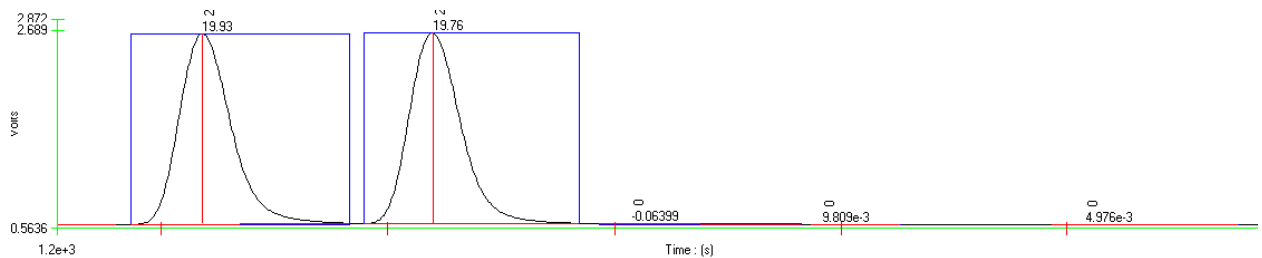
Precision data for ammonia using 5.0 mg N/L standard

% RSD = 0.57

Standard Deviation (s) = 0.029 mg N/L, Mean (x) = 5.004mg N/L, Known value = 5.0 mg N/L

File Name: supp hi 7 28 09.omn

Acq. Date: 28 July 2009

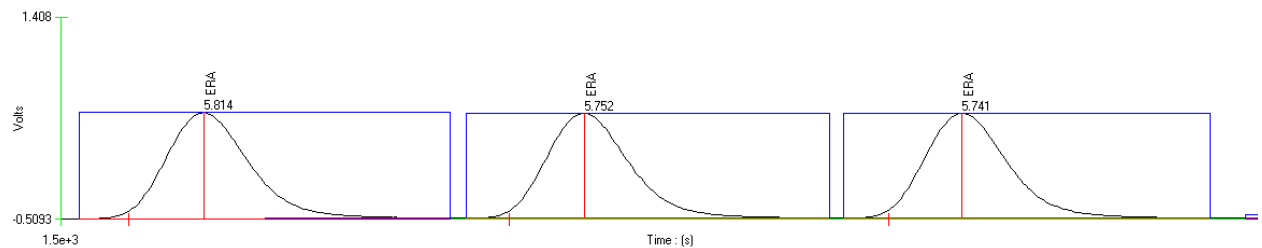


Carryover Study: 20 mg N/L standard followed by 3 blanks

Carryover Passed

File Name: supp hi 7 28 09.omn

Acq. Date: 28 July 2009



External QC: ERA Simple Nutrients

Average Determined Value: 5.77 mg N/L

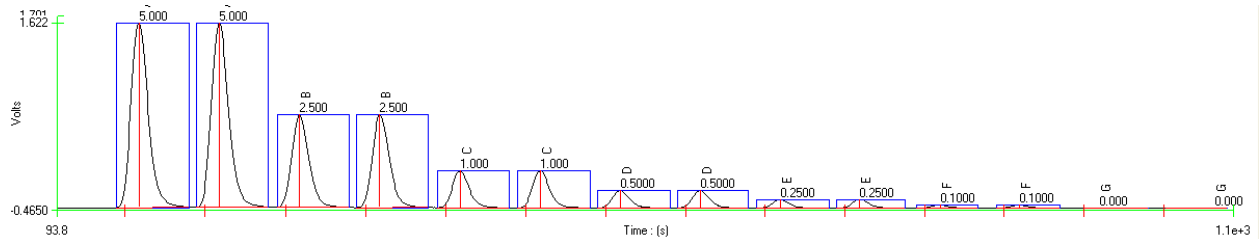
Known: 5.83 mg N/L

Acceptance Range: 4.85 - 6.81 mg N/L

ERA PN 739 Lot No: 1007739A

File Name: supp hi 7 28 09.omn
 Acq. Date: 28 July 2009

Calibration Data for Ammonia Range 2



File Name: supp 3 7 27 09 Mid Range un P.omn
 Acq. Date: 27 July 2009

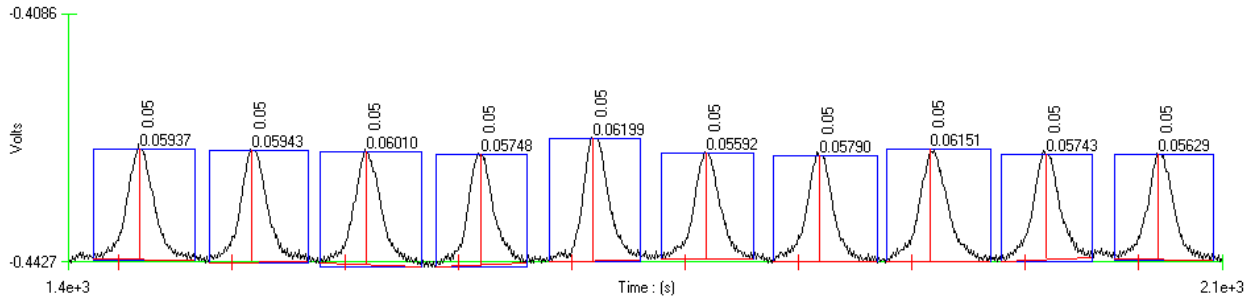
Calibration Graph and Statistics

Ammonia N

	Known Conc.	Rep.	Peak Area (V.s)	Peak Height (V)	% RSD	% Resid.	Unused	Det. Conc	Date	Time
1	5.000	1	34.42	2.056	0.2	-0.1	<input type="checkbox"/>	5.005	7/27/2009	10:48:52 AM
2	5.000	2	34.33	2.052	0.2	0.1	<input type="checkbox"/>	4.993	7/27/2009	10:50:00 AM
3	2.500	1	17.14	1.035	0.0	-0.1	<input type="checkbox"/>	2.502	7/27/2009	10:51:07 AM
4	2.500	2	17.15	1.034	0.0	-0.1	<input type="checkbox"/>	2.503	7/27/2009	10:52:15 AM
5	1.000	1	6.738	0.4079	1.3	0.5	<input type="checkbox"/>	0.9950	7/27/2009	10:53:22 AM
6	1.000	2	6.865	0.4138	1.3	-1.4	<input type="checkbox"/>	1.013	7/27/2009	10:54:29 AM
7	0.5000	1	3.228	0.1950	0.5	2.8	<input type="checkbox"/>	0.4864	7/27/2009	10:55:36 AM
8	0.5000	2	3.248	0.1960	0.5	2.2	<input type="checkbox"/>	0.4894	7/27/2009	10:56:43 AM
9	0.2500	1	1.575	0.09486	0.9	1.4	<input type="checkbox"/>	0.2469	7/27/2009	10:57:50 AM
10	0.2500	2	1.596	0.09593	0.9	0.0	<input type="checkbox"/>	0.2500	7/27/2009	10:58:57 AM
11	0.1000	1	0.5611	0.03420	1.3	0.0	<input type="checkbox"/>	0.1000	7/27/2009	11:00:03 AM
12	0.1000	2	0.5510	0.03386	1.3	1.8	<input type="checkbox"/>	0.09857	7/27/2009	11:01:10 AM
13	0.000	1	-0.05336	-2.443e-3			<input type="checkbox"/>	0.01101	7/27/2009	11:02:17 AM
14	0.000	2	-0.08642	-2.546e-3			<input type="checkbox"/>	6.219e-3	7/27/2009	11:03:23 AM

Area = 6.902 * Conc - 0.1292
 Conc = 0.1449 * Area + 0.01874
 Correlation Coefficient (r) = 0.99999
 Weighting : None

File Name: supp 3 7 27 09 Mid Range un P.omn
 Acq. Date: 27 July 2009



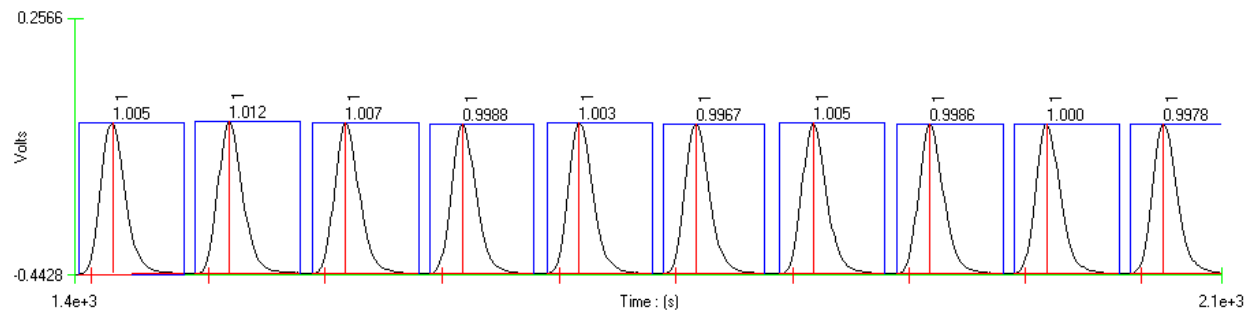
Method Detection Limit for ammonia using 0.05 mg N/L standard

MDL= 0.006; Claiming 0.015 due to y-intercept

Standard Deviation (s) = 0.0021 mg N/L, Mean (x) = 0.0587 mg N/L, Known value = 0.05 mg N/L

File Name: supp 2 7 27 09 Mid Range un P.omn

Acq. Date: 27 July 2009



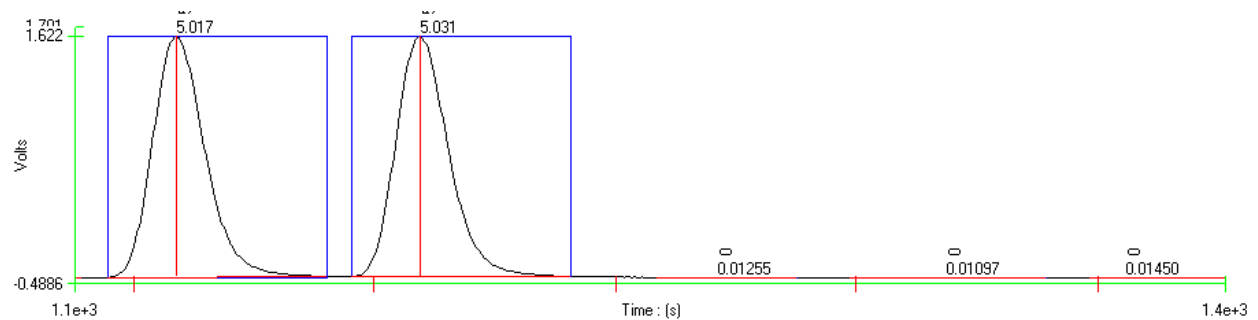
Precision data for ammonia using 1.0 mg N/L standard

% RSD = 0.49%

Standard Deviation (s) = 0.0049 mg N/L, Mean (x) = 1.0024 mg N/L, Known value = 1.0 mg N/L

File Name: supp 3 7 27 09 Mid Range un P.omn

Acq. Date: 27 July 2009

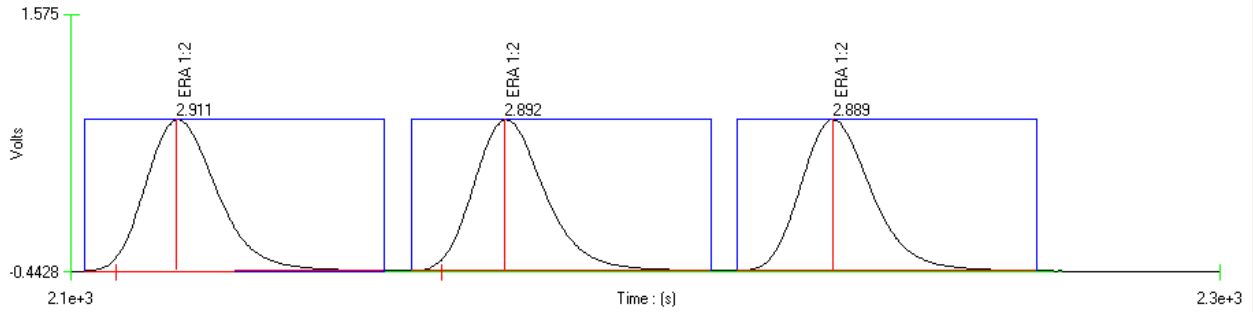


Carryover Study: 5.0 mg N/L standard followed by 3 blanks

Carryover Passed

File Name: supp 3 7 27 09 Mid Range un P.omn

Acq. Date: 27 July 2009



External QC: ERA Simple Nutrients, Diluted 1:2 to bring it within the range of the method.

