

Data Pack

Methods for the Determination of Cyanide

June 2008

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Introduction

This Data Pack contains application information on Lachat methods for the determination of cyanide. Information on several different method types are included: Offline Digestion / Distillation Methods, MicroDIST Methods, Free Cyanide Methods, In-Line Total Cyanide Methods, In-Line Weak Acid Dissociable (WAD) Cyanide Methods, and Amperometric Detection Methods. The summary table on the following page gives an overview of these methods. More detailed information, including support data, can be found in the remainder of this Data Pack.

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

In the US:

(800) 247-7613

International:

(970) 669-3050

On the Web:

www.lachatinstruments.com

E-mail for Support

support@lachatinstruments.com

E-mail for Sales

sales@lachatinstruments.com

Method Type	Lachat Method No.	Range	Method Detection Limit	Sample Throughput	EPA Approval?	Notes
Total Cyanide, Offline Macro Distillation	10-204-00-1-A	0.005 to 0.500 mg CN/L	0.0005 mg CN/L	90 samples / hr	Yes	0.25 M NaOH matrix. Pyridine / barbituric acid.
	10-204-00-1-D	0.2 to 10.0 mg CN/L	0.01 mg CN/L	60 samples / hr		Acetate buffer, 0.25 M NaOH matrix. Pyridine / dimethyl barbituric acid.
MicroDIST distillation technology	10-204-00-1-X	0.005 to 0.500 mg CN/L	0.001 mg CN/L	80 samples / hr	Yes	0.25 M NaOH matrix. Pyridine / barbituric acid.
	10-204-00-1-X2	0.002 to 0.500 mg CN/L	0.38 ug CN/L	144 samples / hr	Yes	0.25 M NaOH matrix. Pyridine / barbituric acid. High Throughput Method
Free Cyanide	10-204-00-1-F	50 to 500 mg CN/L		80 samples / hr	Equivalent	0.25 M NaOH matrix. Pyridine / barbituric acid.
	10-204-00-1-G	2 to 500 ug CN/L	0.5 ug CN/L	51 samples / hr		0.25 M NaOH matrix. Pyridine free. Thiocyanate interferes nearly quantitatively.
	10-204-00-1-H	LR: 2 to 100 ug CN/L HR: 0.1 to 5.0 mg CN/L	LR: 0.47 ug CN/L HR: 0.0138 mg CN/L	60 samples / hr		
Total Cyanide, In-Line Preparation	10-204-00-2-C	2 to 100 ug CN/L	0.21 ug CN/L	18 samples / hr	Equivalent	Acidic, UV digestion. Selective against thiocyanate. Recovers ferricyanide nearly quantitatively. Settleable particulates not suitable for method.
	10-204-00-2-D	5 to 500 ug CN/L	0.51 ug CN/L	18 samples / hr	Equivalent	
	10-204-00-2-E	2 to 100 ug CN/L	0.50 ug CN/L	16 samples / hr	Equivalent	
	10-204-00-2-G	LR: 2 to 100 ug CN/L HR: 0.1 to 5.0 mg CN/L	LR: 0.16 ug CN/L HR: 0.015 mg CN/L	10 to 13 samples / hr		Pyridine Free, Acidic, UV digestion. Selective against thiocyanate. Recovers ferricyanide nearly quantitatively. Settleable particulates not suitable for method.
Weak Acid Dissociable Cyanide, In-Line Preparation	10-204-00-3-A	10 to 500 ug CN/L	1.0 ug CN/L	21 samples / hr		Pyridine / barbituric acid. Does not recover ferricyanide. Settleable particulates not suitable for method.
	10-204-00-4-B	2 to 100 ug CN/L	0.16 ug CN/L	18 samples / hr		Pyridine free. Does not recover ferricyanide. Settleable particulates not suitable for method. Requires 2 SPM's.
Cyanide determination through Amperometric Detection	10-204-00-5-A	2 to 400 ug CN/L	0.65 ug CN/L	18 samples / hr	Equivalent	Ligand Exchange method. Settleable particulates not suitable for the method. Detector only available in US.
	10-204-00-5-B	2 to 500 ug CN/L	0.234 ug CN/L	12 samples / hr		In-Line Total CN method. Settleable particulates not suitable for the method. Acidic, UV digestion. Selective against thiocyanate. Recovers Ferricyanate nearly quantitatively. Detector only available in US.
	10-204-00-5-X	5 to 400 ug CN/L	0.975 ug CN/L (distilled) 0.20 ug CN/L (nondistilled)	16 samples / hr		MicroDIST method. 0.1 M NaOH trapping solution diluted to 0.025 M during distillation. Detector available only in US.

Off-line Digestion/Distillation Methods

QuikChem® Method 10-204-00-1-A

Determination of Cyanide (Macro Distillation Method) in Waters

0.005 to 0.500 mg CN⁻/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 –

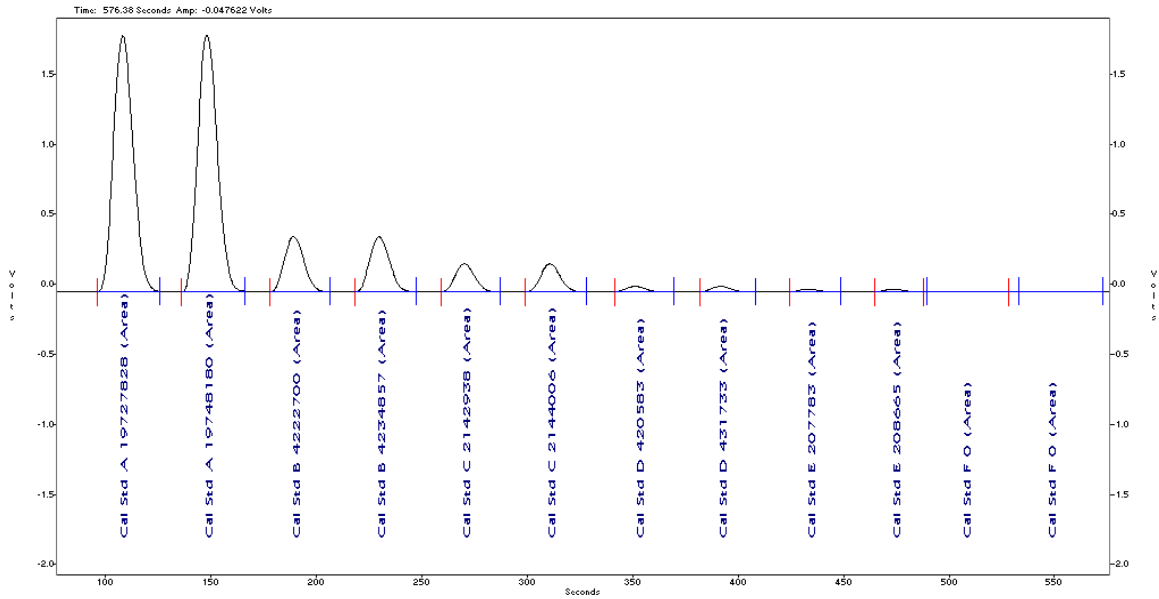
1. For strong acid dissociable and weak acid dissociable cyanide, non-volatile interferences are eliminated or minimized by the distillation procedure.
2. Some of the known interferences are aldehydes, nitrate-nitrite, and oxidizing agents, such as chlorine, thiocyanide, thiosulfate, and sulfide. Multiple 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 see Parts and Price list for Ordering Information

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

17.2. SUPPORT DATA FOR QUIKCHEM 8000

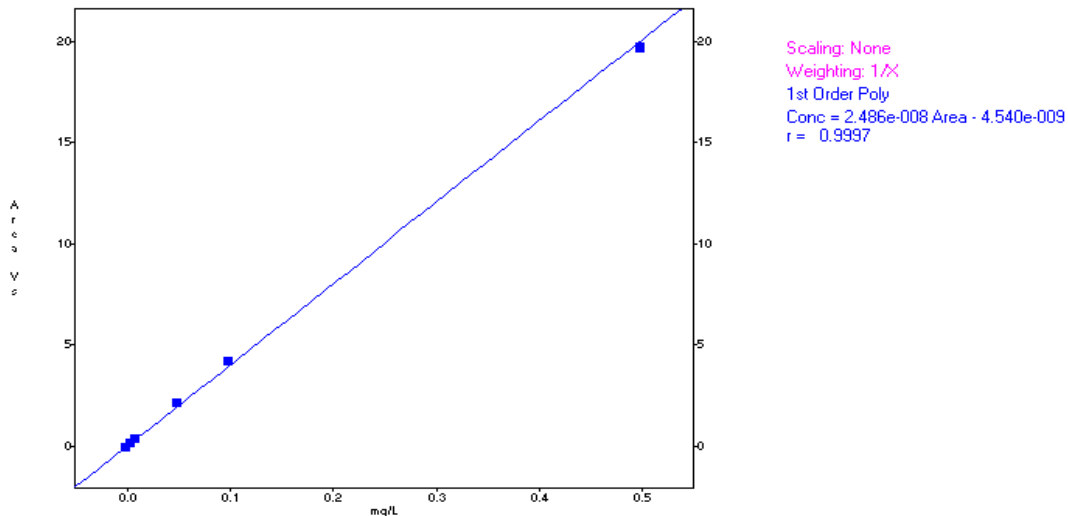


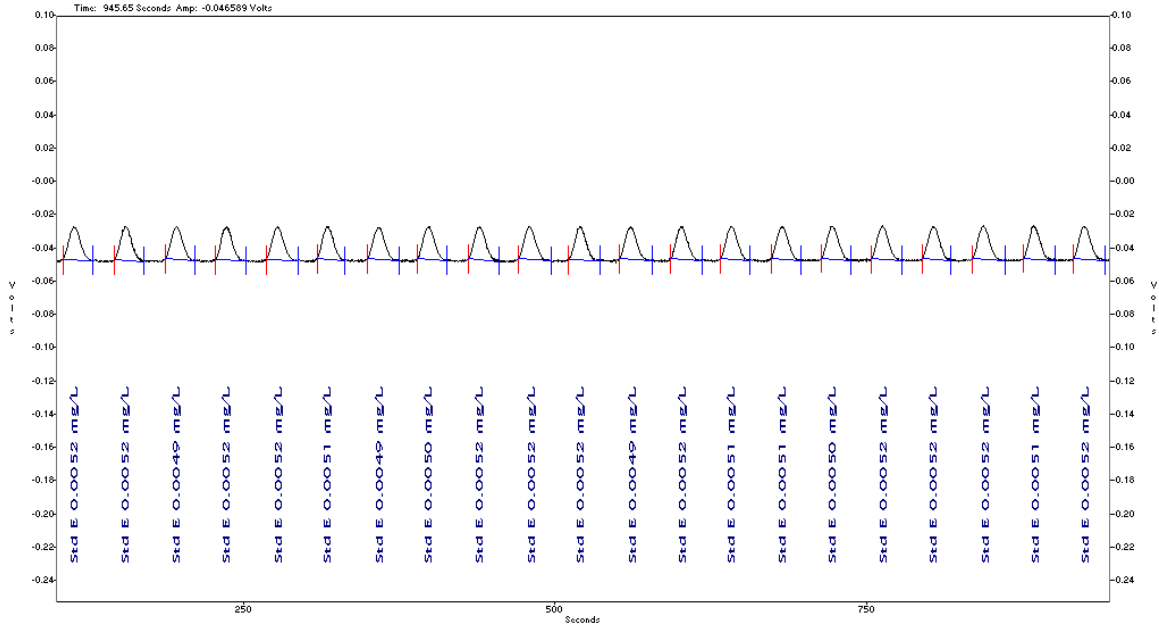
Calibration Data for Cyanide

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	---





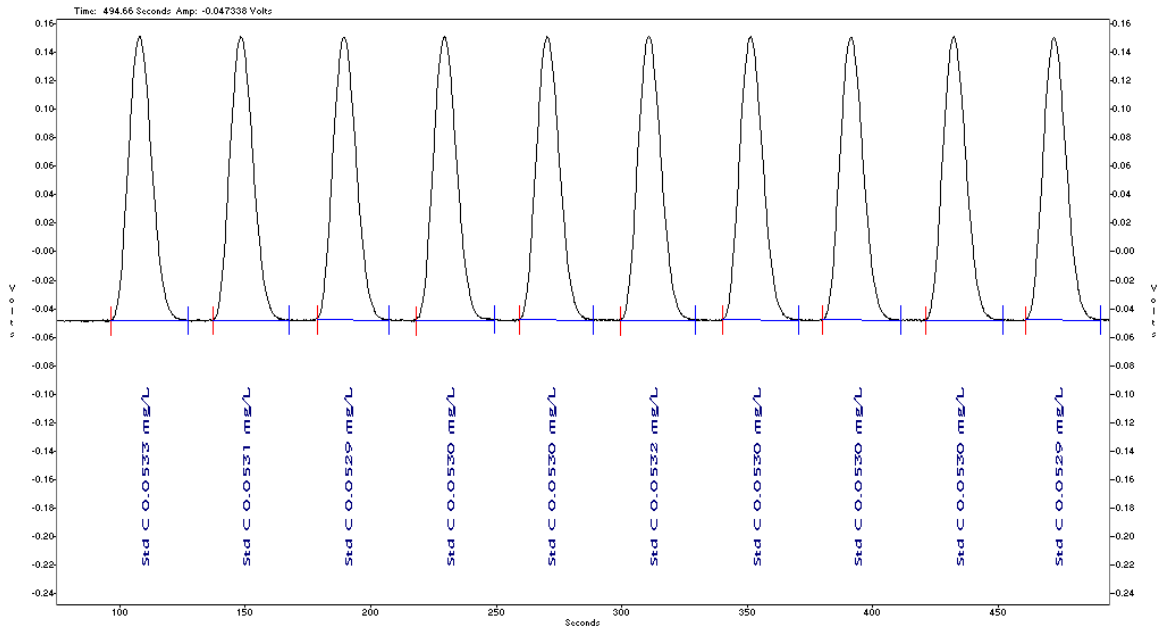
Method Detection Limit claimed for Cyanide using Non Distilled 0.005 mg CN⁻/L Standards