Average Determined Value: 2.897 mg N/L

Known: 2.915 mg N/L

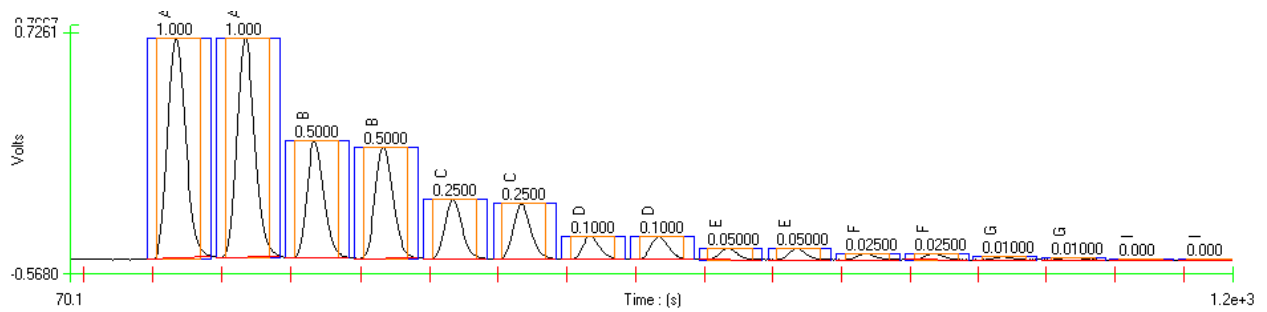
Acceptance Range: 2.425 -3.405 mg N/L

ERA PN 739 Lot No: 1007739A

File Name: supp 3 7 27 09 Mid Range un P.omn

Acq. Date: 27 July 2009

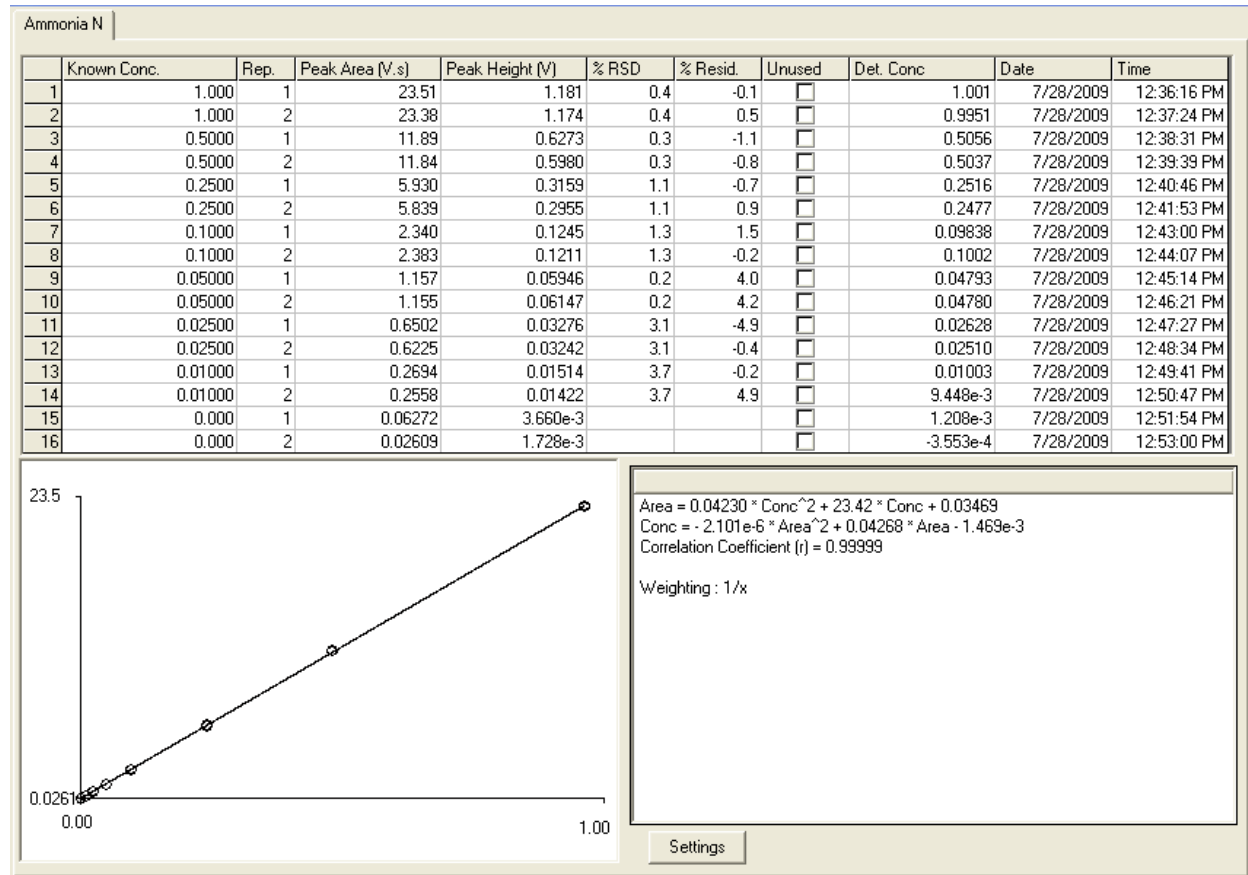
Calibration Data for Ammonia Range 3



File Name: supp 1 7 28 09.omn

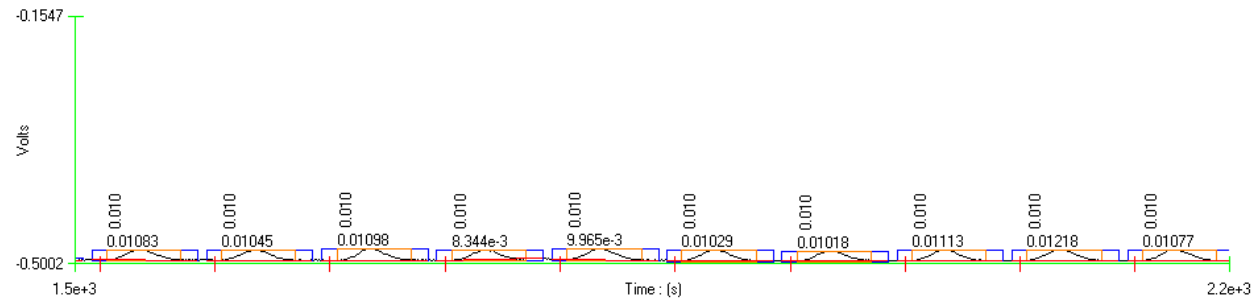
Acq. Date: 28 July 2009

Calibration Graph and Statistics



File Name: supp 1 7 28 09.omn

Acq. Date: 28 July 2009



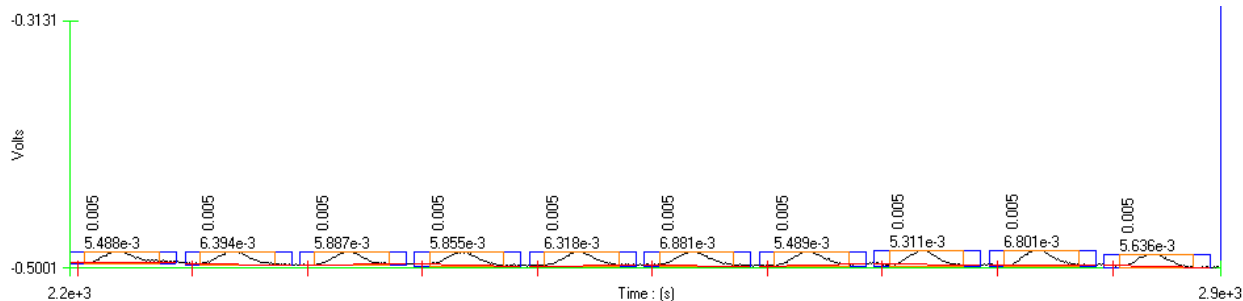
Method Detection Limit for ammonia using 0.01 mg N/L standard

MDL= 0.0027 mg N/L

Standard Deviation (s) = 0.001 mg N/L, Mean (x) = 0.0105 mg N/L, Known value = 0.01 mg N/L

File Name: supp 1 7 28 09.omn

Acq. Date: 28 July 2009



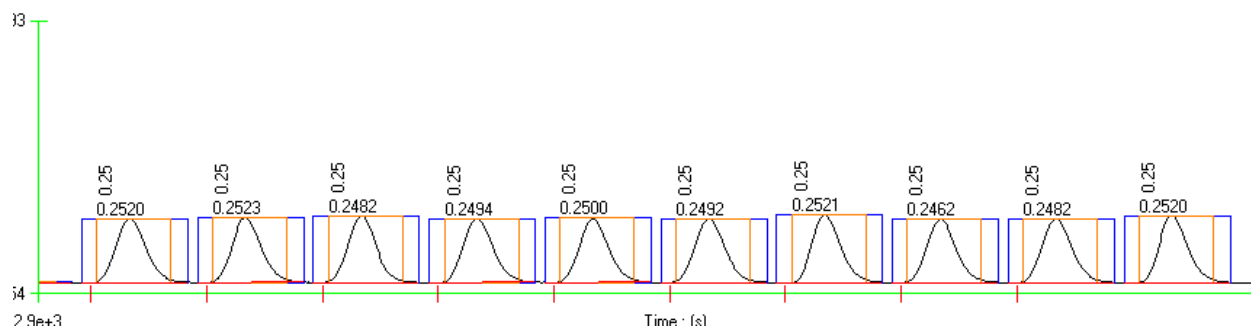
Method Detection Limit for ammonia using 0.005 mg N/L standard

MDL= 0.0021 mg N/L

Standard Deviation (s) = 0.00056 mg N/L, Mean (x) = 0.006 mg N/L, Known value = 0.005 mg N/L

File Name: supp 1 7 28 09.omn

Acq. Date: 28 July 2009



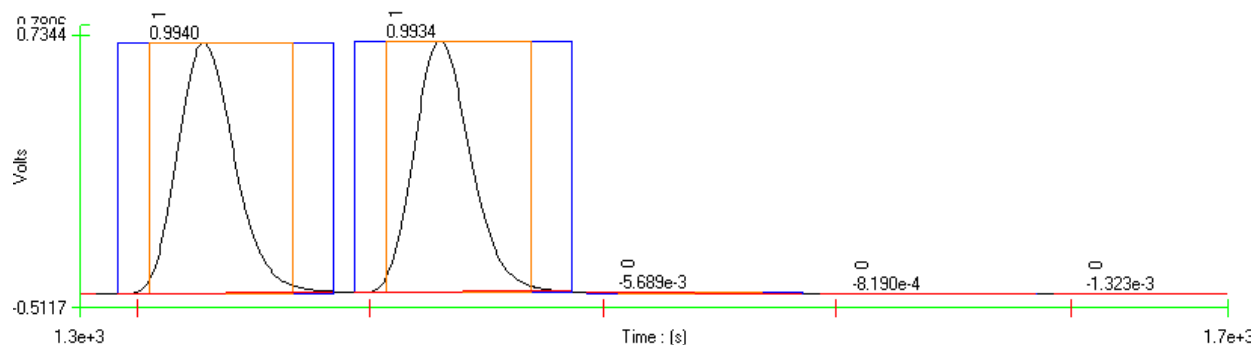
Precision data for ammonia using 0.25 mg N/L standard

% RSD = 0.84

Standard Deviation (s) = 0.0021 mg N/L, Mean (x) = 0.249 mg N/L, Known value = 0.25 mg N/L

File Name: supp 1 7 28 09.omn

Acq. Date: 28 July 2009



Carryover Study: 1.0 mg N/L standard followed by 10 blanks

Carryover Passed

File Name: supp 1 7 27 09.omn

Acq. Date: 27 July 2009

For acid-preserved samples, 80-107-06-1-B (high and mid-range) and 80-107-06-1-C (low range) are also available.

Chloride, Mercuric Thiocyanate Method

QuikChem® Method 80-117-07-1-A

Chloride in Waters

(ULTRA LOW FLOW METHOD)

6.0 to 300.0 mg Cl⁻/L

2.5-100 mg Cl⁻/L

0.25-20 mg Cl⁻/L

– Principle –

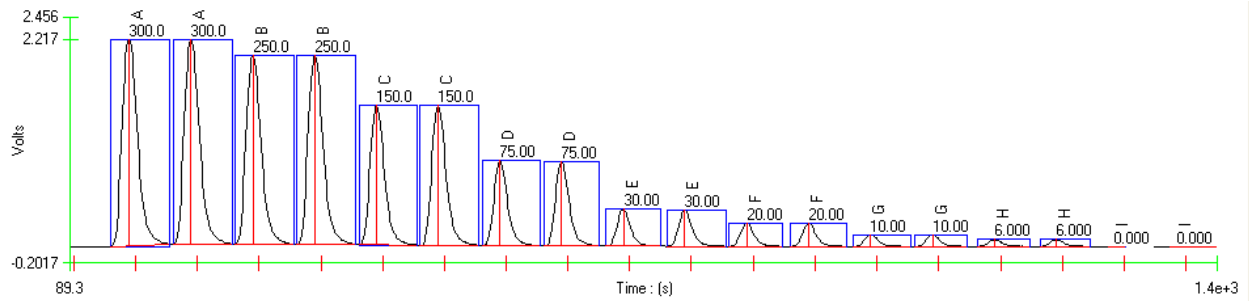
Thiocyanate ion is liberated from mercuric thiocyanate by the formation of soluble mercuric chloride. In the presence of ferric ion, free thiocyanate ion forms the highly colored ferric thiocyanate, of which the absorbance is proportional to the chloride concentration. Ferric thiocyanate absorbs strongly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

The 80- series of methods (Ultra Low Flow) use a pump speed of 10, and so must be run alone or with other ULF methodologies.

– 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⁻, I⁻) give a positive interference.

Calibration Data for Chloride, Range 1



File Name: OM_7-8-2009_9-52-11AM.OMN
Acq. Date: 08 July 2009

Calibration Graph and Statistics

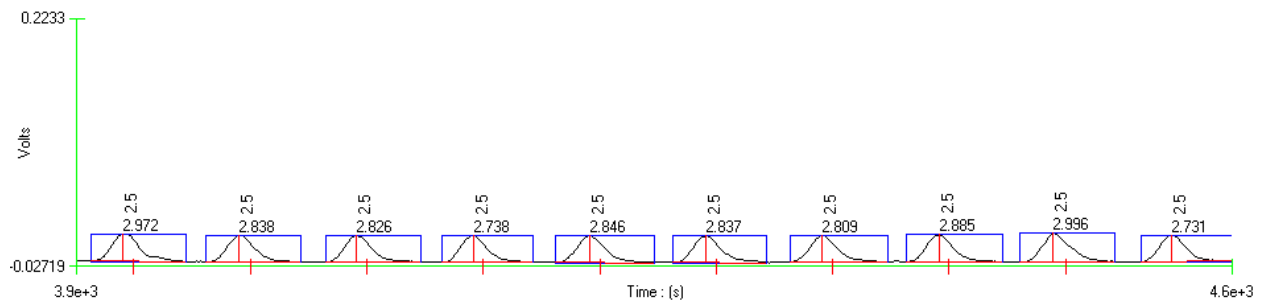
Known Conc.	Rep.	Peak Area (V.s)	Peak Height (V)	% RSD	% Resid.	Unused	Det. Conc	Date	Time
300.0	1	45.01	2.235	0.8	-1.5	<input type="checkbox"/>	296.0	7/8/2009	9:53:46
300.0	2	44.50	2.218	0.8	-0.4	<input type="checkbox"/>	291.2	7/8/2009	9:54:56
250.0	1	40.04	2.042	0.4	1.3	<input type="checkbox"/>	250.4	7/8/2009	9:56:06
250.0	2	40.26	2.052	0.4	0.7	<input type="checkbox"/>	252.4	7/8/2009	9:57:15
150.0	1	28.10	1.509	1.0	2.0	<input type="checkbox"/>	154.1	7/8/2009	9:58:25
150.0	2	28.49	1.522	1.0	0.6	<input type="checkbox"/>	157.0	7/8/2009	9:59:34
75.00	1	16.32	0.9167	0.2	-2.4	<input type="checkbox"/>	77.32	7/8/2009	10:00:43
75.00	2	16.26	0.9136	0.2	-2.0	<input type="checkbox"/>	77.02	7/8/2009	10:01:53
30.00	1	6.885	0.3961	0.2	-2.1	<input type="checkbox"/>	28.93	7/8/2009	10:03:02
30.00	2	6.870	0.3921	0.2	-1.9	<input type="checkbox"/>	28.86	7/8/2009	10:04:11
20.00	1	4.452	0.2554	0.6	1.9	<input type="checkbox"/>	18.33	7/8/2009	10:05:20
20.00	2	4.413	0.2529	0.6	2.8	<input type="checkbox"/>	18.17	7/8/2009	10:06:31

Area = - 2.898e-4 * Conc^2 + 0.2348 * Conc - 0.04134
 Conc = 0.06532 * Area^2 + 3.615 * Area + 0.9440
 Correlation Coefficient (r) = 0.99997

Weighting : 1/x

Settings

File Name: OM_7-8-2009_9-52-11AM.OMN
Acq. Date: 08 July 2009

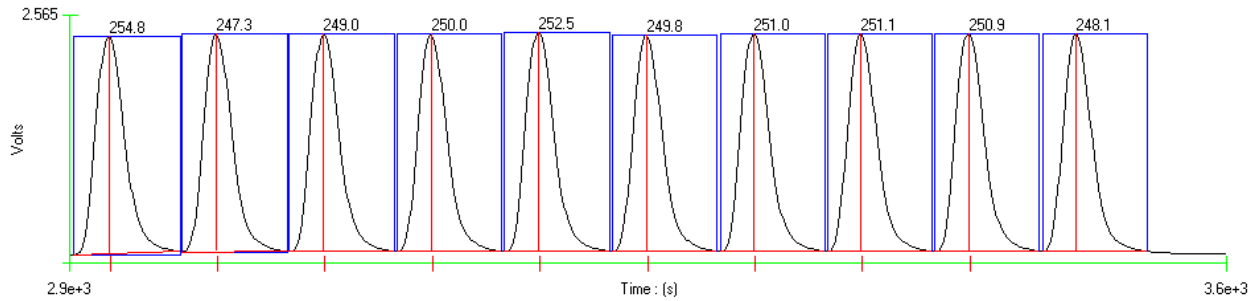


Method Detection Limit for chloride using 2.5 mg Cl/L standard

MDL= 1.0 mg Cl/L (due to y-intercept) (Calculated value 0.24 mg Cl/L)

Standard Deviation (s) = 0.086 mg Cl/L, Mean (x) = 2.85 mg Cl/L, Known value = 2.5 mg Cl/L

File Name: OM_7-8-2009_9-52-11AM.OMN
Acq. Date: 08 July 2009



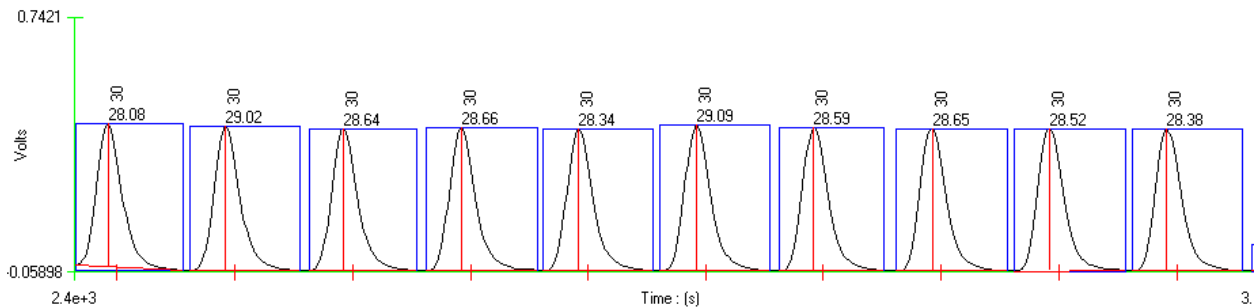
Precision data for chloride using 250 mg Cl/L standard

% RSD = 0.87

Standard Deviation (s) = 2.17 mg Cl/L, Mean (x) = 250.45 mg Cl/L, Known value = 250 mg Cl/L

File Name: 6 30 high 2.OMN

Acq. Date: 30 June 2009



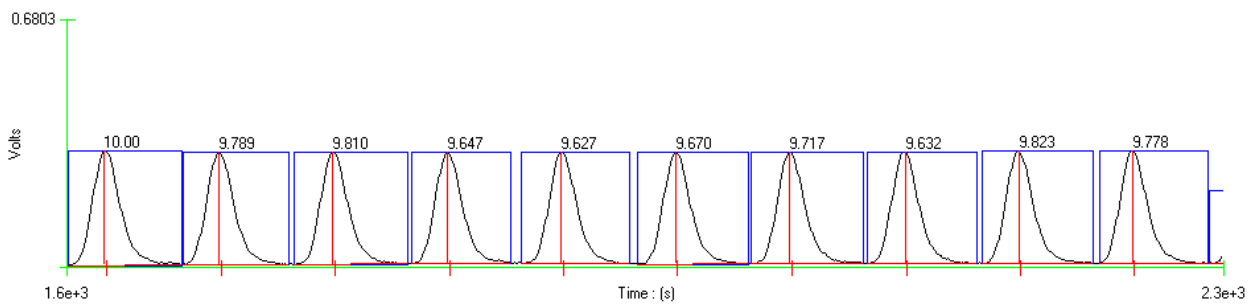
Precision data for chloride using 30 mg Cl/L standard

% RSD = 1.05

Standard Deviation (s) = 0.30 mg Cl/L, Mean (x) = 28.6 mg Cl/L, Known value = 30 mg Cl/L

File Name: 6 30 high 2.OMN

Acq. Date: 30 June 2009



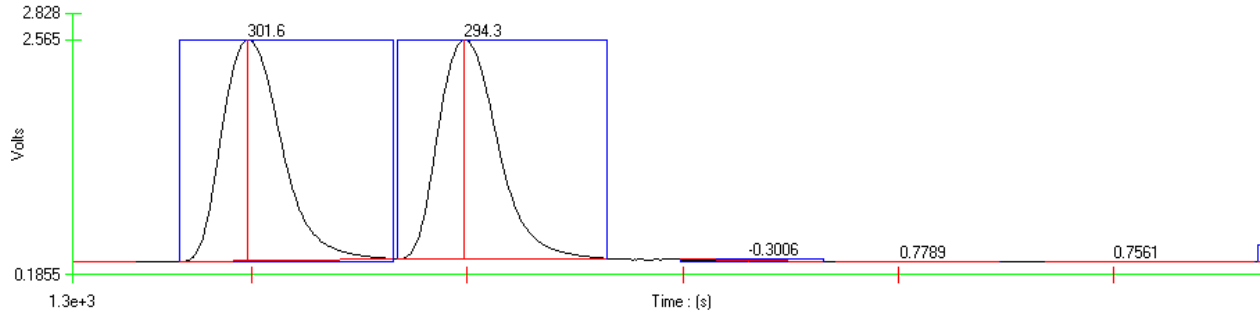
Precision data for chloride using 10 mg Cl/L standard

% RSD = 1.19

Standard Deviation (s) = 0.115 mg Cl/L, Mean (x) = 9.75 mg Cl/L, Known value = 10 mg Cl/L

File Name: 6 30 high 2.OMN

Acq. Date: 30 June 2009



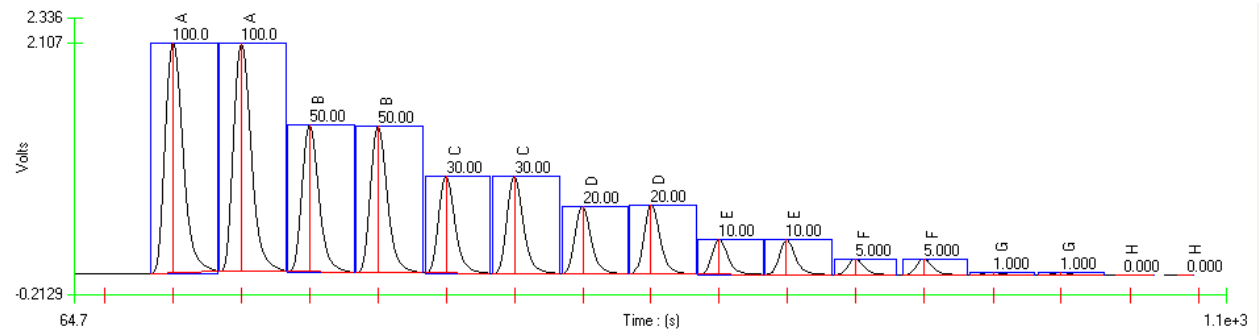
Carryover Study: 300 mg Cl/L standard followed by 3 blanks