MDL = 0.0005 mg CN⁻/L

Standard Deviation (s) = 0.0001 mg CN⁻/L, Mean (x) = 0.0051 mg CN⁻/L, Known value = 0.005 mg CN⁻/L

File Name: 010920s2.fdt

Acq. Date: 20 September 2001



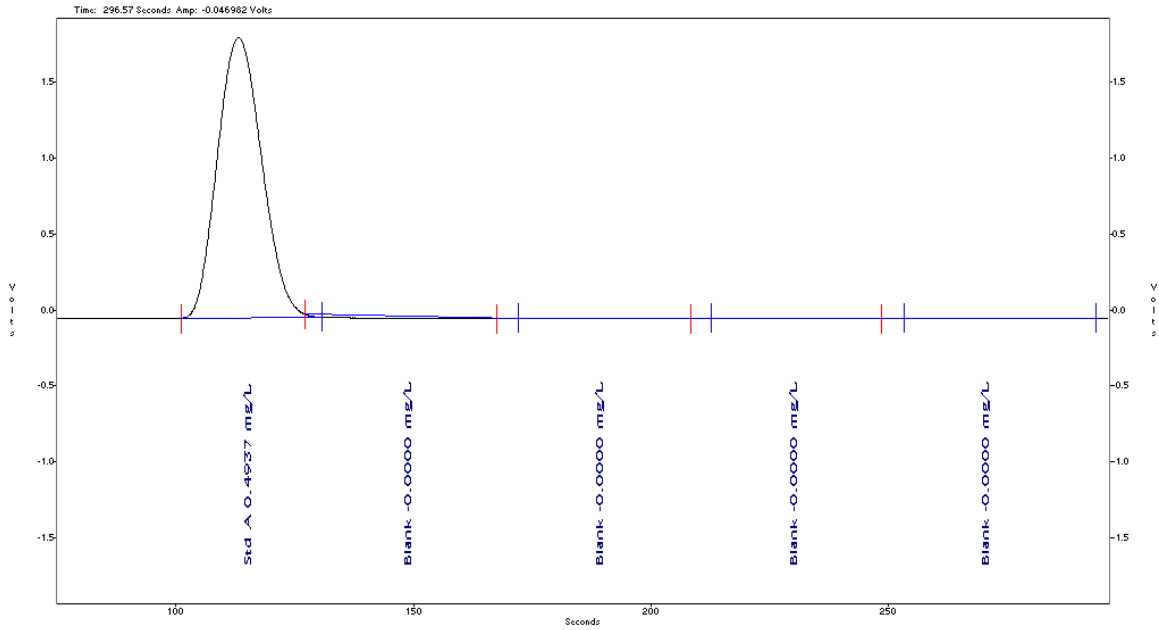
Precision data for cyanide using 0.05 mg CN⁻/L standard

% RSD = 0.255

Standard Deviation (s) = 0.0001 mg CN⁻/L, Mean (x) = 0.053 mg CN⁻/L, Known value = 0.05 mg CN⁻/L

File Name: 010920s1.fdt

Acq. Date: 20 September 2001



Carryover Study: 0.5 mg CN⁻/L standard followed by 4 blanks

Carryover Passed

File Name: 010920s3.fdt

Acq. Date: 20 September 2001

Methods available for Total Cyanide, Off-line Macro Distillation/digestion			
Method Number	Method Type	Range	Comments
10-204-00-1-A[#]	Total Cyanide after Distillation	0.005-0.5 mg CN ⁻ /L	0.25 M NaOH matrix. Pyridine/barbituric acid
10-204-00-1-D	Total Cyanide after Distillation	0.2-10.0 mg CN ⁻ /L	Acetate buffer, 0.25 M NaOH matrix. Pyridine/dimethyl barbituric acid

MicroDist™ Methods

QuikChem® Method 10-204-00-1-X

(See also Amperometric Methods)

Cyanide in drinking and wastewaters using MICRO DIST

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.

– Special Apparatus –

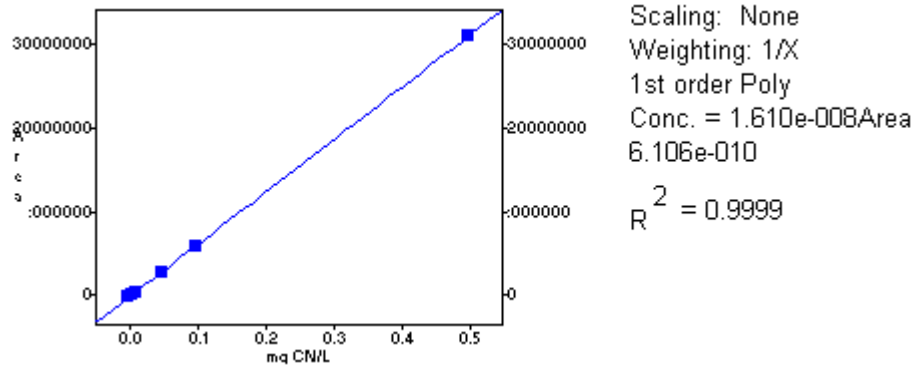
Please see Parts and Price list for Ordering Information

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

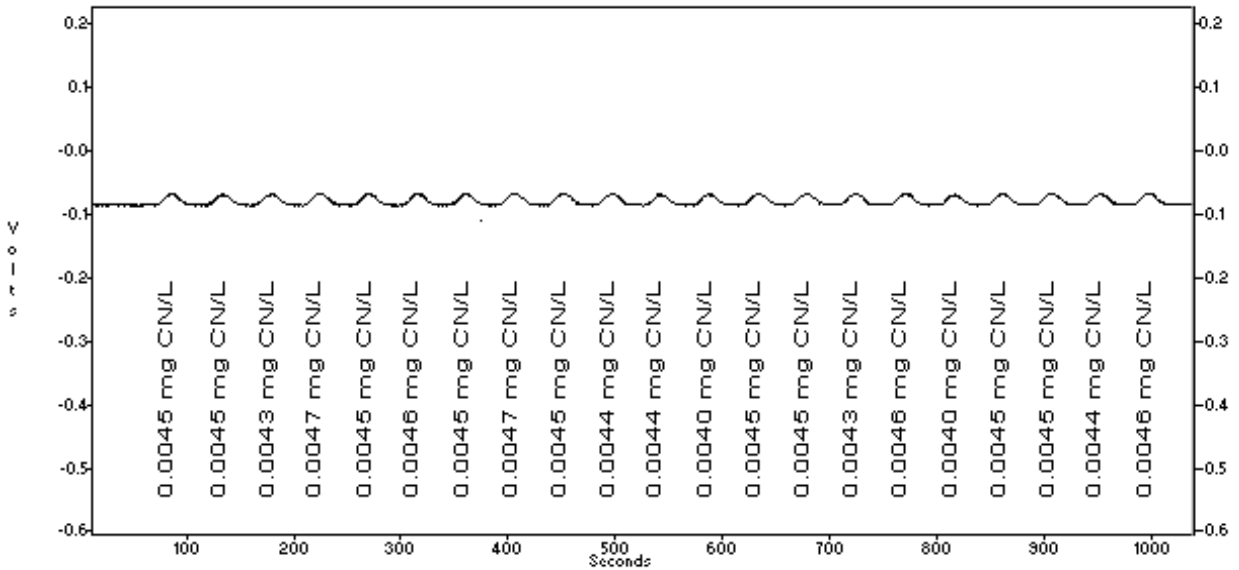
17.4. Support Data for the QC8000

Calibration Graph and Statistics

Level	Area	mg CN ⁻ /L	Determined	Rep 1	Rep 2	Replicate RSD	Replicate %RSD	% residual
1	31230856	0.5	0.503	3120856	31241620	6214.6	0.0	-0.6
2	615006	0.1	0.099	6150060	6158613	4937.8	0.1	1.0
3	295031	0.05	0.0475	2950321	2972790	12972.8	0.4	5.0
4	636723	0.01	0.0103	636723	659544	13175.8	2.0	-2.5
5	333965	0.005	0.0054	333965	356110	12784.4	3.7	-7.5
6	0	0	0	0	0	0.0	0.0	---



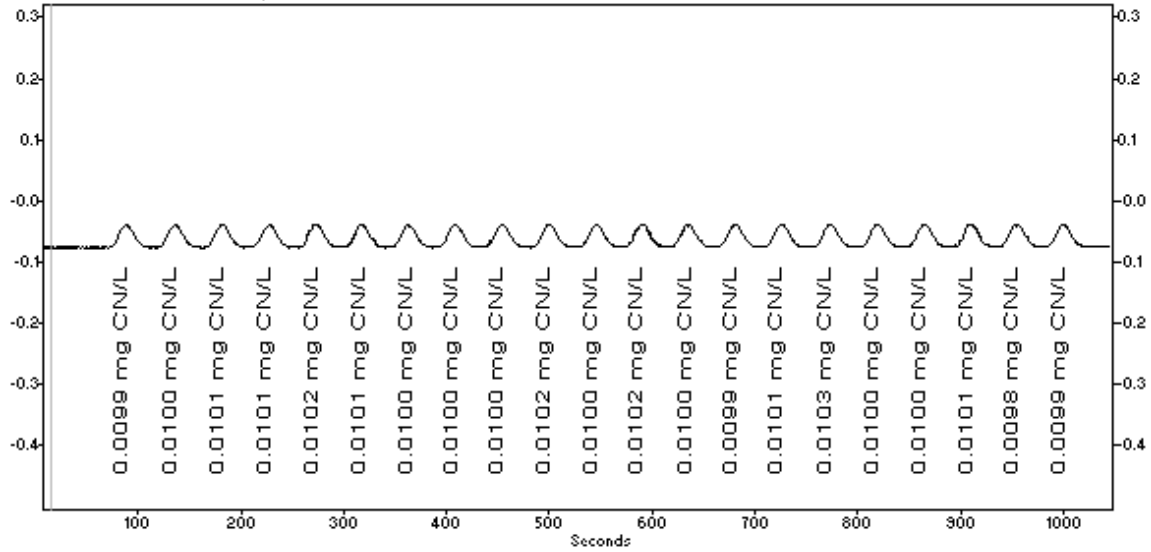
Method Detection Limit using Distilled Standards



MDL = 0.00061 mg CN/L

ACQ. TIME: Sep 26, 1994 16:37:13
DATA FILENAME: C:\OMNION\DATA\1020401A\092694M2.FDT
METHOD FILENAME: C:\OMNION\METHODS\1020401A\1020401A.met

Method Detection Limit for Cyanide using Non Distilled Standards



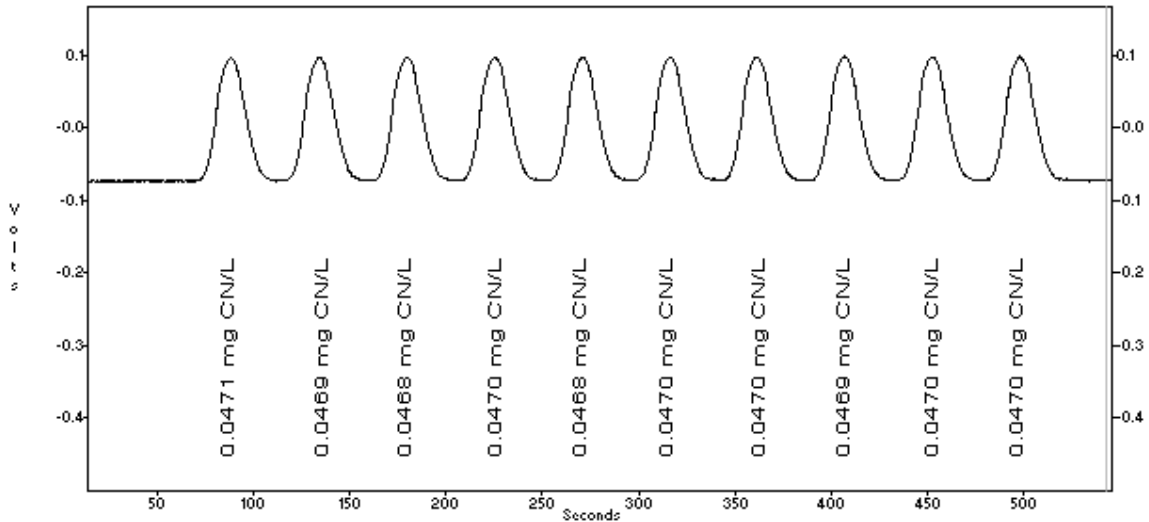
Method Detection Limit for cyanide using 0.01 mg/L standard

MDL= 0.0003 mg/L

Standard Deviation (s) = 0.0001 mg/L, Mean (x) = 0.010 mg/L, Known value = 0.01 mg/L

File Name:092194M1.fdt

Acq. Date: 21 September 1994



Precision data for Cyanide using 0.05 mg/L standard

% RSD = 0.21 %

Standard Deviation (s) = 9.72e-005 mg/L, Mean (x) = 0.0470 mg/L, Known value = 0.05 mg/L

File Name:092194P2.fdt

Acq. Date: 21 September 1994

Available Micro DistTM Methods	(See also Amperometric methods)		
Method Number	Method Type	Range	Comments
10-204-00-1-X[#]	Micro Dist TM	0.005-0.500 mg CN/L	0.25 M NaOH matrix. Pyridine/barbituric acid
10-204-00-1-X(2)[#]	Micro Dist TM	0.002-0.500 mg CN/L	0.25 M NaOH matrix. Pyridine/barbituric acid. High Throughput Method (about 125 samples per hour)

Free Cyanide

QuikChem® Method 10-204-00-1-G

Cyanide in Waters

2.0 to 500 µg CN/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 solutions. The cyanide ion in the absorbing solution is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at pH less than 8. The CNCl then forms a violet-blue dye by reacting with an isonicotinic acid-barbituric acid reagent. The color is read at 600 nm.

– 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 4500 – CN⁻ B., referenced in Section 16 for details of preliminary sample treatment to remove volatile interferences.