Carryover Passed

File Name: 6 30 high 2.OMN

Acq. Date: 30 June 2009

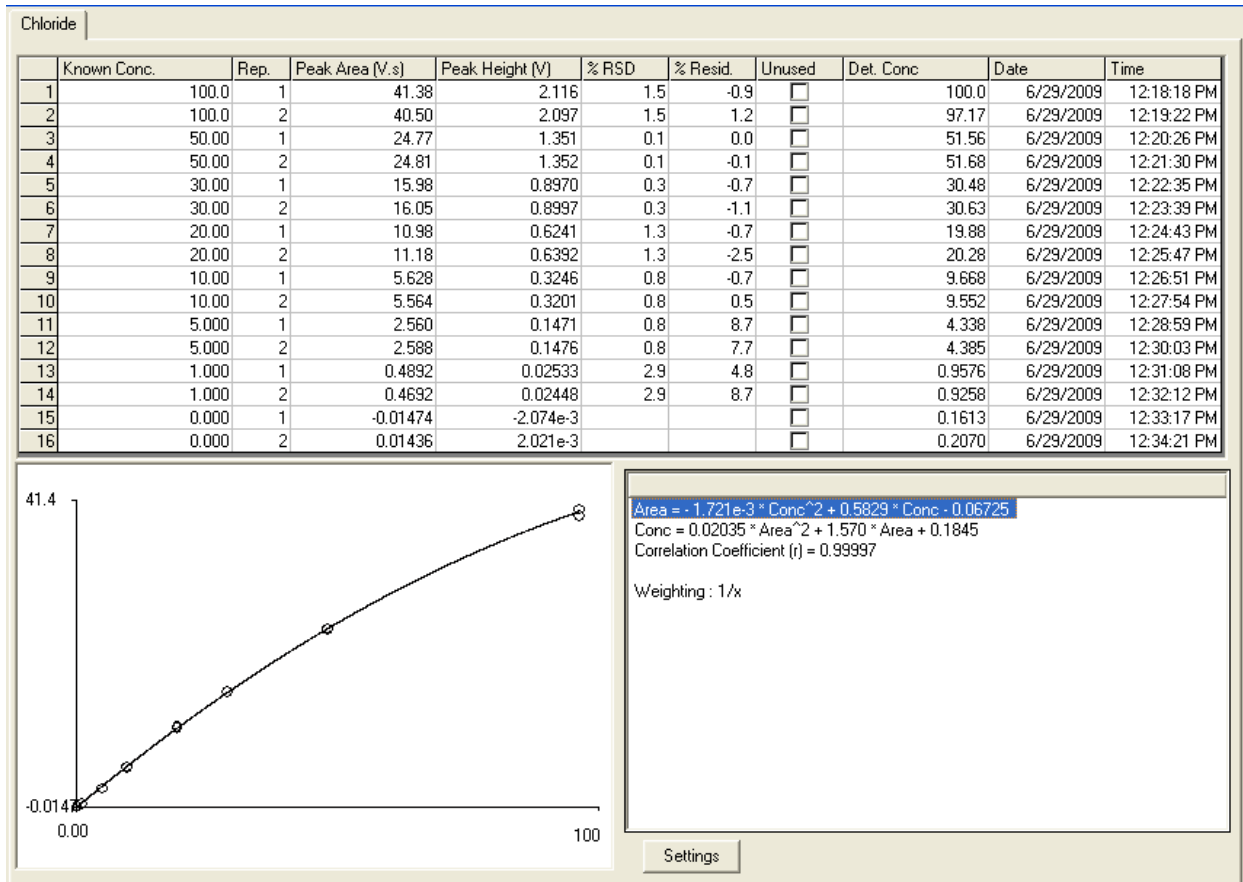
Calibration Data for Chloride, Range 2



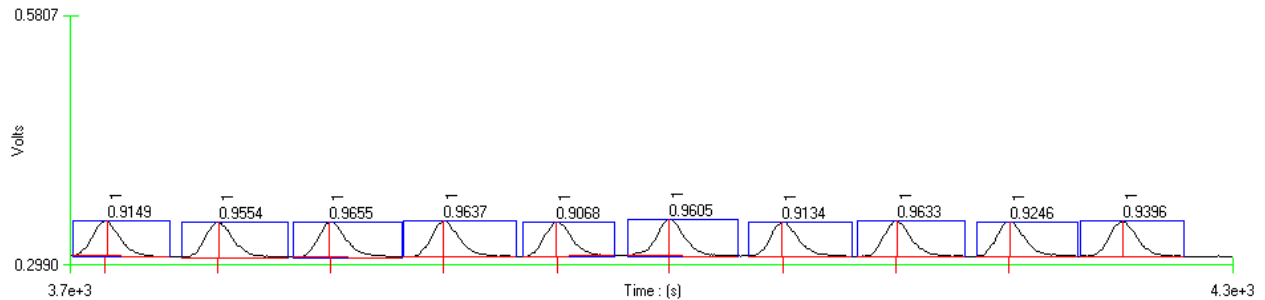
File Name: supp low 1 6 30 09.OMN

Acq. Date: 30 June 2009

Calibration Graph and Statistic



File Name: supp low 1 6 30 09.OMN
 Acq. Date: 30 June 2009



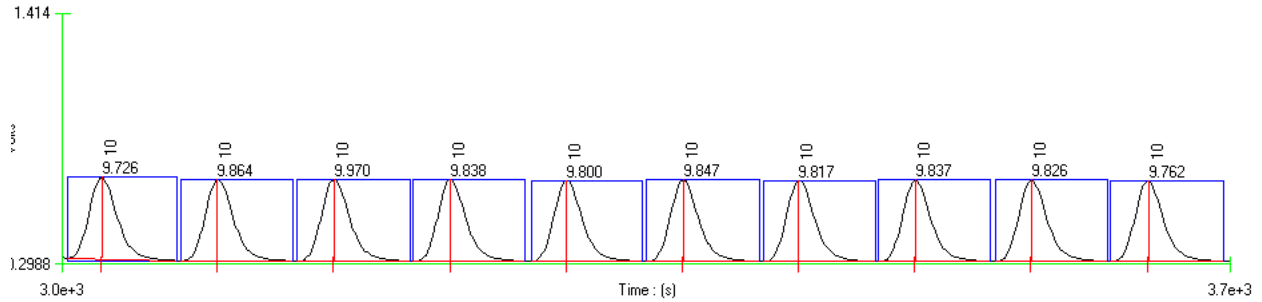
Method Detection Limit for chloride using 1.0 mg Cl/L standard

MDL= 0.2 mg Cl/L due to y-intercept (calculated as 0.07 mg Cl/L)

Standard Deviation (s) = 0.025mg Cl/L, Mean (x) = 0.94 mg Cl/L, Known value = 2.5 mg Cl/L

File Name: ULF Cl low.OMN

Acq. Date: 26 June 2009



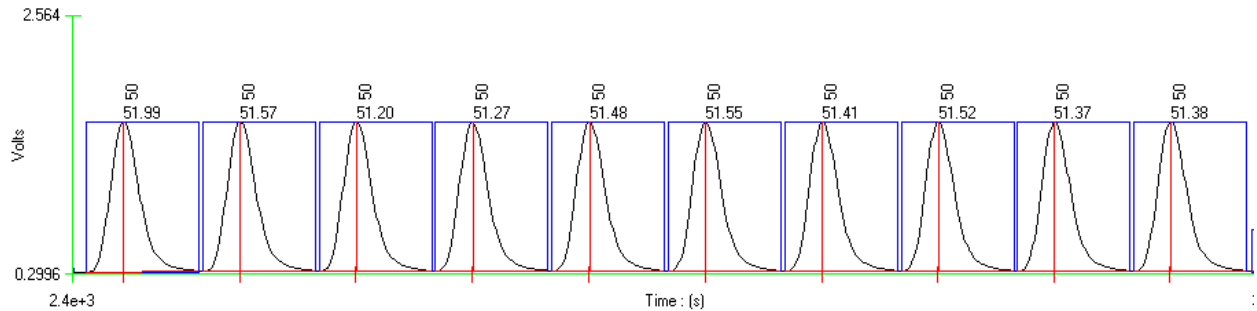
Precision data for chloride using 10 mg Cl/L standard

% RSD = 0.69

Standard Deviation (s) = 0.068 mg Cl/L, Mean (x) = 9.83 mg Cl/L, Known value = 10 mg Cl/L

File Name: ULF Cl low.OMN

Acq. Date: 26 June 2009



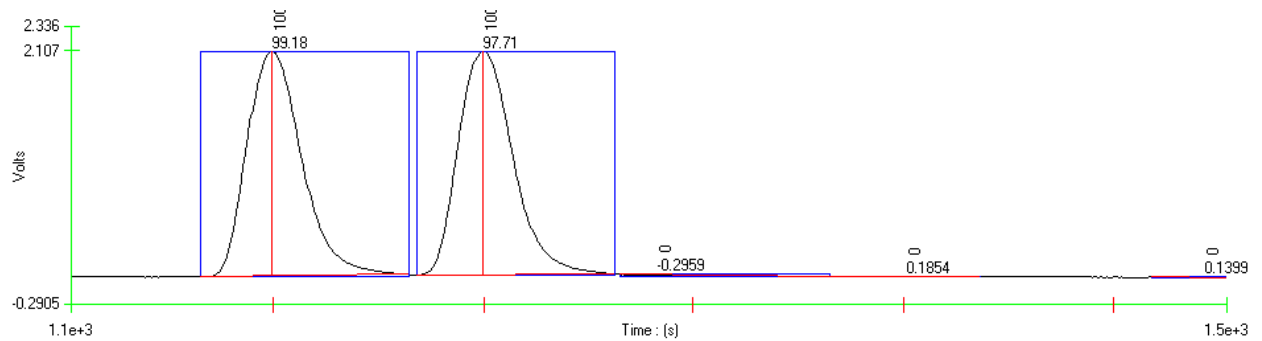
Precision data for chloride using 50 mg Cl/L standard

% RSD = 0.41

Standard Deviation (s) = 0.21 mg Cl/L, Mean (x) = 51.47 mg Cl/L, Known value = 50 mg Cl/L