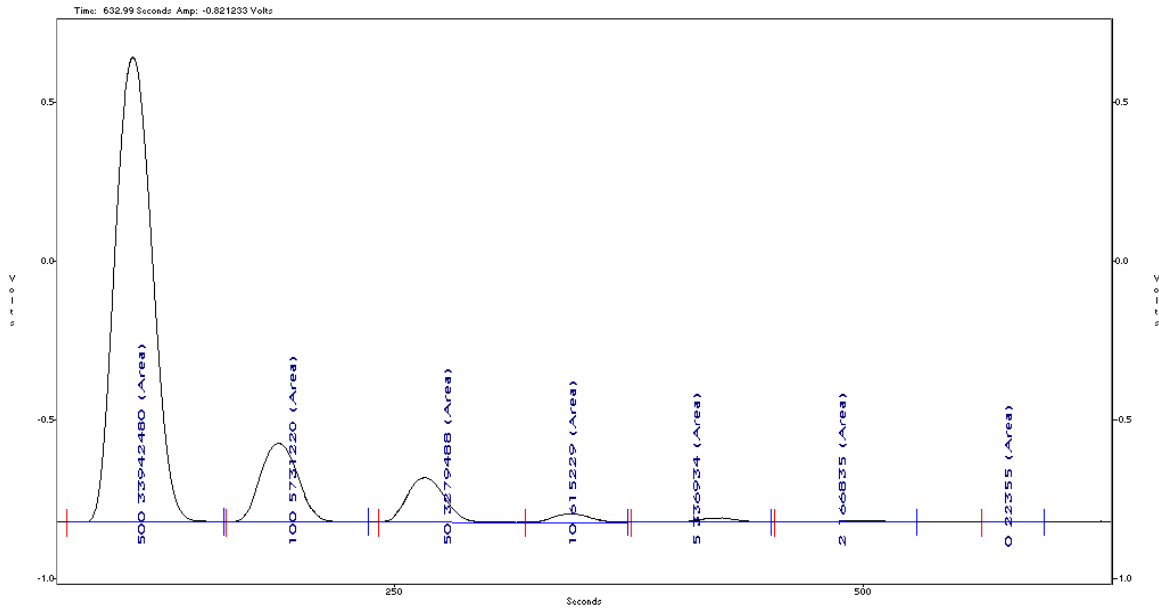
– Special Apparatus –

Please see Parts and Price list for Ordering Information

1. Heating Unit
2. Macro Distillation setup

SUPPORT DATA FOR QUIKCHEM 8000

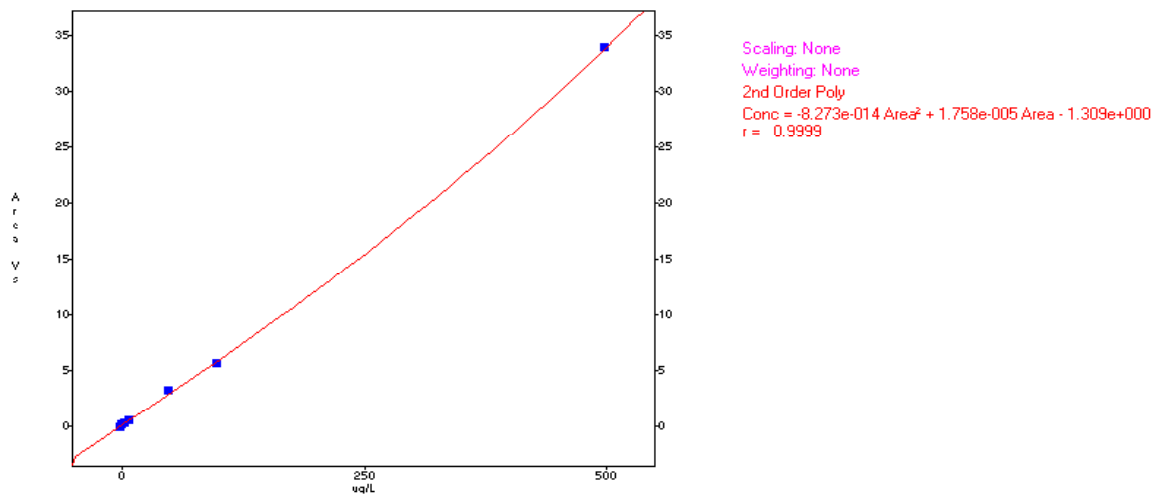
Calibration Data for Cyanide



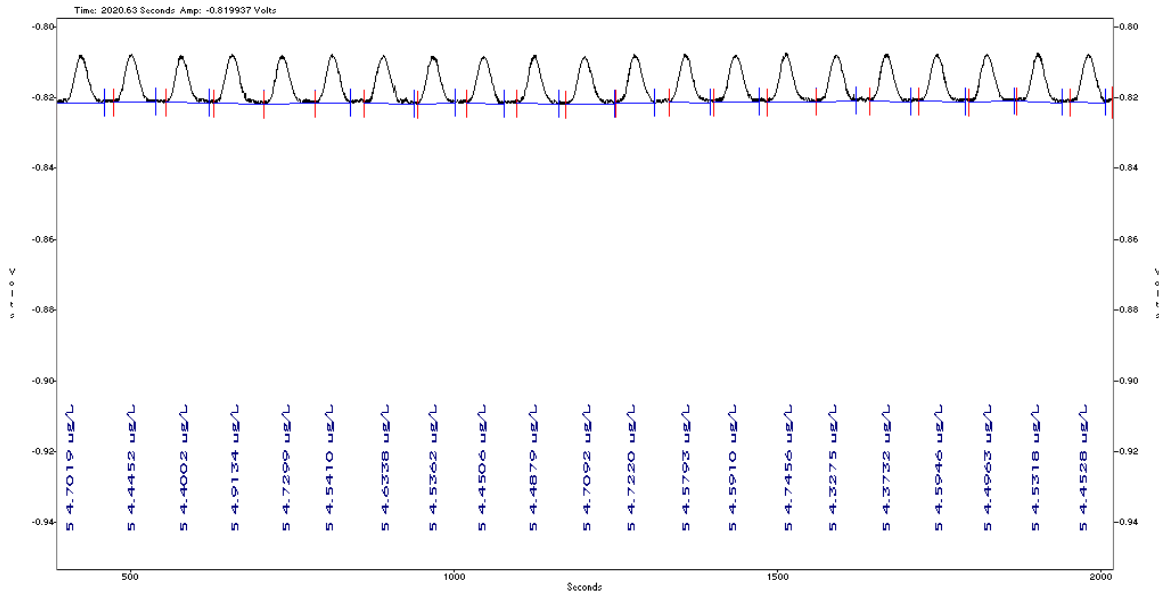
Data File Name: 000711c1.fdt
ACQ Date: 11 July 2000

Calibration Graph and Statistics

Level	Area	µg CN ⁻ /L	Determined	% residual
1	33942480	500	500	0
2	5731220	100	96.7	3.3
3	3279488	50.0	44.6	-10.9
4	615229	10.0	9.5	5.3
5	336934	5.0	4.6	7.9
6	166835	2.0	1.6	18.9
7	22355	0.0	---	---



Method Detection Limit for Cyanide using Non Distilled Standards



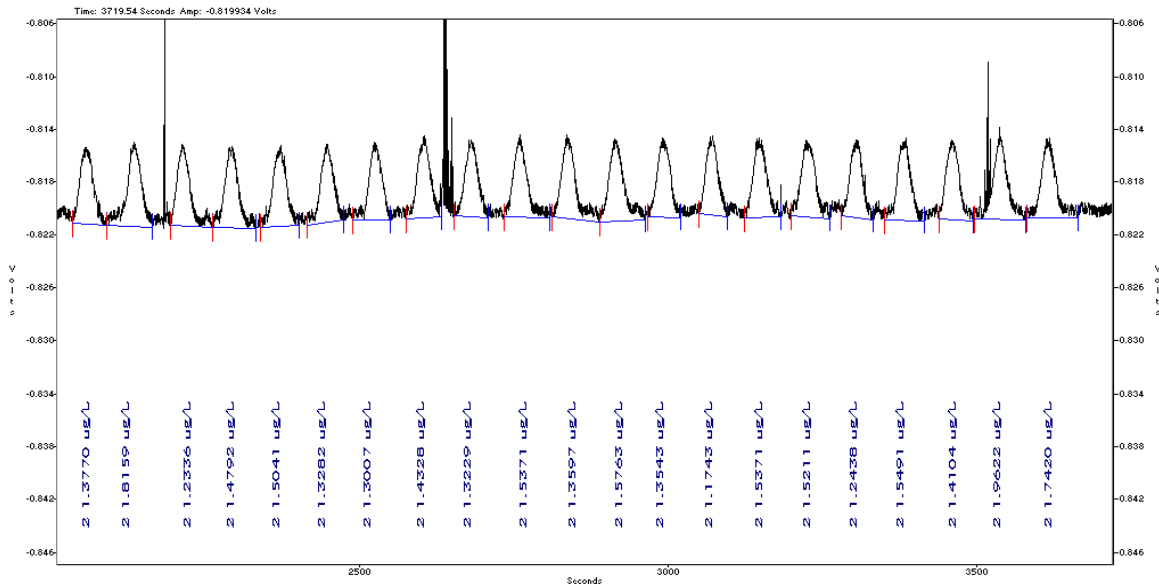
Method Detection Limit for cyanide using a 5.0 µg CN⁻/L Standard

MDL = 0.37 µg CN⁻/L

Standard Deviation (s) = 0.15 µg CN⁻/L, Mean (X) = 4.57 µg CN⁻/L, Known Value = 5.0 µg CN⁻/L

Data File Name: 000711mc.fdt

ACQ. Date: 11 July 2000



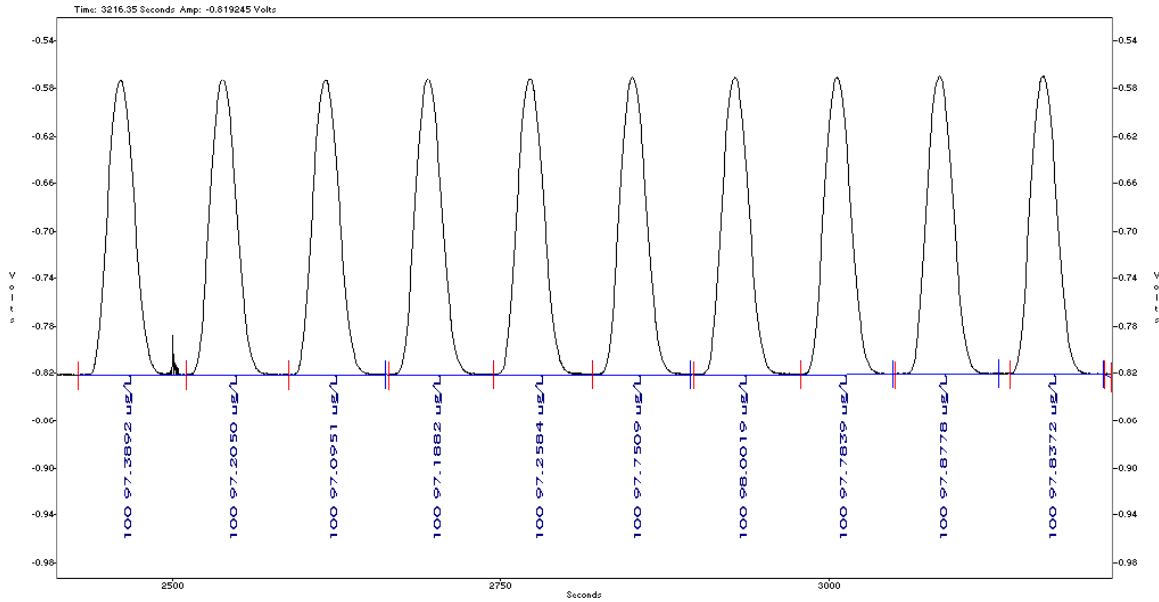
Method Detection Limit for cyanide using a 2 µg CN⁻/L Standard

MDL = 0.50 µg CN⁻/L

Standard Deviation (s) = 0.20 µg CN⁻/L, Mean (X) = 1.46 µg CN⁻/L, Known Value = 2 µg CN⁻/L

Data File Name: 000711mc.fdt

ACQ. Date: 11 July 2000



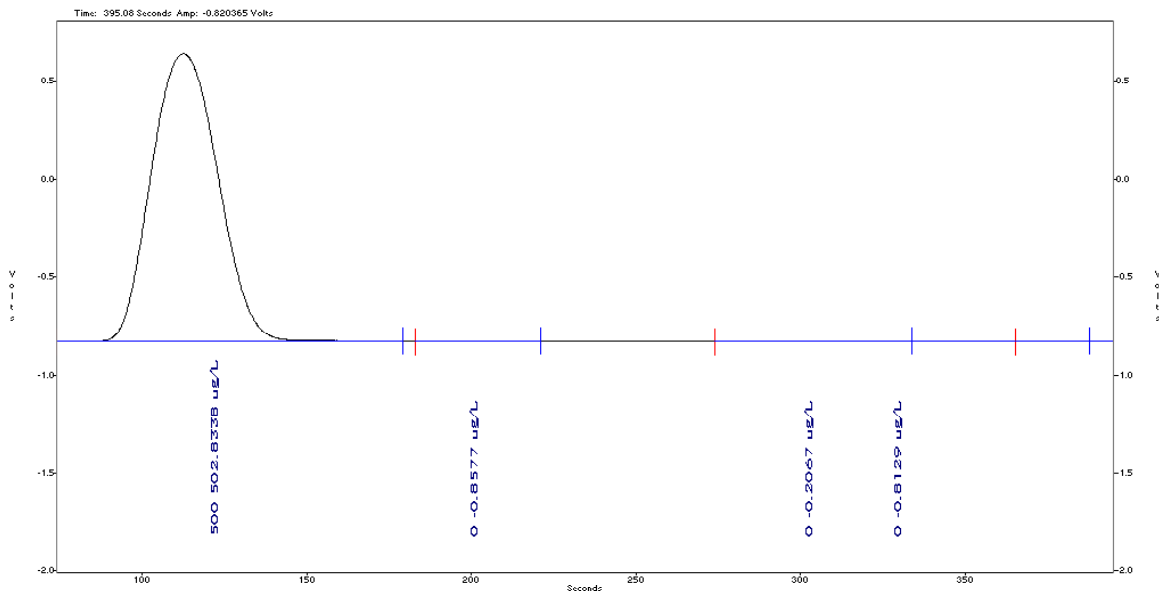
Precision data for cyanide, using a 100 µg CN/L Standard

%RSD = 0.35

Standard Deviation (s) = 0.34 µg CN⁻/L, Mean (X) = 97.54 µg CN⁻/L, Known Value = 100 µg CN/L

Data File Name: 000711cs.fdt

ACQ. Date: 11 July 2000



Carryover Study: 500 µg CN/L Standard followed by 3 blanks

Carryover passed

Data File Name: 000711mc.fdt

ACQ Date 11 July 2000

Available Methods for Free CN			
Method Number	Method Type	Range	Comments
10-204-00-1-F	Free Cyanide	50-500 mg CN ⁻ /L	0.25M NaOH matrix. Pyridine/barbituric acid
10-204-00-1-G	Free Cyanide	2-500 µg CN ⁻ /L	0.025 M NaOH matrix. Pyridine free. Thiocyanate interferes nearly quantitatively.
10-204-00-1-H	Free Cyanide	2-100 µg CN ⁻ /L; 0.1-5.0 mg CN ⁻ /L	0.025 M NaOH matrix. Pyridine free. Thiocyanate interferes nearly quantitatively.

In-Line Total Cyanide

(See also Amperometric methods)
QuikChem® Method 10-204-00-2-C

Total Cyanide in Waters

(Low Flow Method)
2 to 100 µg CN⁻/L
(MDL = 0.21 µg CN⁻/L)

– Principle –

Using in-line cyanide distillation, a liquid sample is first mixed with phosphoric acid, heated to 140°C, and then UV irradiated to break down metal-CN⁻ complexes. The HCN_(g) from the sample matrix passes through a Teflon membrane and is trapped into a sodium hydroxide solution. The CN⁻ is determined colorimetrically (pyridine-barbituric acid chemistry) at 570 nm. Both standards and samples are distilled in-line. The distillation and gas diffusion steps are similar to those described in ISO method, ISO/TC147/SC2-N-Water Quality-Determination of total cyanide and free cyanide by flow analysis.

– Interferences –

1. Thiocyanate gives a positive response of less than 1% with this method. A 100 µg CN⁻/L as SCN⁻ sample gave an average response of 0.03 µg CN⁻/L.

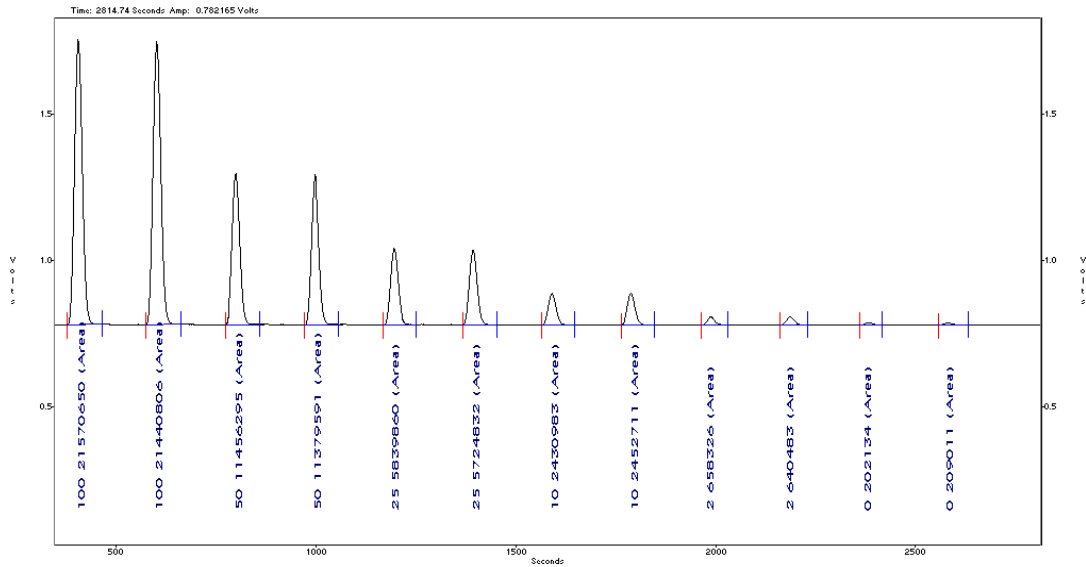
– Special Apparatus –

Please see Parts and Price list for Ordering Information

1. In-Line Sample Preparation Module (Lachat Part No. A30113 [110V], A30213 [220V]).
2. The colorimetric determination step requires a standard heating unit.
3. 60 position racks for samples are required to allow replicate sample analyses from a single tube. XYZ with 60 Position rack (Lachat Part No. A81122 [110V]/A81222 [220V]); RAS (Lachat Part No. A81136 [110V]/A81236 [220V])
4. PVC PUMP TUBES MUST BE USED WITH THIS METHOD.

SUPPORT DATA FOR QUIKCHEM 8000

Calibration Data for Cyanide

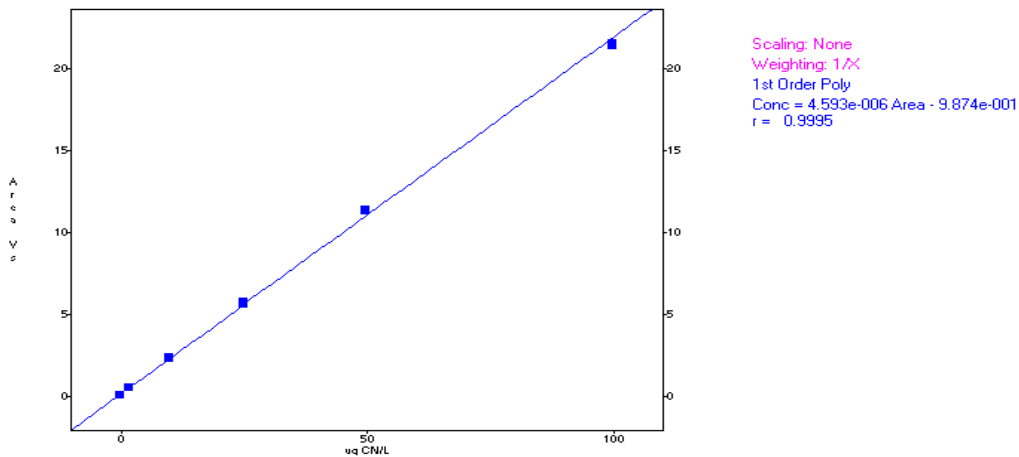


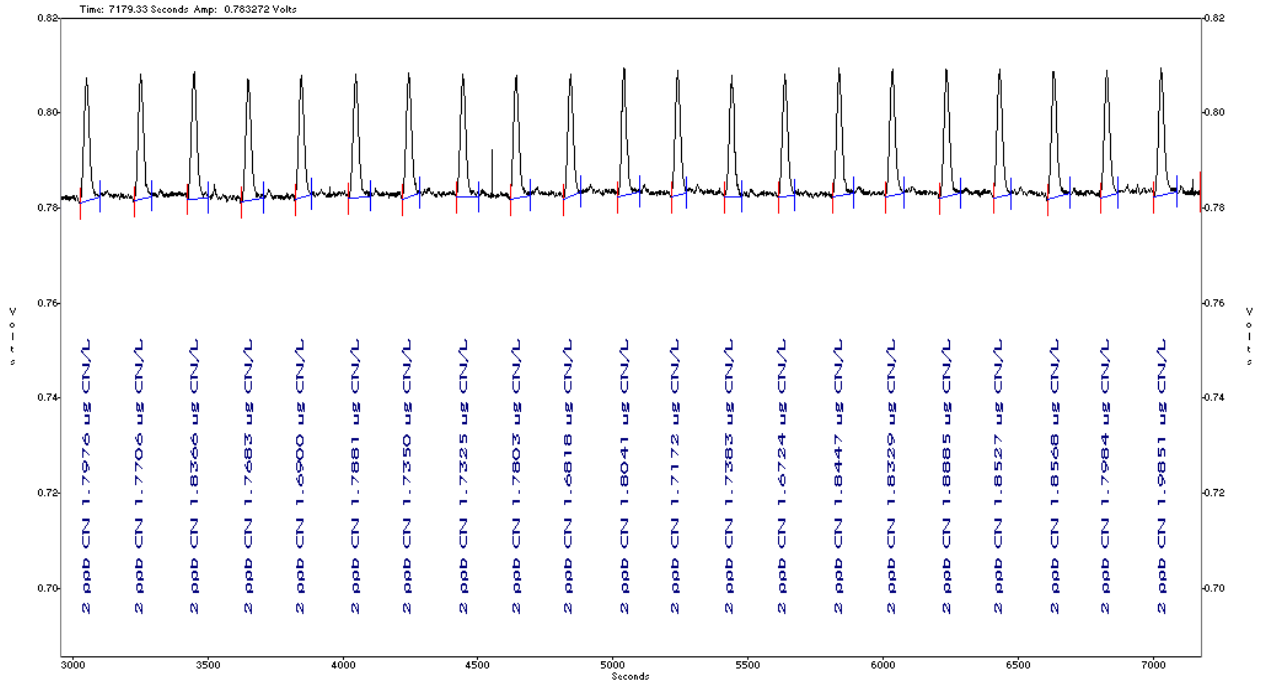
File Name: Test68.fdt

Acq. Date: 24 August 2000

Calibration Graph and Statistics

Level	Area	µg CN/L	Determined	Replicate %RSD	% residual
1	21505728	100	97.8	0.4	2.2
2	11417943	50	48.55	0.5	-2.9
3	5782346	25	24.43	1.4	-2.3
4	2441847	10	9.77	0.6	-2.3
5	649405	2	1.99	1.9	0.2
6	205573	0	---	2.4	---





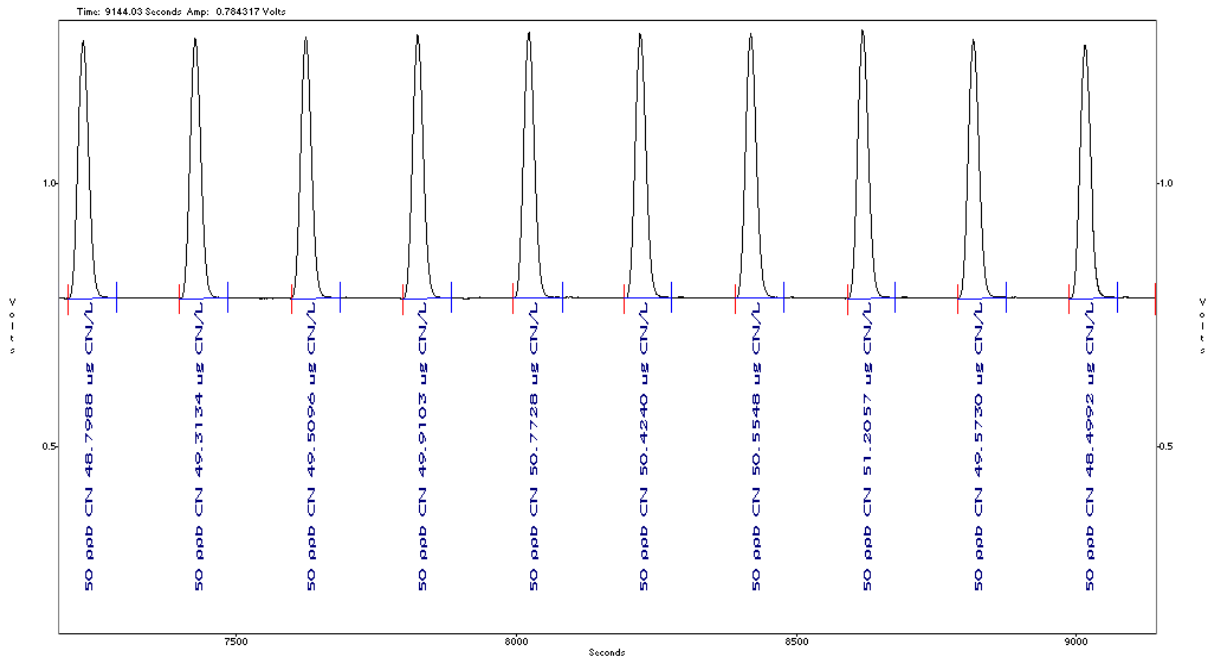
Method Detection Limit for Cyanide using the 2 µg CN/L standard

MDL = 0.21 µg CN/L

Standard Deviation (s) = 0.0826 µg CN/L, Mean (x) = 1.832 µg CN/L, Known value = 2.0 µg CN/L

File Name: Test68.fdt

Acq. Date: 24 August 2000



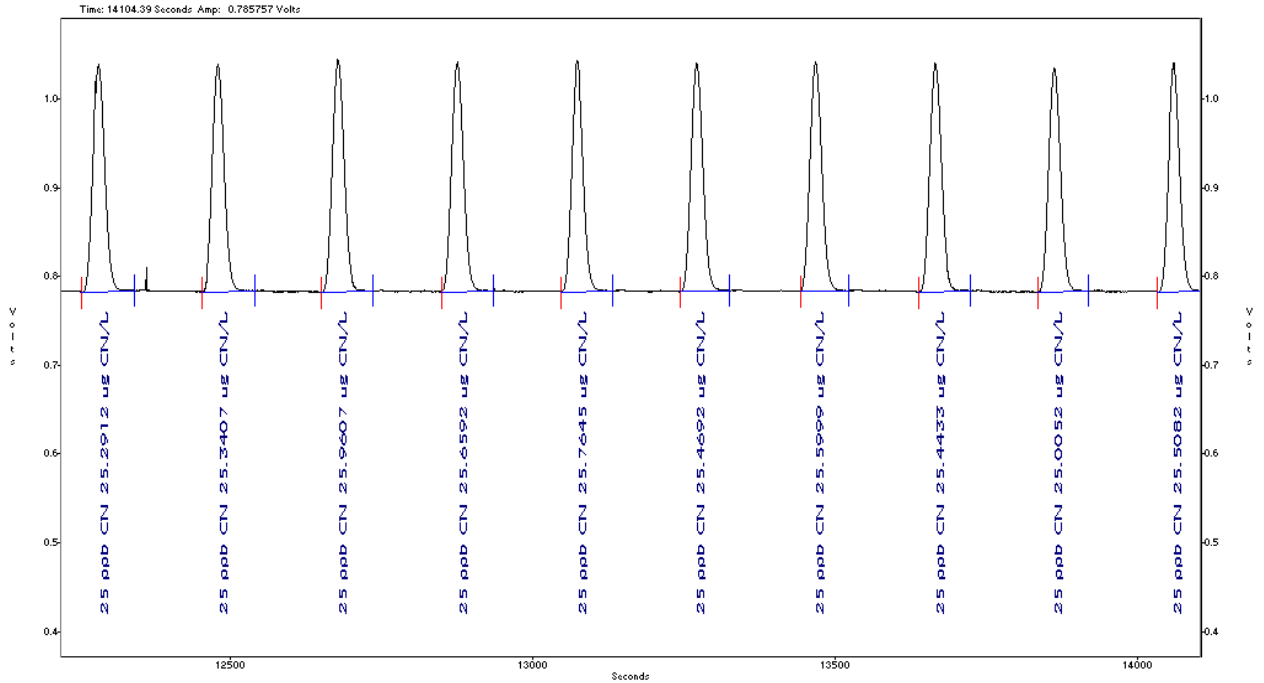
Precision data for cyanide using 50 µg CN/L standard