File Name: ULF Cl low.OMN

Acq. Date: 26 June 2009

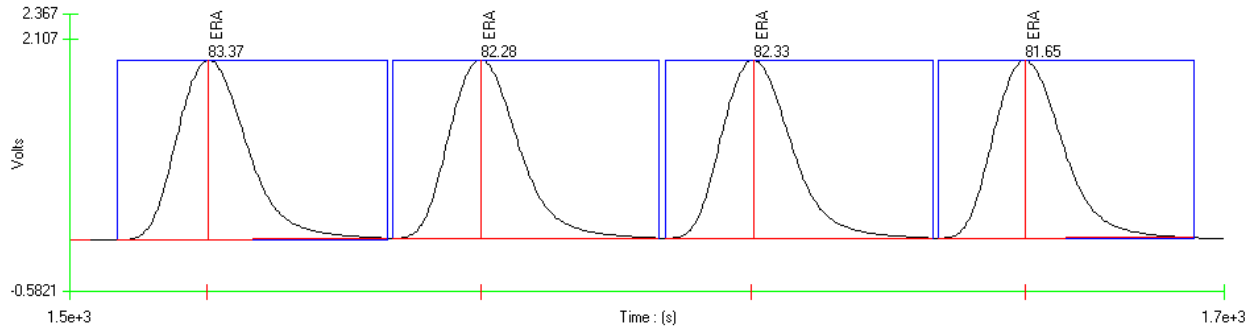


Carryover Study: 100 mg Cl/L standard followed by 3 blanks

Carryover Passed

File Name: supp low 1 6 30 09.OMN

Acq. Date: 30 June 2009



ERA

Measured value: 82.41mg/L

Certified value: 78.5 mg/L

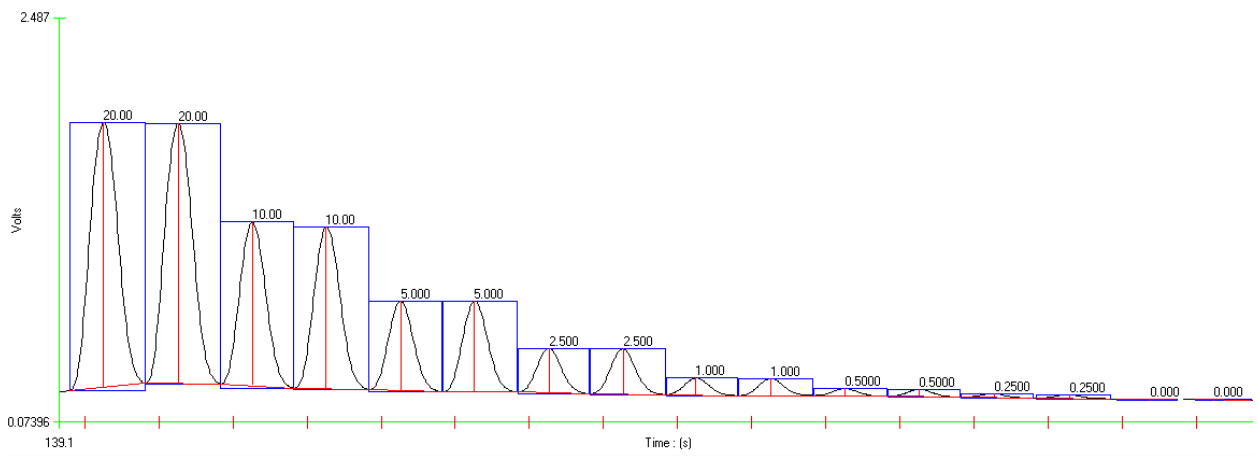
Acceptable Range: 67.3-89.7 mg/L

ERA PN 698; Lot Number S077698

File Name: supp low 1 6 30 09.OMN

Acq. Date: 30 June 2009

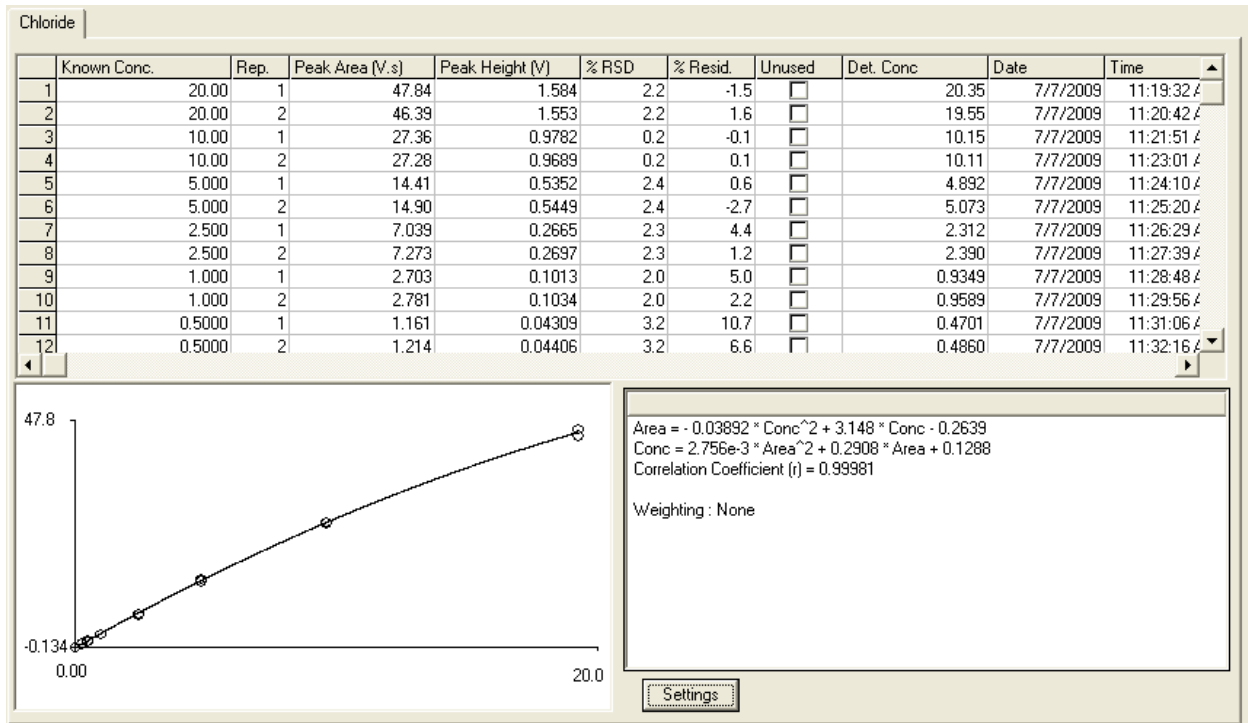
Calibration Data for Chloride, Range 3



File Name: low low 3.omn

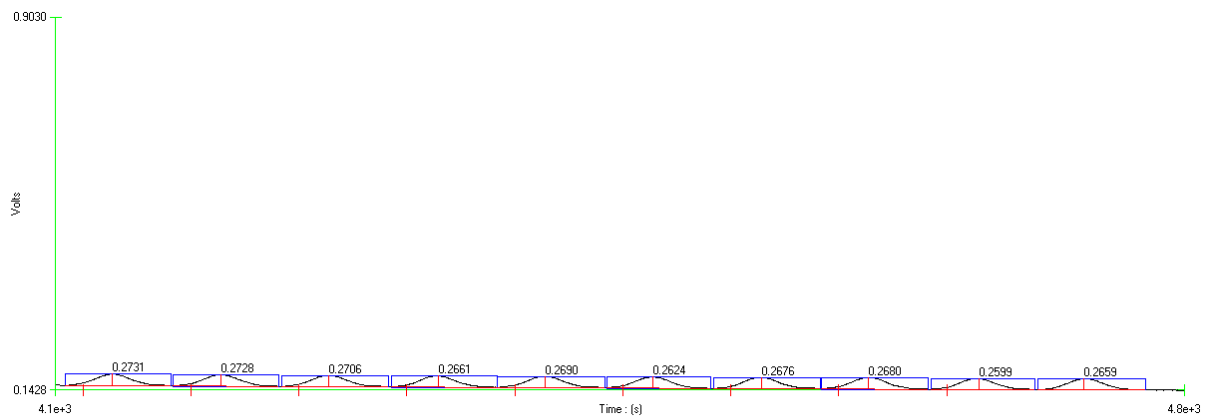
Acq. Date: 07 July 2009

Calibration Graph and Statistics



File Name: low low 3.omn

Acq. Date: 07 July 2009



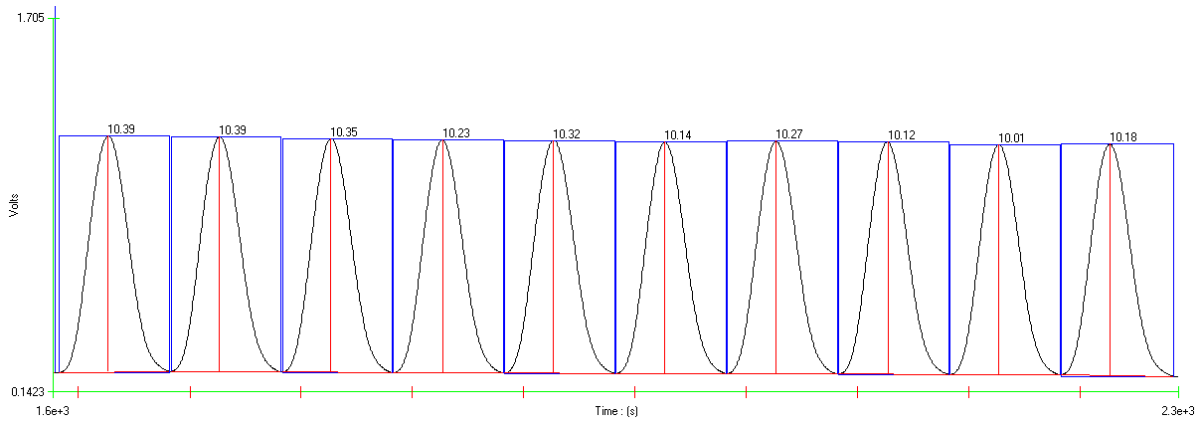
Method Detection Limit for chloride using 0.25 mg Cl/L standard

MDL= 0.13 mg Cl, due to y-intercept. (Calculated as 0.012 mg Cl/L)

Standard Deviation (s) = 0.0042 mg Cl/L, Mean (x) = 0.268 mg Cl/L, Known value = 0.25 mg Cl/L

File Name: low low 3.omn

Acq. Date: 07 July 2009



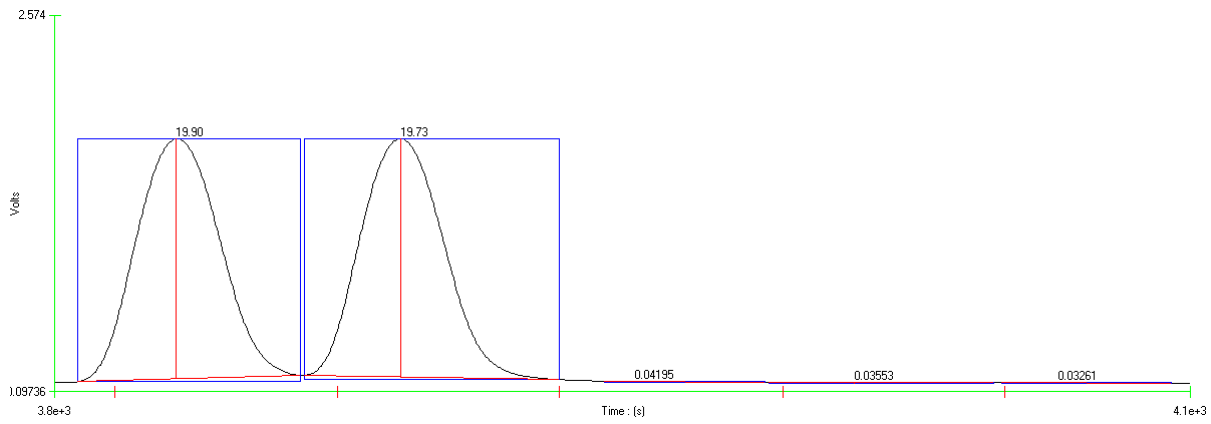
Precision data for chloride using 10 mg Cl/L standard

% RSD = 1.24

Standard Deviation (s) = 0.124mg Cl/L, Mean (x) = 10.24 mg Cl/L, Known value = 10 mg Cl/L

File Name: low low 3.omn

Acq. Date: 07 July 2009



Carryover Study: 20 mg Cl/L standard followed by 3 blanks

Carryover Passed

File Name: low low 3.omn

Acq. Date: 07 July 2009

Cyanide, Pyridine Barbituric Acid Method

QuikChem® Method 80-204-00-1-X

Cyanide in Drinking and Wastewaters using MICRO

DIST™

(Ultra Low Flow Method)

0.005 to 0.5 mg CN⁻/L

– Principle –

By means of a passive miniature distillation device, MICRO DIST, the cyanide in the samples is released by digesting and acidifying cyanide complexes, converting them to hydrocyanic acid (HCN). The cyanide ion is trapped in a 1.0 M sodium hydroxide absorbing solution which is diluted to 0.25 M solution during the distillation. By means of flow injection analysis, the 0.25 M NaOH distillate is converted to cyanogen chloride by reaction with chloramine-T, pyridine and barbituric acid to give a red-colored complex. The absorbance of this complex is measured at 570 nm by measuring the peak area resulting from the sample. The peak area is proportional to the concentration of the cyanide in the sample.

The determinative step of this method is described here only so that a complete method is provided. Any approved continuous-flow method may be used for the determination of cyanide in the 0.25 M NaOH MICRO DIST distillates.

***Note:** Other approved distillation procedures may use different concentrations of NaOH in the absorber or trapping solution. Because the determinative method's sensitivity is pH dependent, it is important to ensure that the pH of the absorber solution from any distillation procedure is adjusted to match the pH of calibration standards specified in the approved determinative method being used.*

USEPA Reference Method "Methods for the Chemical Analysis of Water and Wastes", EPA-600/4-79-020, Revised March 1983 and 1979, Method 335.2 specifies that absorbance be measured at 578 nm using a batch spectrophotometer. USEPA Reference Method "Methods for the Determination of Inorganic Substances in Environmental Samples", EPA-600/R-93-100, August 1993, Method 335.4 specifies that absorbance be measured at 570 nm. The latter wavelength is specified in this Modified Method because Method 335.4 uses an automated continuous flow-through detector similar to that used in the following flow injection method.

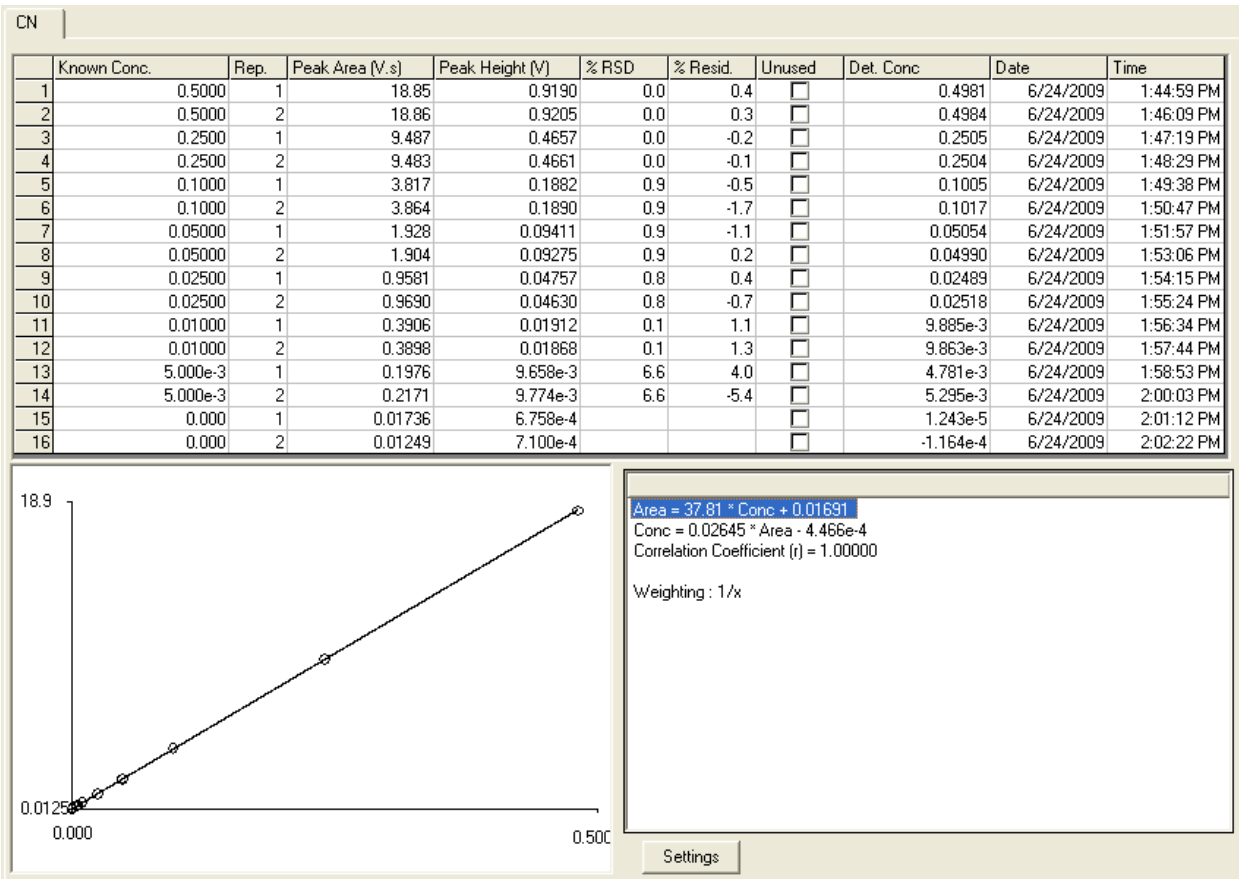
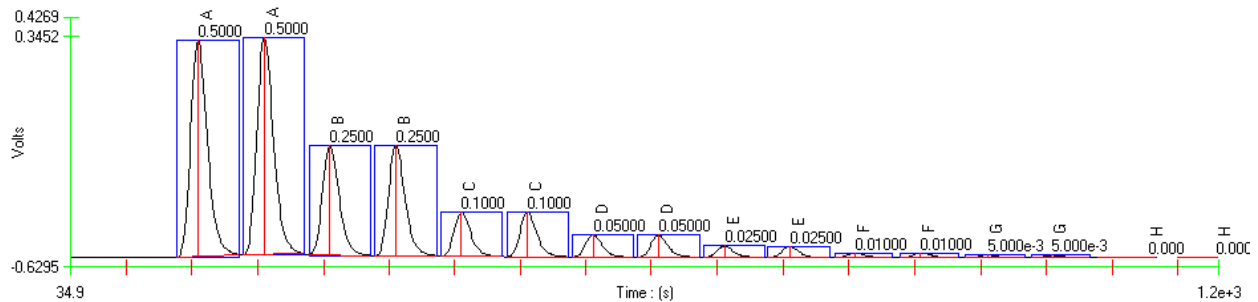
The 80- series of methods (Ultra Low Flow) use a pump speed of 10, and so must be run alone or with other ULF methodologies.