% RSD = 1.77

Standard Deviation (s) = 0.876 µg CN/L, Mean (x) = 49.846 µg CN/L, Known value = 50 µg CN/L

File Name: Test68.fdt

Acq. Date: 24 August 2000



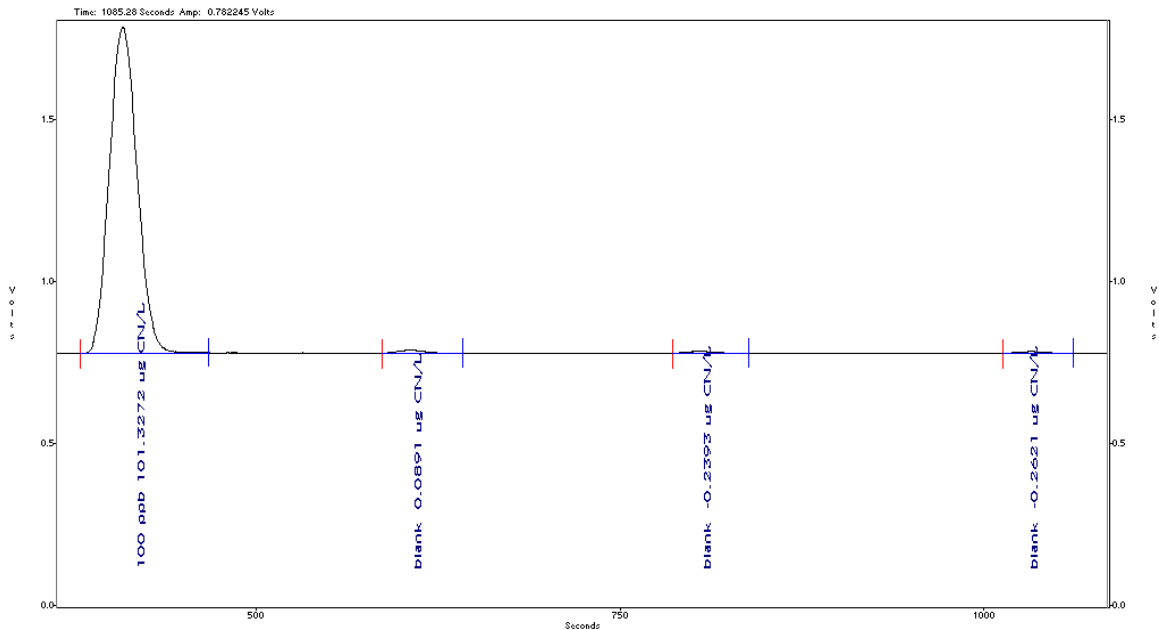
Precision data for cyanide using 25 µg CN⁻/L standard

% RSD = 1.06

Standard Deviation (s) = 0.267 µg CN⁻/L, Mean (x) = 25.529 µg CN⁻/L, Known value = 25 µg CN⁻/L

File Name: Test68.fdt

Acq. Date: 24 August 2000



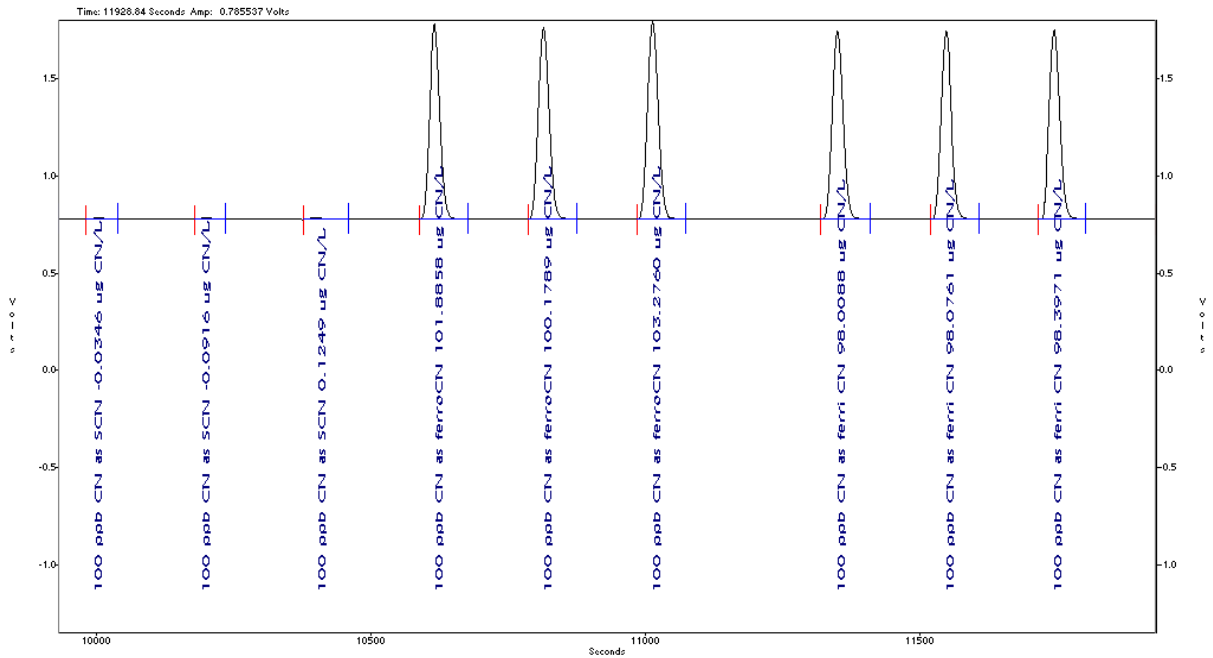
Carryover Study: 100 µg CN⁻/L standard followed by 3 blanks

Carryover Passed

File Name: ILCNCRY.fdt

Acq. Date: 24 August 2000

Recovery of Ferri-, Ferro- and Thio Cyanates



File name Test68.fdt
Acq Date 24 August 2000

Compound	Amount in Sample	Value Obtained	%Recovery*
Ferro-cyanide	100 µg	101.69	101.69
Ferri-cyanide	100 µg	98.07	98.07
Thiocyanate	100 µg	0.03	0.03

* (determined/known) * 100

Conclusion: While Ferri- and Ferro- cyanide are recovered at a level greater than 95%, thiocyanate is not a significant interferent in this method.

Available Methods for In-line Total CN			
Method Number	Method Type	Range	Comments
10-204-00-2-C[^]	In-line Total CN ⁻	2-100 µg CN ⁻ /L	Acidic, UV digestion. Selective against thiocyanate. Recovers ferri-cyanide nearly quantitatively. Settleable particulates not suitable for the method. Industrial wastes need review prior to purchase.
10-204-00-2-D[^]	In-line Total CN ⁻	5-500 µg CN ⁻ /L	Acidic, UV digestion. Selective against thiocyanate. Recovers ferri-cyanide nearly quantitatively. Settleable particulates not suitable for the method. Industrial wastes need review prior to purchase.
10-204-00-2-E[^]	In-line Total CN ⁻	2-100 µg CN ⁻ /L	Acidic, UV digestion. Selective against thiocyanate. Lower recovery of ferri-cyanide. Settleable particulates not suitable for the method. Industrial wastes need review prior to purchase.
10-204-00-2-G	In-line Total CN ⁻	2-100 µg CN ⁻ /L; 0.1-5.0 mg CN ⁻ /L	Pyridine Free. Acidic, UV digestion. Selective against thiocyanate. Recovers ferricyanide nearly quantitatively. Settleable particulates not suitable for the method. Industrial wastes need review prior to purchase.

In-Line Weak Acid Dissociable Cyanide

QuikChem® Method 10-204-00-3-A

Weak Acid Dissociable (WAD) Cyanide in Waters

10.0 to 500 µg CN⁻/L

– Principle –

Using the In-Line Sample Preparation Module, a liquid sample is mixed with a KHP and zinc sulfate buffer at 115°C. The HCN gas from the sample matrix crosses a gas permeable membrane where it is trapped by a sodium hydroxide solution. The CN⁻ is then determined colorimetrically using the pyridine-barbituric acid chemistry at 570 nm. Both standards and samples are distilled in-line.

– Interferences –

1. Thiocyanate up to 50 mg/L and sulfide up to 10 mg/L do not interfere in the determination of cyanide at 0.1 mg/L.
2. The signal obtained for a cyanide standard made in 0.025 M NaOH is equivalent to that made in 0.1 M NaOH. However, the signal for a standard made in 0.25 M NaOH is 20% lower than the signal for a standard made in 0.025 M NaOH. As such, the concentration of NaOH in the preserved sample is somewhat important and should be kept the same in standards and samples.
3. 10 mg CN/L as ferrocyanide (Fe(II)) responds as 59.8 µg CN/L.

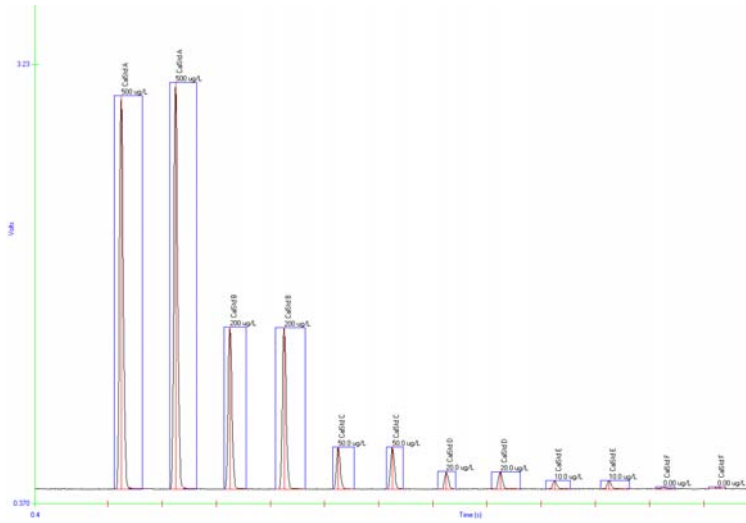
– Special Apparatus –

Please see Parts and Price list for Ordering Information

1. In-Line Sample Preparation Module (Lachat Part No. A30113 [110V], A30213 [220V])
2. The determination step requires a separate heating unit

SUPPORT DATA FOR QUIKCHEM 8000

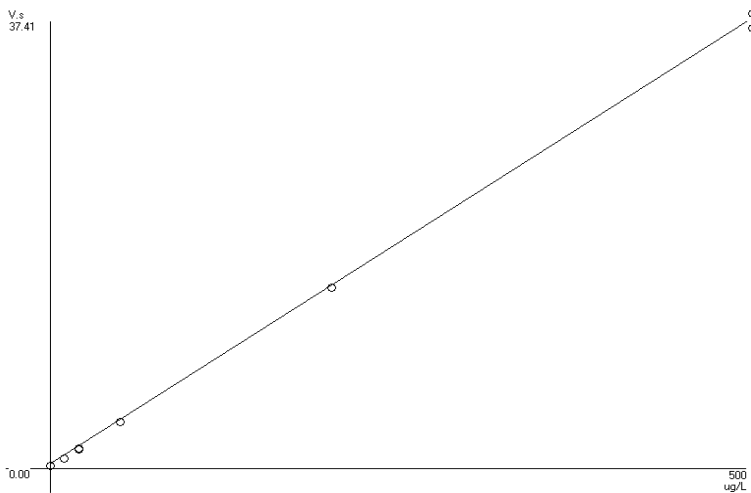
Calibration Data for WAD Cyanide



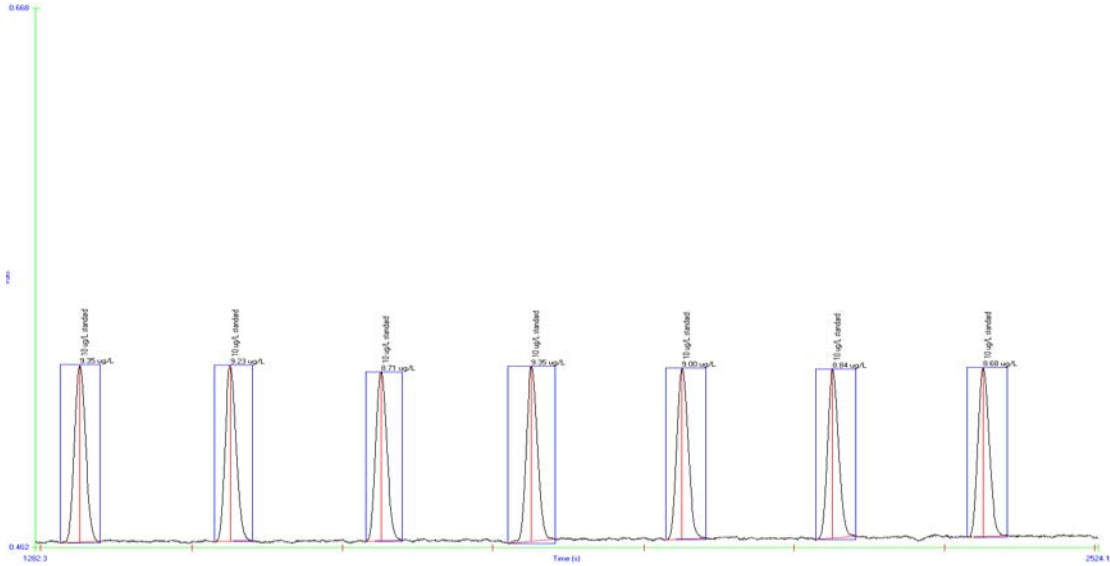
File Name: WAD CN Run 2.OMN
Acq. Date: 10 May 2004

Calibration Graph and Statistics

Cyanide						
	Known Concentration (ug/L)	Rep	Peak Area (V. s)	Peak Height (V)	% Residual	Unused
1	500	1	36.8	2.56	1.7	<input type="checkbox"/>
2	500	2	38.0	2.65	-1.6	<input type="checkbox"/>
3	200	1	15.1	1.06	-0.3	<input type="checkbox"/>
4	200	2	15.1	1.05	-0.3	<input type="checkbox"/>
5	50.0	1	3.89	0.271	-0.3	<input type="checkbox"/>
6	50.0	2	3.90	0.273	-0.5	<input type="checkbox"/>
7	20.0	1	1.65	0.114	0.0	<input type="checkbox"/>
8	20.0	2	1.63	0.112	1.1	<input type="checkbox"/>
9	10.0	1	0.813	0.0562	9.8	<input type="checkbox"/>
10	10.0	2	0.815	0.0554	9.6	<input type="checkbox"/>
11	0.00	1	0.221	0.0141		<input type="checkbox"/>
12	0.00	2	0.202	0.0134		<input type="checkbox"/>



Calibration graph for WAD Cyanide



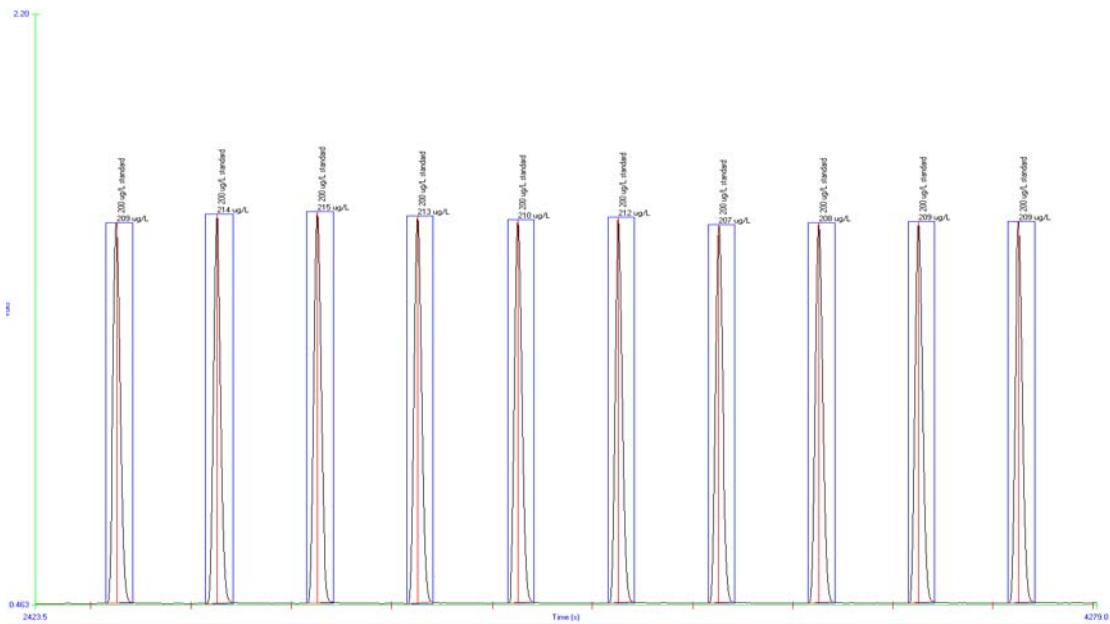
Method Detection Limit for WAD cyanide using 10 µg CN/L standard

MDL= 1 µg CN/L

Standard Deviation (s) = 0.29 µg CN/L, Mean (x) = 9.02 µg CN/L, Known value = 10.00 µg CN/L

File Name: WAD CN Run 1.OMN

Acq. Date: 11 May 2004



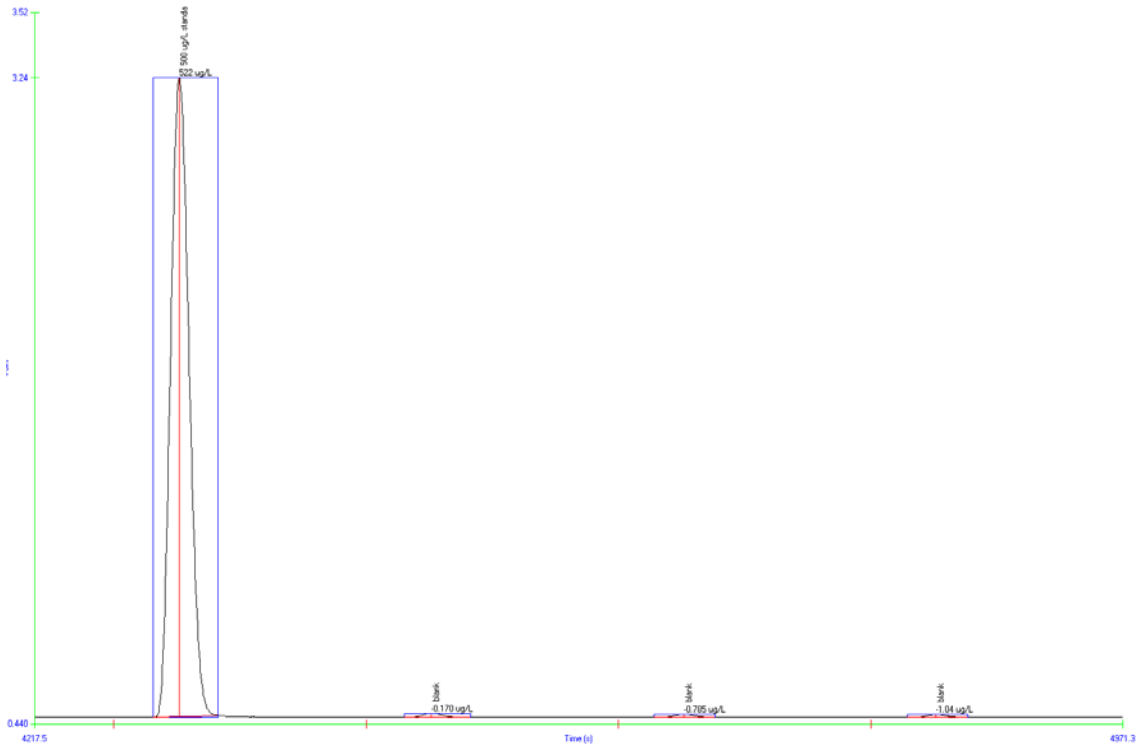
Precision data for WAD cyanide using 200 µg CN/L standard

% RSD = 1.29

Standard Deviation (s) = 2.71 µg CN/L, Mean (x) = 210.59 µg CN/L, Known value = 200 µg CN/L, %residual -5.3%

File Name: WAD CN Run 1.OMN

Acq. Date: 11 May 2004

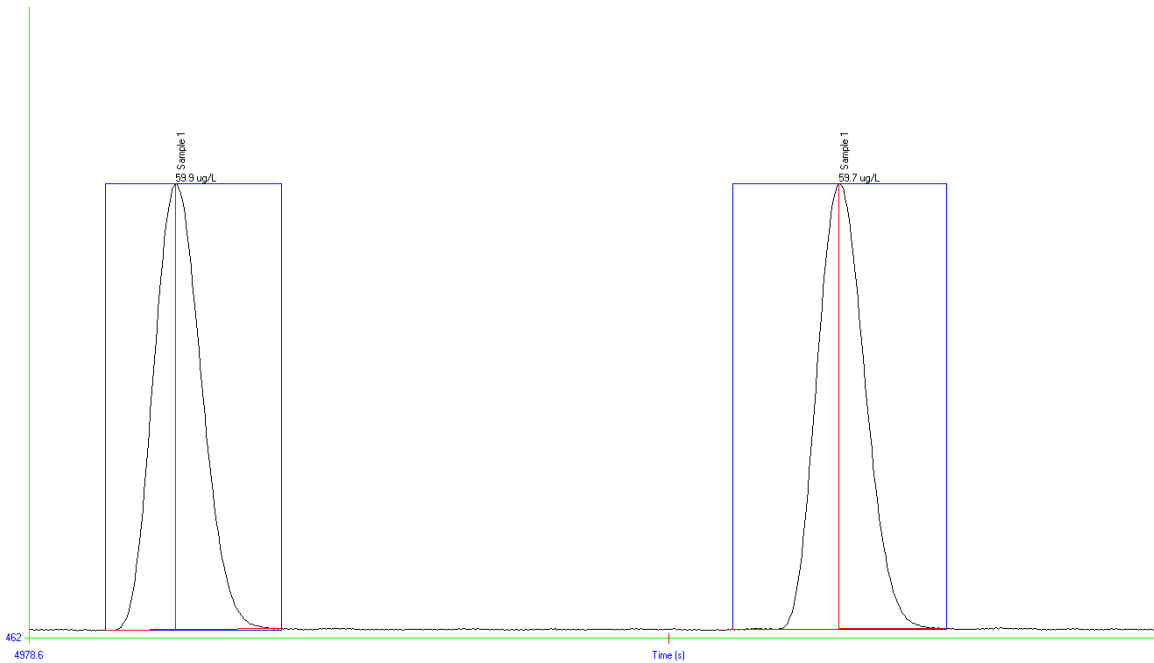


Carryover Study: 500 µg CN⁻/L standard followed by 3 blanks

Carryover Passed

File Name: WAD CN Run 1.OMN

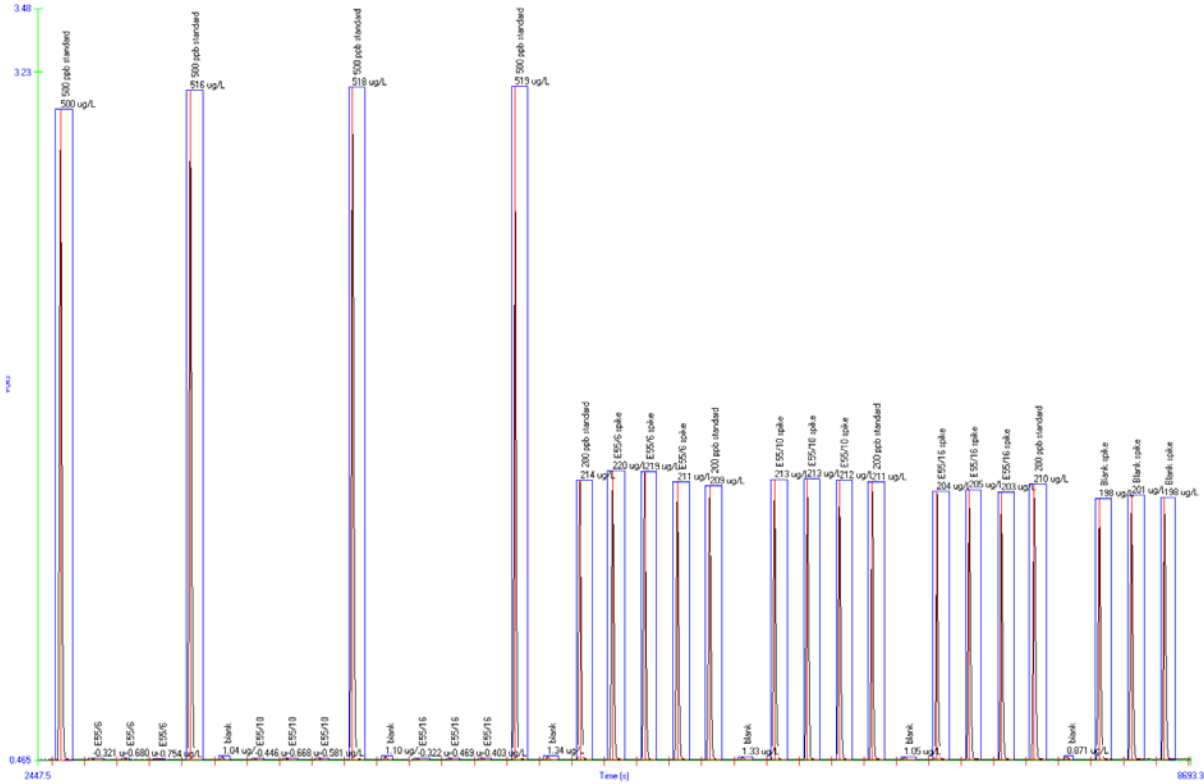
Acq. Date: 11 May 2004



A 10 mg CN⁻/L as ferrocyanide gives a 59.8 µg CN⁻/L response.