– Special Apparatus –

Please contact Lachat Sales for ordering information

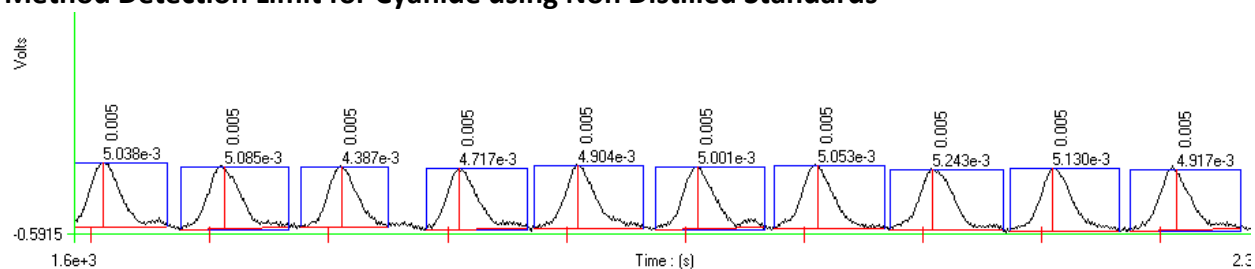
1. Heating Unit
2. MICRO DIST Block and Cyanide-1 tubes. The MICRO DIST block is available from Lachat Instruments, Loveland, Colorado as Lachat part number A17100. The Cyanide-1 tubes are available as Lachat part number A17001
3. Filter paper, No. 2

Calibration Graph and Statistics (Non-distilled standards)



File Name:092194M1.fdt
 Acq. Date: 21 September 1994

Method Detection Limit for Cyanide using Non Distilled Standards



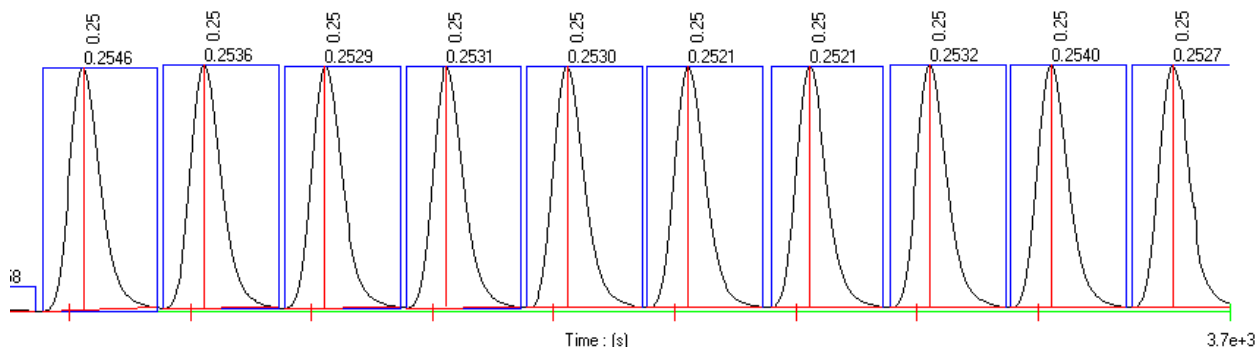
Method Detection Limit for cyanide using 0.005 mg/L standard

MDL = 0.001 mg/L

Standard Deviation (s) = 0.0001 mg/L, Mean (x) = 0.010 mg/L, Known value = 0.001 mg/L

File Name:092194M1.fdt

Acq. Date: 21 September 1994



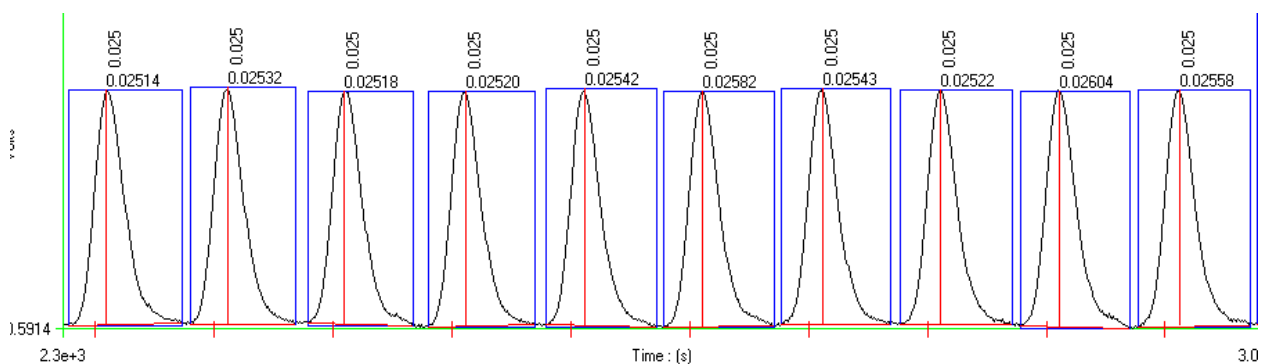
Precision data for Cyanide using 0.250 mg/L standard

% RSD = 0.31 %

Standard Deviation (s) = 0.00078 mg/L, Mean (x) = 0.253 mg/L, Known value = 0.250 mg/L

File Name:ULF CN RUN 2.OMN

Acq. Date: 24 June 2009



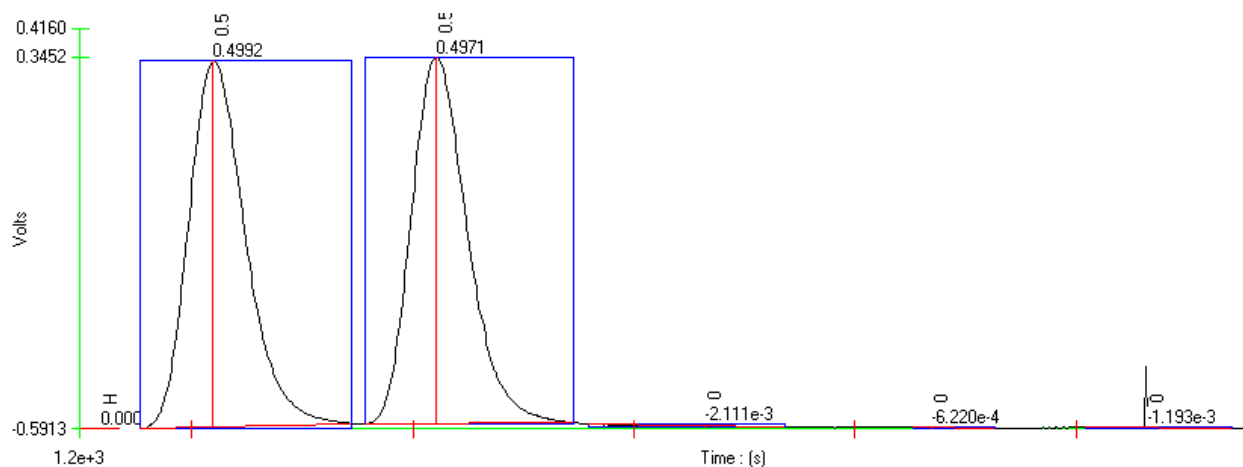
Precision data for Cyanide using 0.025 mg/L standard

% RSD = 1.15 %

Standard Deviation (s) = 0.000294 mg/L, Mean (x) = 0.0254 mg/L, Known value = 0.025 mg/L

File Name:ULF CN RUN 2.OMN

Acq. Date: 24 June 2009



Carryover test. Two injections of the high standard, followed by three blanks.

Carryover passed.

File Name:ULF CN RUN 2.OMN

Acq. Date: 24 June 2009

(A method for samples that have been prepared using a Macro Distillation, 80-204-00-1-A, is also available.)

Nitrate-Nitrite Cadmium Reduction Method

QuikChem® Method 80-107-04-1-A

Nitrate/Nitrite in Waters

(ULTRA LOW FLOW METHOD)

0.10 to 10.0 mg N/L as NO_3^- or NO_2^-

0.01 to 1.0 mg N/L as NO_3^- or NO_2^-

0.001 to 0.10 mg N/L as NO_3^- or NO_2^-

– Principle –

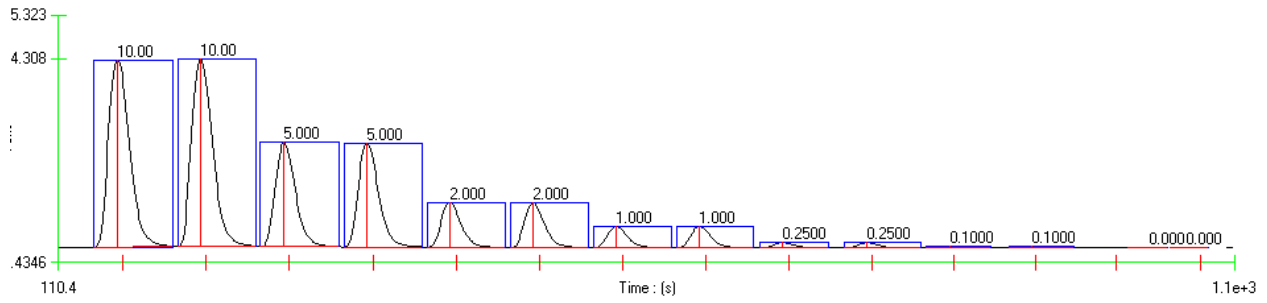
Nitrate is quantitatively reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite (reduced nitrate plus original nitrite) is then determined by diazotizing with sulfanilamide followed by coupling with N-(1-naphthyl)ethylenediamine dihydrochloride. The resulting water soluble dye has a magenta color which is read at 520 nm. Nitrite alone also can be determined by removing the cadmium column.

The 80- series of methods (Ultra Low Flow) use a pump speed of 10, and so must be run alone or with other ULF methodologies.

– Interferences –

1. Residual chlorine can interfere by oxidizing the cadmium column.
2. Low results would be obtained for samples that contain high concentrations of iron, copper or other metals. In this method, EDTA is added to the buffer to reduce this interference.
3. Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. This interference is eliminated by pre-extracting the sample with an organic solvent.
4. Sample turbidity may interfere. Turbidity can be removed by filtration through a 0.4 μM pore diameter membrane filter prior to analysis

Calibration Data for Nitrate/Nitrite Range 1: 0.1-10 mg N/L



Nitrate Calibration Graph and Statistics

Nitrate

Known Conc.	Rep.	Peak Area (V.s)	Peak Height (V)	% RSD	% Resid.	Unused	Det. Conc	Date	Time
10.00	1	88.04	4.379	0.3	-0.1	<input type="checkbox"/>	10.01	6/5/2009	10:57:57 AM
10.00	2	87.66	4.375	0.3	0.3	<input type="checkbox"/>	9.962	6/5/2009	10:59:06 AM
5.000	1	46.37	2.426	0.3	0.2	<input type="checkbox"/>	5.001	6/5/2009	11:00:14 AM
5.000	2	46.56	2.432	0.3	-0.2	<input type="checkbox"/>	5.022	6/5/2009	11:01:23 AM
2.000	1	19.63	1.042	0.3	-2.5	<input type="checkbox"/>	2.047	6/5/2009	11:02:31 AM
2.000	2	19.72	1.044	0.3	-3.0	<input type="checkbox"/>	2.057	6/5/2009	11:03:39 AM
1.000	1	9.273	0.4924	0.2	3.8	<input type="checkbox"/>	0.9580	6/5/2009	11:04:48 AM
1.000	2	9.303	0.4932	0.2	3.5	<input type="checkbox"/>	0.9610	6/5/2009	11:05:56 AM
0.2500	1	2.256	0.1200	0.9	4.9	<input type="checkbox"/>	0.2372	6/5/2009	11:07:05 AM
0.2500	2	2.283	0.1210	0.9	3.7	<input type="checkbox"/>	0.2400	6/5/2009	11:08:14 AM
0.1000	1	0.8929	0.04715	0.1	1.2	<input type="checkbox"/>	0.09890	6/5/2009	11:09:22 AM
0.1000	2	0.8943	0.04725	0.1	1.1	<input type="checkbox"/>	0.09904	6/5/2009	11:10:29 AM
0.000	1	-0.01411	-8.327e-4			<input type="checkbox"/>	7.121e-3	6/5/2009	11:11:36 AM
0.000	2	-0.01768	-1.110e-3			<input type="checkbox"/>	6.760e-3	6/5/2009	11:12:43 AM