File Name: WAD CN Run 1.omn

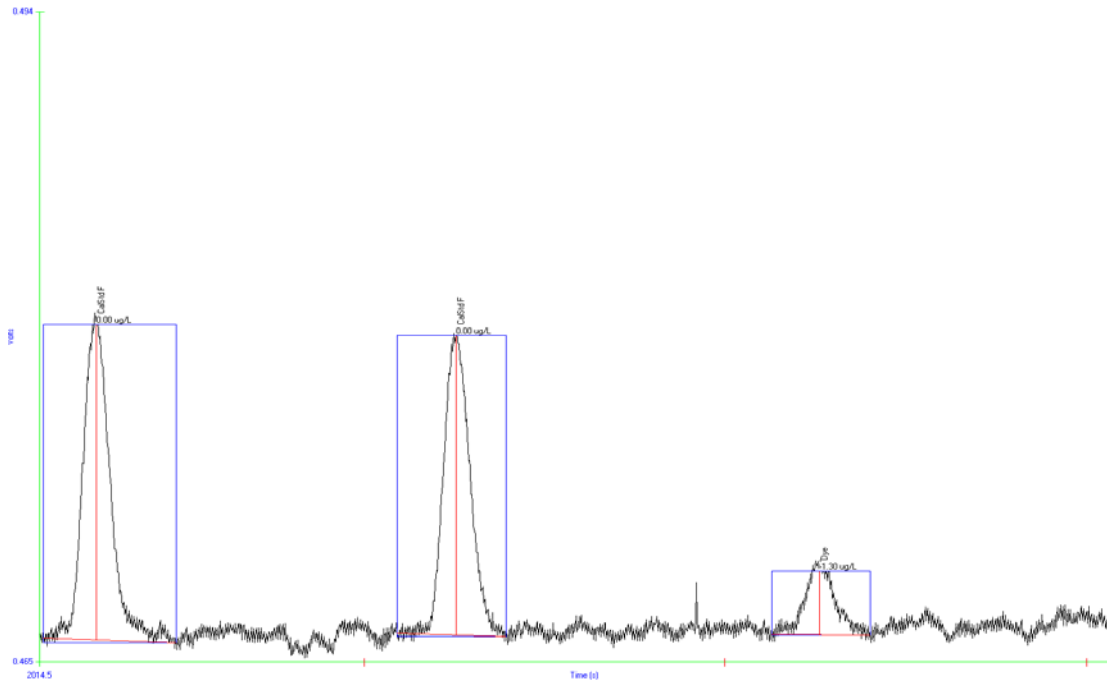
Acq. Date: 11 May 2004



Spike recoveries in wastewater samples.
File Name: WAD CN Run 2.omn
Acq. Date: 11 May 2004

Results of Spike Recoveries into Wastewater Samples.

Sample ID	Unspiked	Spiked	Spike Level	Spike Recovery
E55/6	0	218.83	200 µg CN/L	109.42%
E55/10	0	212.7	200 µg CN/L	106.35%
E55/16	0	203.97	200 µg CN/L	102%
Blank	0	199.17	200 µg CN/L	99.59%



Peaks for blanks versus Universal Dye. If a peak for universal dye that is greater than that for the blank is obtained, the membrane should be replaced.

Available Methods for In-Line, Weak Acid Dissociable CN			
Method Number	Method Type	Range	Comments
10-204-00-3-A	In-Line WAD	10-500 µg CN ⁻ /L	Pyridine/barbituric acid. Does not recover ferricyanide. Settleable particulates not suitable for the method. Industrial wastes need review prior to purchase.
10-204-00-4-B	In-Line WAD	2-100 µg CN ⁻ /L	Does not recover ferricyanide. Pyridine-free. Settleable particulates not suitable for the method. Industrial wastes need review prior to purchase. Requires 2 SPMs.

Amperometric Methods

QuikChem® Method 10-204-00-5-A

DETERMINATION OF AVAILABLE CYANIDE WITH LIGAND DISPLACEMENT AND FLOW INJECTION ANALYSIS (FIA) UTILIZING GAS DIFFUSION SEPARATION AND AMPEROMETRIC DETECTION

2.0 to 400 µg CN⁻/L

– Principle –

Ligand-exchange reagents are added at room temperature to a cyanide-containing sample in a pretreatment step. The ligand-exchange reagents form thermodynamically stable complexes with the transition metal ions, resulting in the release of cyanide ion from the metal-cyano complexes. The addition of hydrochloric acid converts cyanide ions to hydrogen cyanide (HCN_(g)) that passes through a gas diffusion membrane into an alkaline receiving solution where it is converted back to cyanide ion. The cyanide ion is monitored amperometrically with a silver working electrode, silver/silver chloride reference electrode, and platinum/stainless steel counter electrode, at an applied potential of zero volts. The current generated is proportional to the cyanide concentration present in the original sample.

– Interferences –

1. High levels of carbonate can release CO₂ into the acceptor stream and cause an interference with the amperometric detector that results in a slight masking effect (15 percent negative bias with 20 ppb cyanide in 1500 ppm carbonate).
2. Sulfide will diffuse through the gas diffusion membrane and can be detected in the amperometric flowcell. Oxidized products of sulfide can also rapidly convert CN⁻ to SCN⁻ at a high pH.
3. Refer to Section 4 of this method for additional information regarding interferents in the analysis of cyanide.

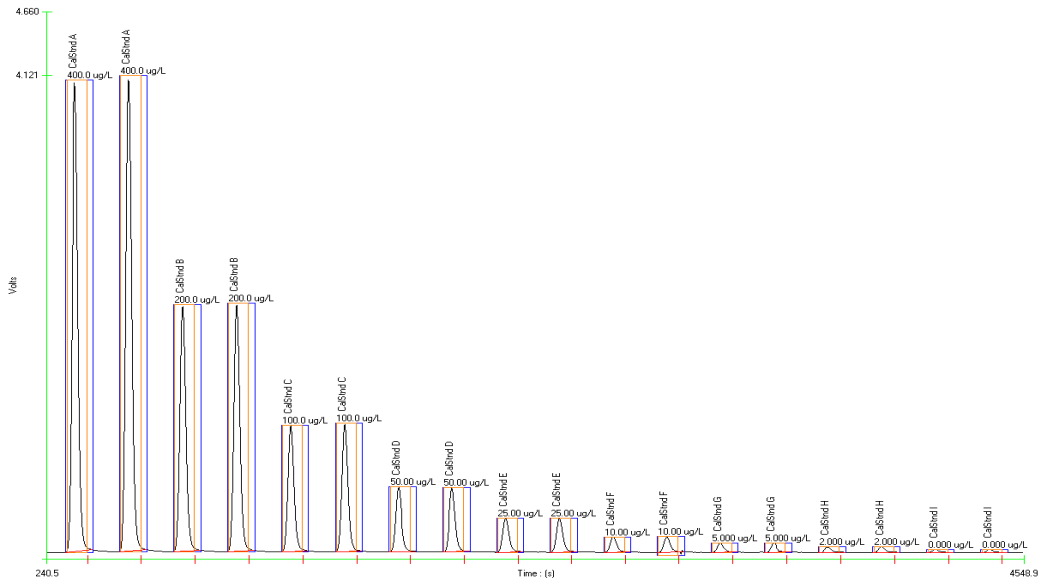
– Special Apparatus –

Please see Parts and Price list for Ordering Information

1. Amperometric Detector apparatus (Lachat Part No. 84920)
2. For XYZ sampler with 60 position racks (Lachat Part No. A81122/A81222)
3. Sample tubes are needed for 60 Position Samplers (Lachat Part No. 21042)
4. Heating Unit (Lachat Part No. A85100).
5. Direct Voltage Detector (Lachat Part No. 84967 for the QC8000; 85272 for the QC8500)

SUPPORT DATA FOR QUIKCHEM 8000/8500

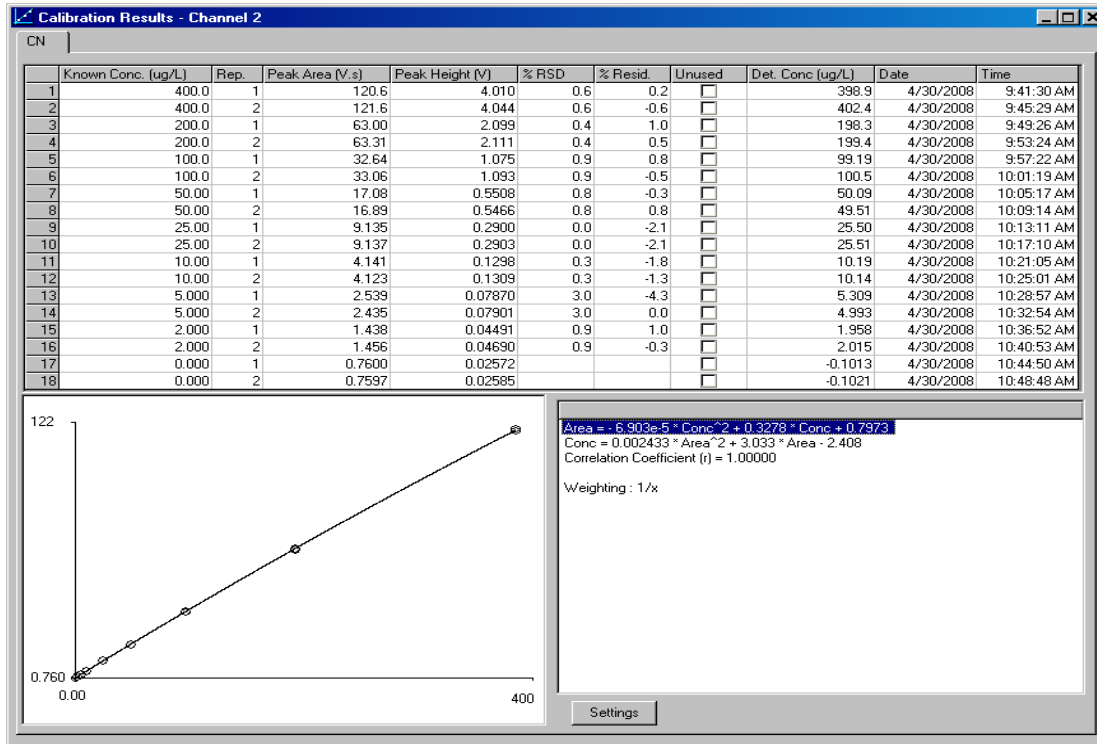
Calibration Data for Cyanide

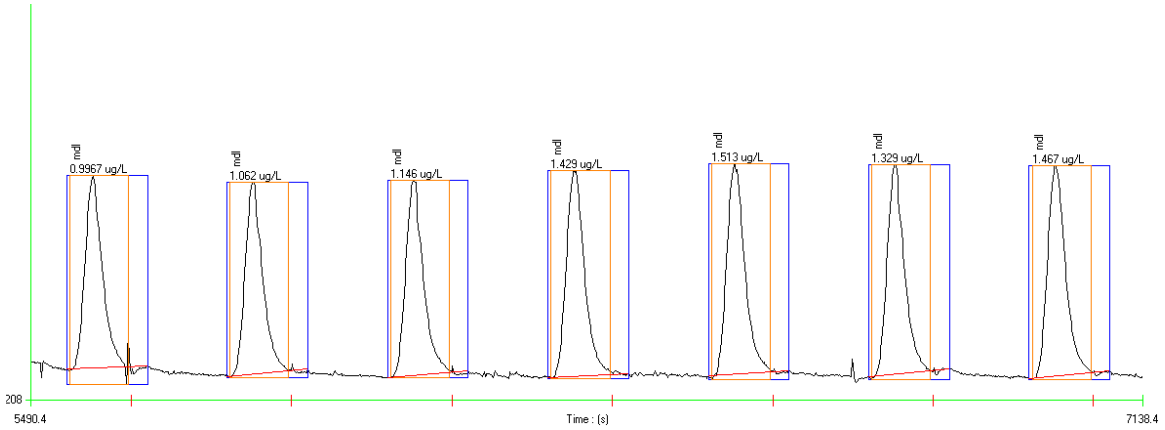


File Name: 4-30 cal support.OMN

Acq. Date: 30 April 2008

Calibration Graph and Statistics





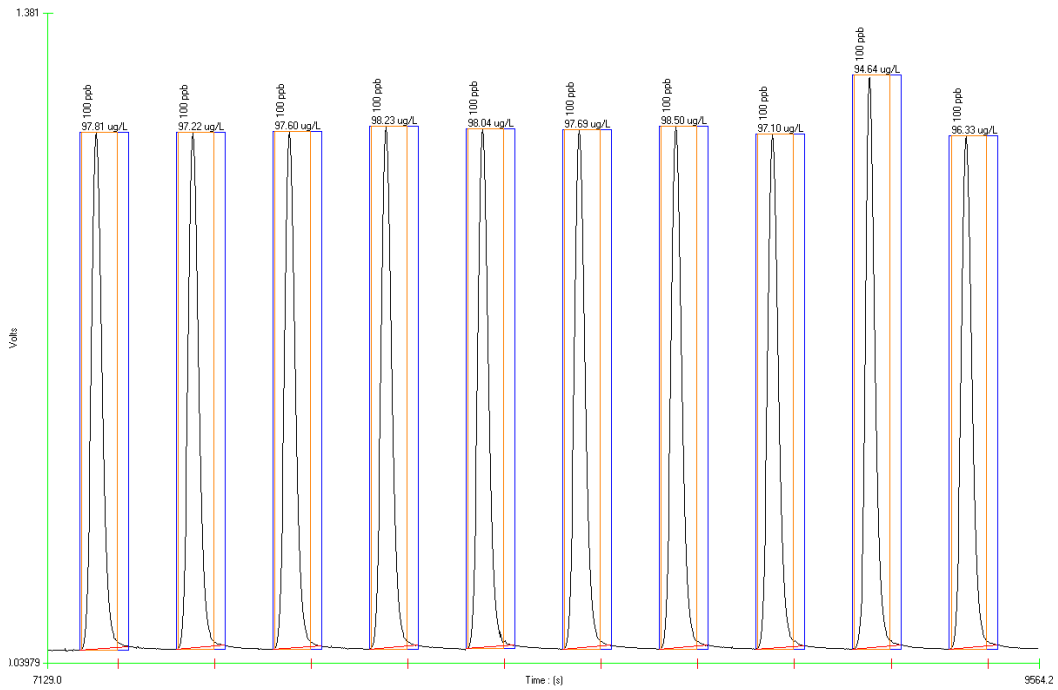
Method Detection Limit for cyanide using a 1.0 µg CN⁻/L standard

MDL = 0.65 µg CN⁻/L

Standard Deviation (s) = 0.208 µg CN⁻/L, Mean (x) = 1.28 µg CN⁻/L, Known value = 1.0 µg CN⁻/L