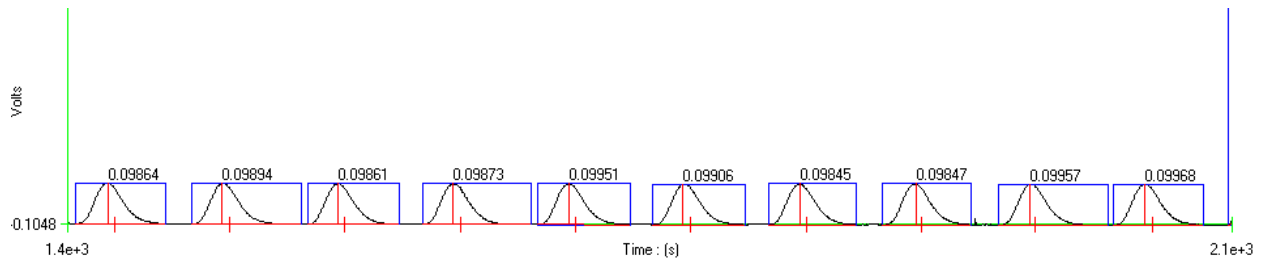
First Order
 Second Order
 Third Order
 Point to Point

Calibration by Area
 Calibration by Height

No Weighting
 1/x weighting
 1/x^2 weighting

Forced through Zero

Results



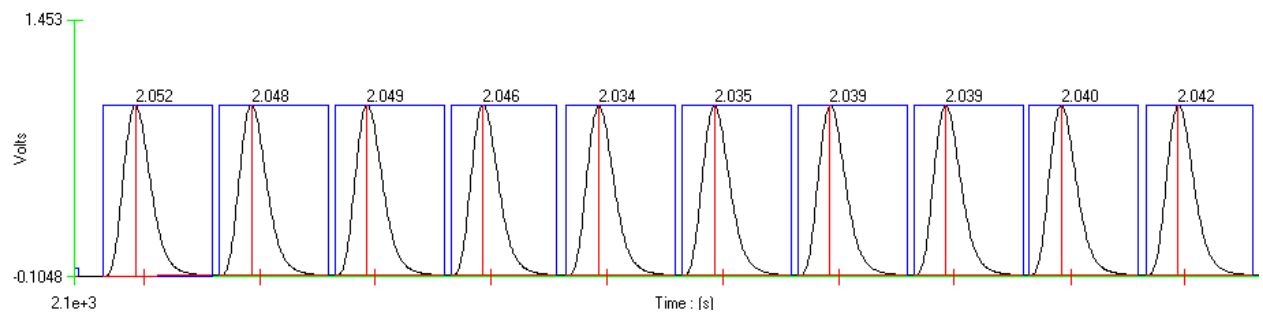
Method Detection Limit using 0.10 mg N/L standard

MDL = 0.002 mg N/L

Standard Deviation (s) = 0.000486 mg N/L, Mean (X) = 0.09895 mg N/L, Known Value = 0.10 mg N/L

Filename: Support Nitrate high range 6 5 2009

ACQ Date: June 5 2009



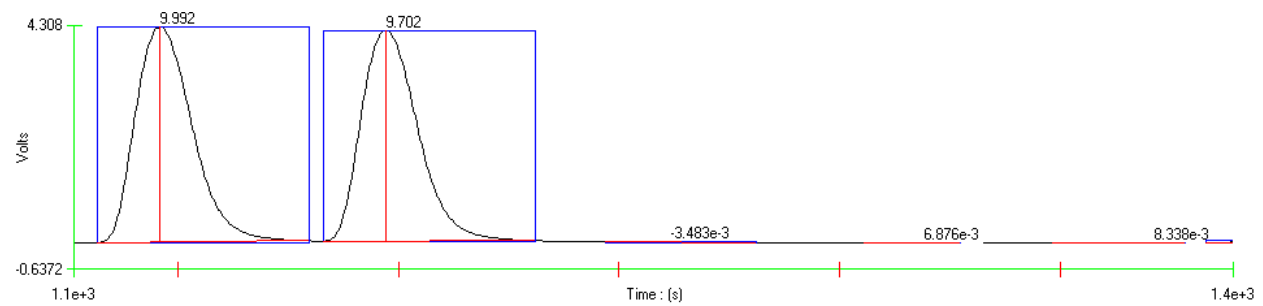
Precision Data using a 2.0 mg N/L standard

%RSD = 0.30

Standard Deviation (s) = 0.0061 mg N/L, Mean (X) = 2.042 mg N/L, Known Value = 2.00 mg N/L

Filename: Support Nitrate high range 6 5 2009

ACQ Date: June 5 2009

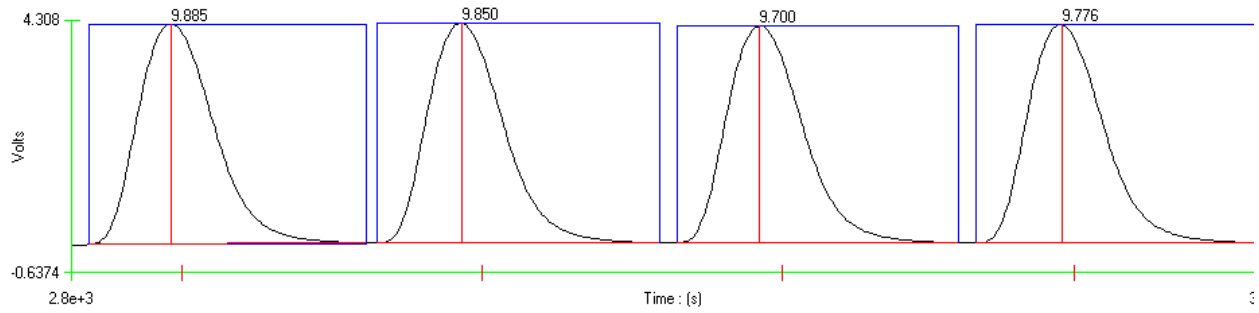


Carryover Study using two 10.0 mg N/L standards followed by three blanks

Carryover passed

Filename: Support Nitrate high range 6 5 2009

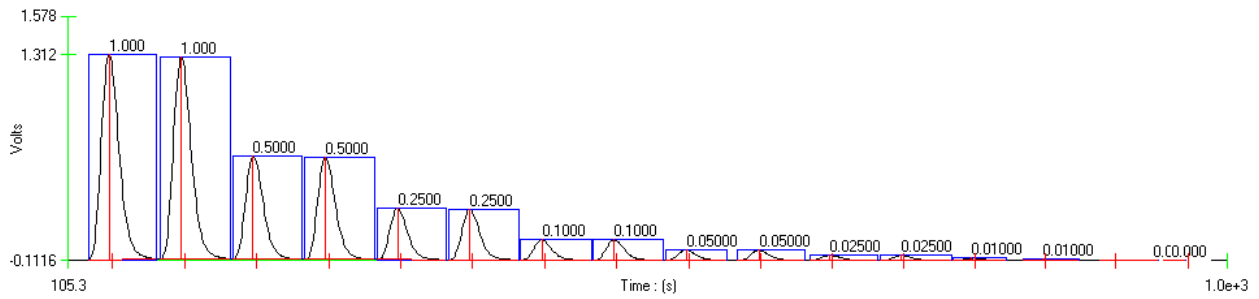
ACQ Date: June 5 2009



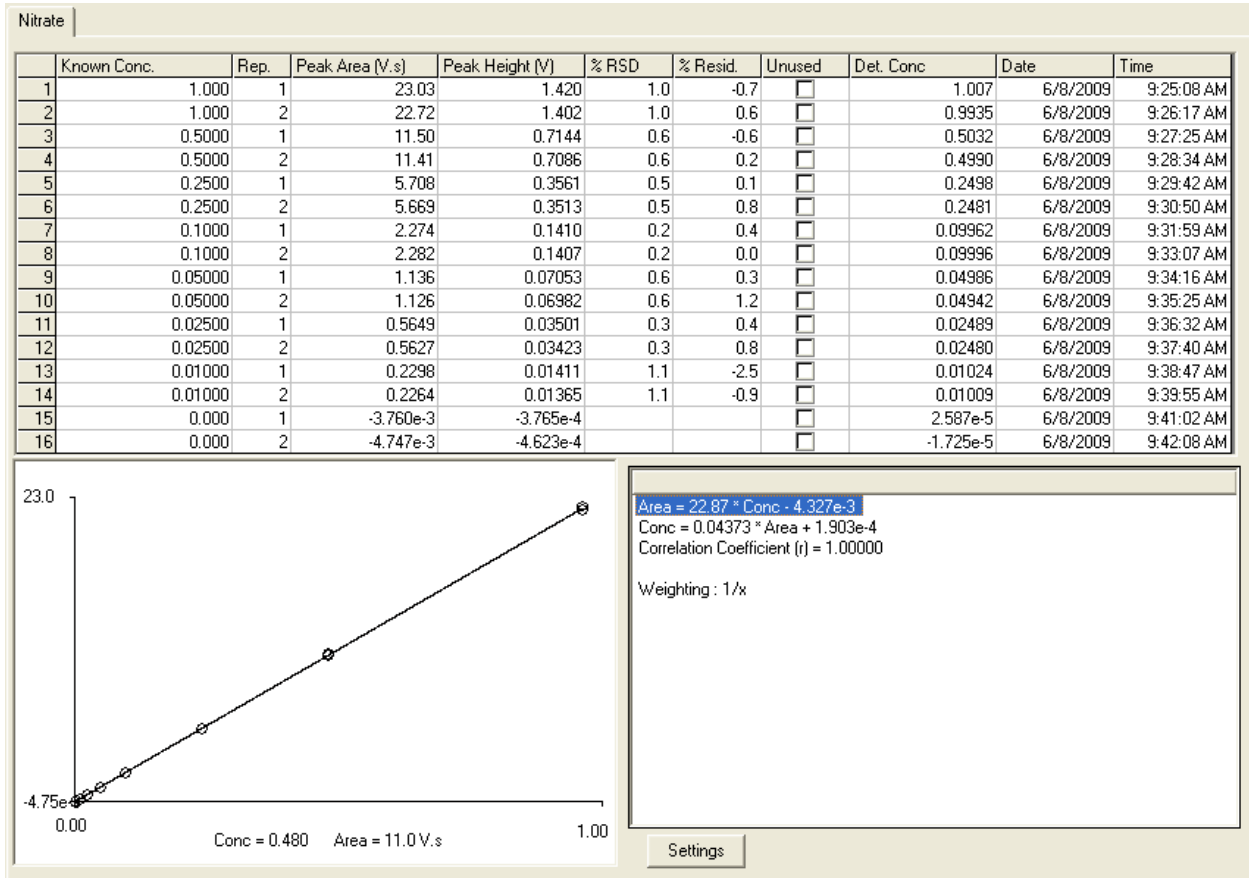
Filename: Support Nitrate high range 6 5 2009
 ACQ Date: June 5 2009

*Column Efficiency 2 injections of a 10 mg N/L as nitrite followed by 2 injections of a 10 mg N/L as nitrate standard.
 % Recovery: 99.6%*

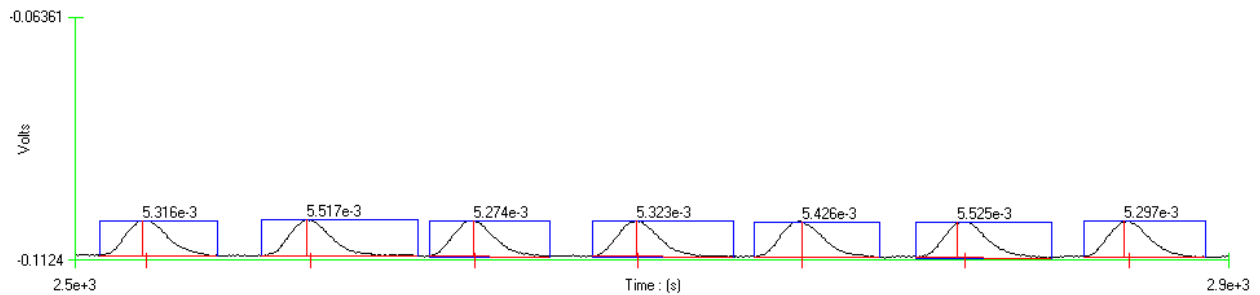
Calibration Data for Nitrate/Nitrite Range 2: 0.01-1.0 mg N/L



Nitrate Calibration Graph and Statistics



Filename: Mid range nitrate 6 8 09 run 1.omn
 Acq Date: 08 June 2009



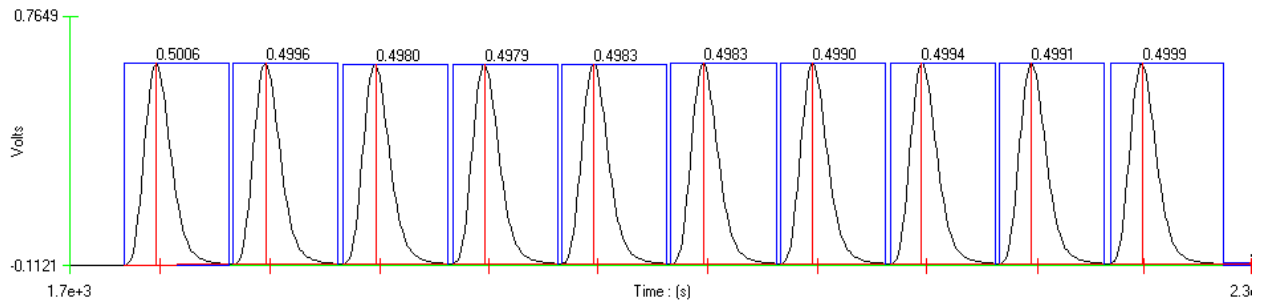
Method Detection Limit using 0.005 mg N/L standard

MDL = 0.001 mg N/L

Standard Deviation (s) = 0.00012 mg N/L, Mean (X) = 0.0053 mg N/L, Known Value = 0.005 mg N/L

Filename: Mid range nitrate 6 8 09 run 1.omn

Acq Date: 08 June 2009



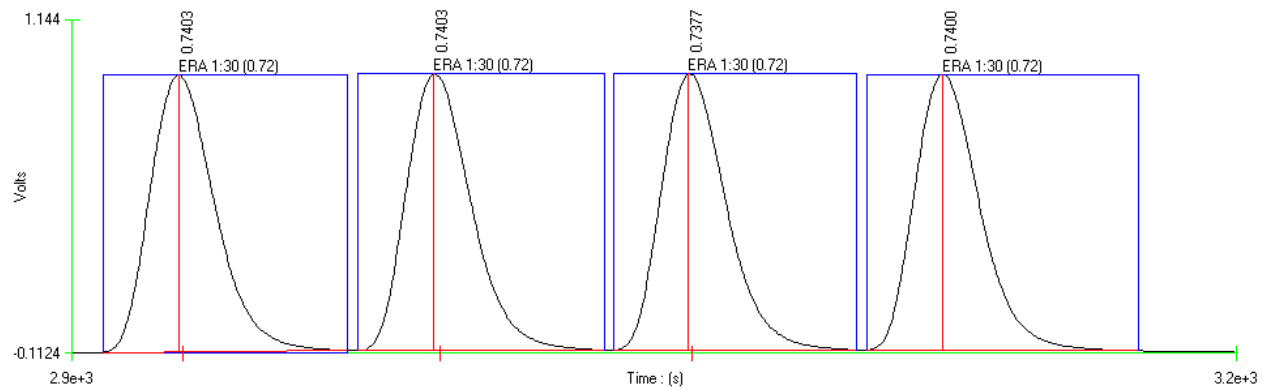
Precision Data using a 0.5 mg N/L standard

%RSD = 0.16

Standard Deviation (s) = 0.00081 mg N/L, Mean (X) = 0.4992 mg N/L, Known Value = 0.5 mg N/L

Filename: Mid range nitrate 6 8 09 run 1.omn

Acq Date: 08 June 2009



External QC Sample, ERA

(Diluted 1:30 to bring into method range).

Known: 0.72 mg N/L

Determined: 0.74 mg N/L

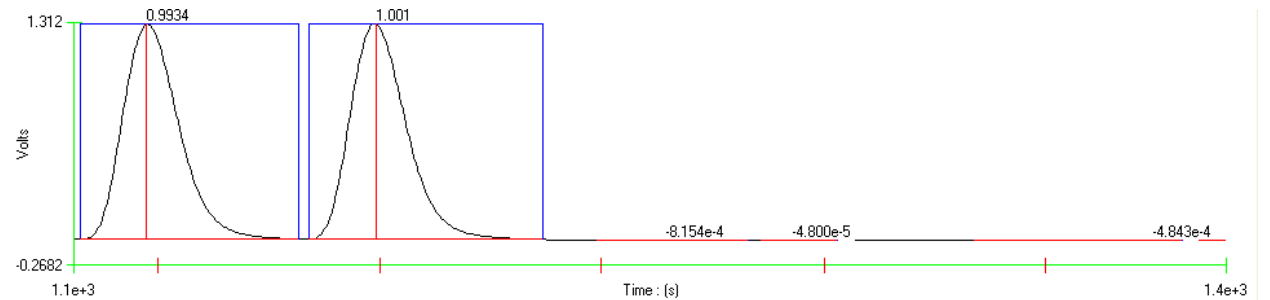
Acceptance Range: 0.63 to 0.81

Simple Nutrients, ERA PM 739

Lot No P1000739A

Filename: Mid range nitrate 6 8 09 run 1.omn

Acq Date: 08 June 2009



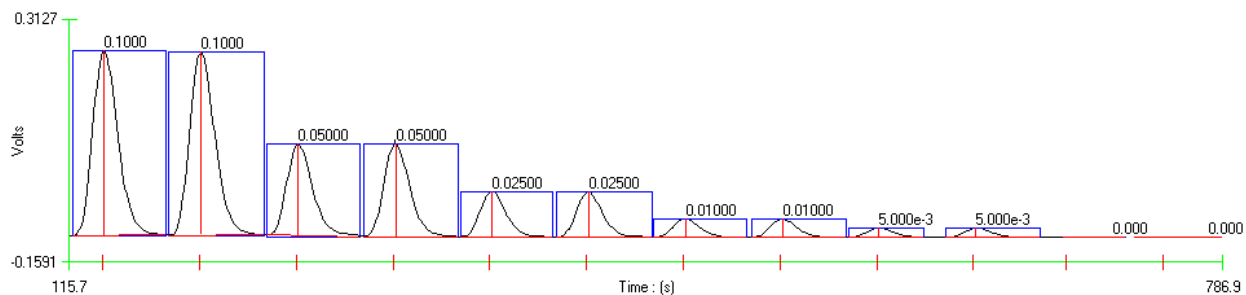
Carryover Study using two 1.0 mg N/L standards followed by three blanks

Carryover passed

Filename: Mid range nitrate 6 8 09 run 1.omn

Acq Date: 08 June 2009

Calibration Data for Nitrate/Nitrite Range 3: 0.1-0.005 mg N/L



Nitrate Calibration Graph and Statistics

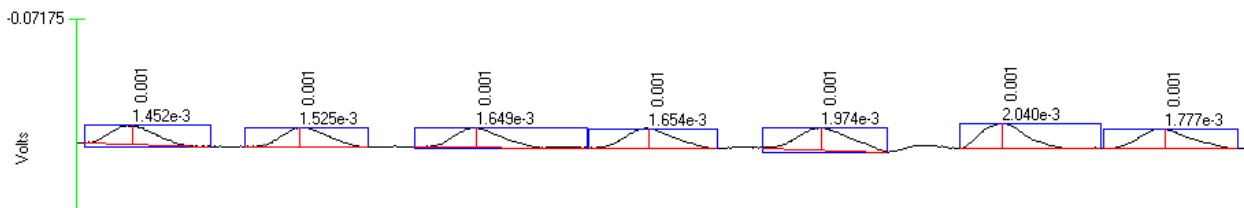
Nitrate

	Known Conc.	Rep.	Peak Area (V.s)	Peak Height (V)	% RSD	% Resid.	Unused	Det. Conc	Date	Time
1	0.1000	1	6.136	0.3605	0.5	0.4	<input type="checkbox"/>	0.09959	6/8/2009	12:00:31 PM
2	0.1000	2	6.090	0.3559	0.5	1.1	<input type="checkbox"/>	0.09885	6/8/2009	12:01:40 PM
3	0.05000	1	3.122	0.1778	0.3	-1.2	<input type="checkbox"/>	0.05061	6/8/2009	12:02:48 PM
4	0.05000	2	3.135	0.1800	0.3	-1.7	<input type="checkbox"/>	0.05082	6/8/2009	12:03:57 PM
5	0.02500	1	1.522	0.08803	0.4	1.6	<input type="checkbox"/>	0.02460	6/8/2009	12:05:05 PM
6	0.02500	2	1.530	0.08781	0.4	1.0	<input type="checkbox"/>	0.02473	6/8/2009	12:06:13 PM
7	0.01000	1	0.6332	0.03580	0.9	-1.6	<input type="checkbox"/>	0.01016	6/8/2009	12:07:22 PM
8	0.01000	2	0.6408	0.03558	0.9	-2.8	<input type="checkbox"/>	0.01028	6/8/2009	12:08:30 PM
9	5.000e-3	1	0.3247	0.01830	0.2	-2.8	<input type="checkbox"/>	5.145e-3	6/8/2009	12:09:39 PM
10	5.000e-3	2	0.3236	0.01812	0.2	-2.5	<input type="checkbox"/>	5.128e-3	6/8/2009	12:10:48 PM
11	0.000	1	-1.723e-3	-6.841e-4			<input type="checkbox"/>	-1.599e-4	6/8/2009	12:11:55 PM
12	0.000	2	-8.168e-3	-5.398e-4			<input type="checkbox"/>	-2.647e-4	6/8/2009	12:13:02 PM

Area = 61.51 * Conc + 8.269e-3
 Conc = 0.01625 * Area - 1.319e-4
 Correlation Coefficient (r) = 0.99995
 Weighting : 1/x

Settings

Filename: 6 8 lo sup run 1.omn
 Acq Date: 08 June 2009



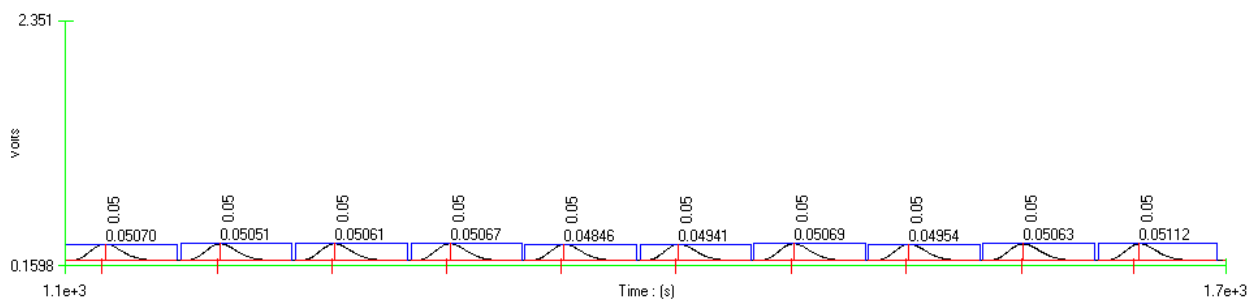
Method Detection Limit using 0.001 mg N/L standard

MDL = 0.0002 mg N/L

Standard Deviation (s) = 0.0007 mg N/L, Mean (X) = 0.00172 mg N/L, Known Value = 0.001 mg N/L

Filename: 6 8 lo sup run 1.omn

Acq Date: 08 June 2009



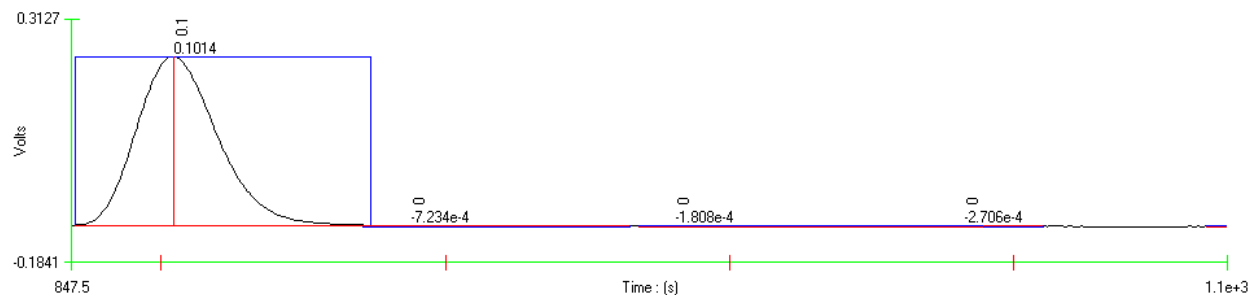
Precision Data using a 0.05 mg N/L standard

%RSD = 1.7%

Standard Deviation (s) = 0.0009 mg N/L, Mean (X) = 0.0502 mg N/L, Known Value = 0.05 mg N/L

Filename: 6 8 lo sup run 1.omn

Acq Date: 08 June 2009



Carryover Study using 0.10 mg N/L standard followed by three blanks

Carryover passed

Filename: 6 8 lo sup run 1.omn

Acq Date: 08 June 2009

Nitrite

QuikChem® Method 80-107-05-1-A

Nitrite in Waters (ULTRA LOW FLOW METHOD)

0.10 to 10.0 mg N/L as NO₂⁻

0.01-1.0 mg N/L as NO₂⁻

– Principle –

Nitrite is 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 540 nm.

The 80- series of methods (Ultra Low Flow) use a pump speed of 10, and so must be run alone or with other ULF methodologies.

– Interferences –

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