File Name: 4-30 cal support.OMN

Acq. Date: 30 April 2008



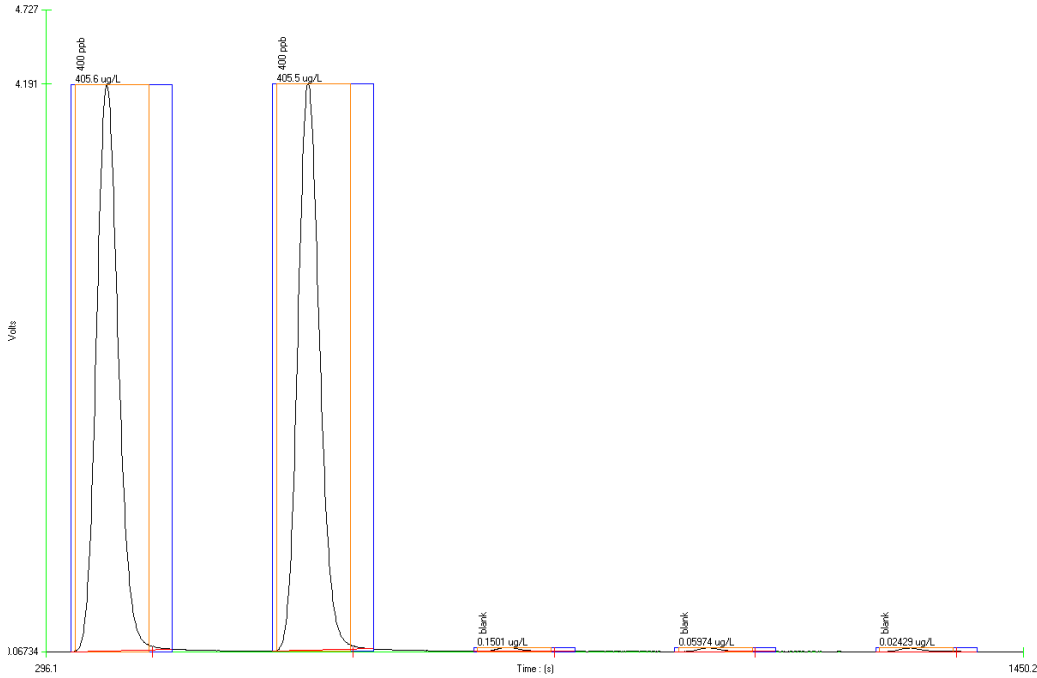
Precision data for cyanide using a 100 µg CN⁻/L standard

% RSD = 1.16 %

Standard Deviation (s) = 1.13 µg CN⁻/L, Mean (x) = 97.32 µg CN⁻/L, Known value = 100 µg CN⁻/L

File Name: 4-30 cal support.OMN

Acq. Date: 30 April 2008

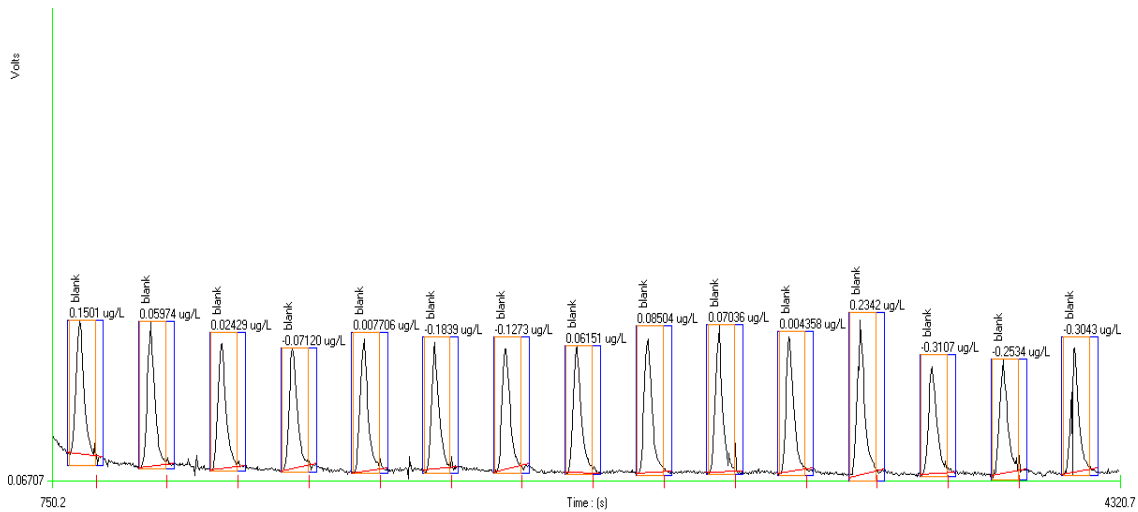


Carryover Study: 400 µg CN⁻/L standard followed by 3 blanks

Carryover Passed

File Name: 4-30 CO DIN.omn

Acq. Date: 30 April 2008



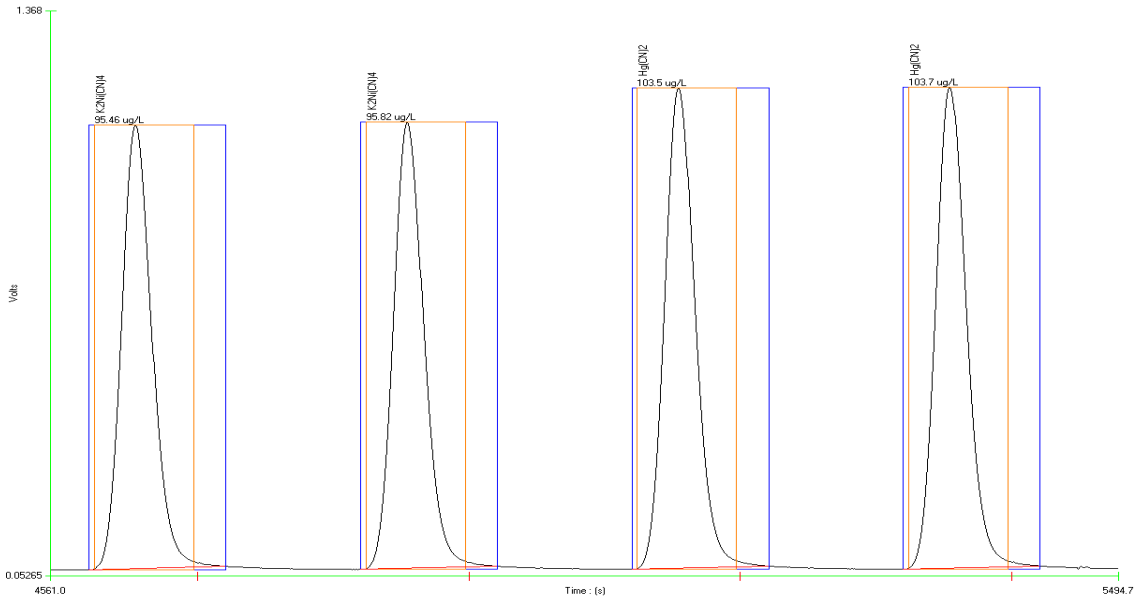
DIN Blanks

Average: -0.037 µg CN⁻/L, SD = 0.166 µg CN⁻/L. Calculated DIN Limits: Detection Limit = 0.498 µg CN⁻/L, Decision Limit = 0.996 µg CN⁻/L, Determination Limit = 1.49 µg CN⁻/L;

File Name: 4-30 CO DIN.omn

Acq. Date: 30 April 2008

Recovery of Nickel cyanide and Mercury cyanide complexes

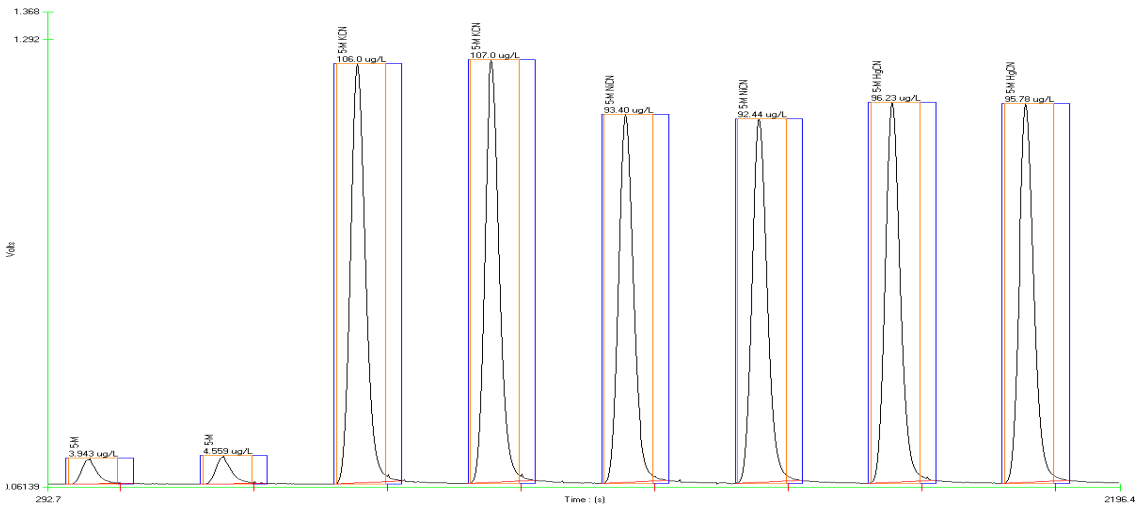


Compound	Amount in Sample	Average Value Obtained	% Recovery*
Nickel cyanide	100.0 µg CN-/L	95.64 µg CN-/L	95.64
Mercury cyanide	100.0 µg CN-/L	103.6 µg CN-/L	103.6

*(determined/known) * 100

Conclusion: Nickel and Mercury cyanide are recovered at levels greater than 95%.

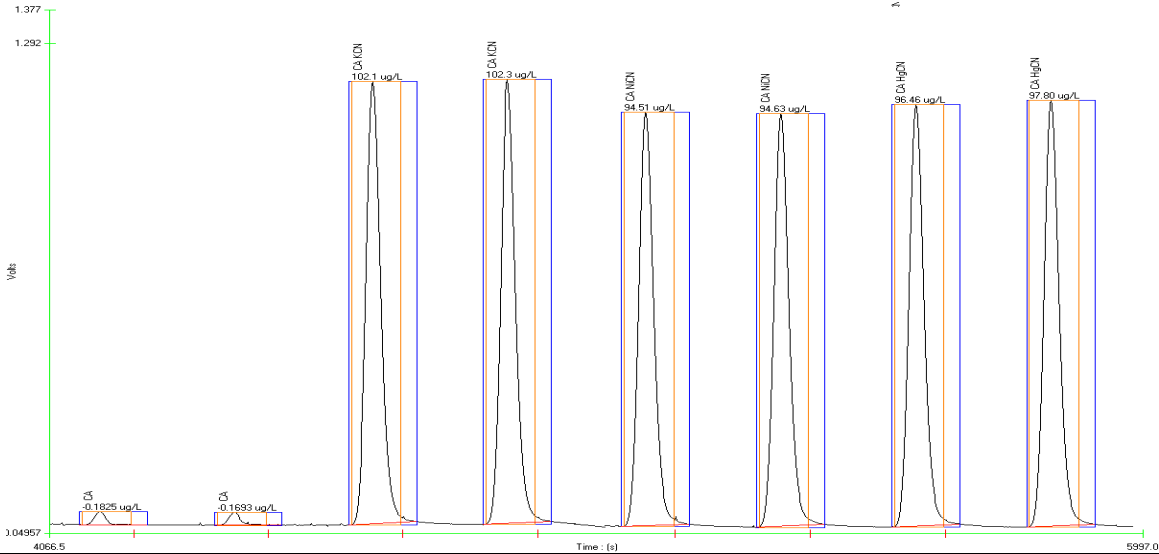
Cyanide Spike Recoveries in 5-Mile Effluent



Sample ID	Average spike recoveries (µg CN-/L)	Spike Level (µg CN-/L)	% Recovery
5-M effluent	4.25	100	---
5-M KCN	106.5	100	102.25
5-M K ₂ Ni(CN) ₄	92.92	100	88.67
5-M Hg(CN) ₂	96.00	100	91.75

Conclusion: Potassium, Nickel and Mercury cyanide are recovered at levels greater than 88%.

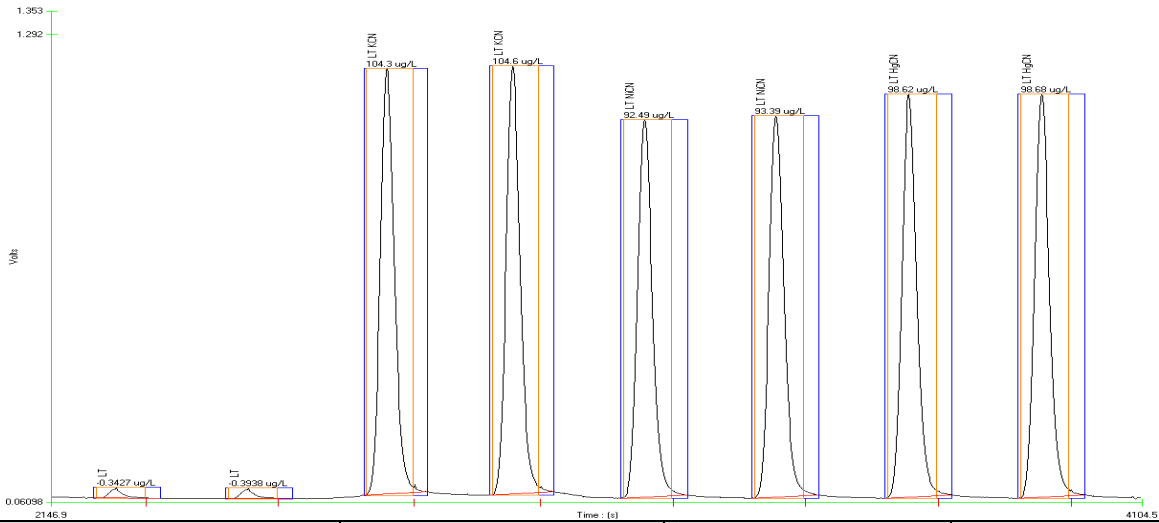
Cyanide Spike Recoveries in California Tap Water



Sample ID	Average spike recoveries ($\mu\text{g CN/L}$)	Spike Level ($\mu\text{g CN/L}$)	% Recovery
CA Tap Water	-0.176	100	---
CA Tap Water KCN	102.2	100	102.4
CA Tap Water $\text{K}_2\text{Ni}(\text{CN})_4$	94.75	100	94.75
CA Tap Water $\text{Hg}(\text{CN})_2$	97.31	100	97.31

Conclusion: Potassium, Nickel and Mercury cyanide are recovered at levels greater than 94%.

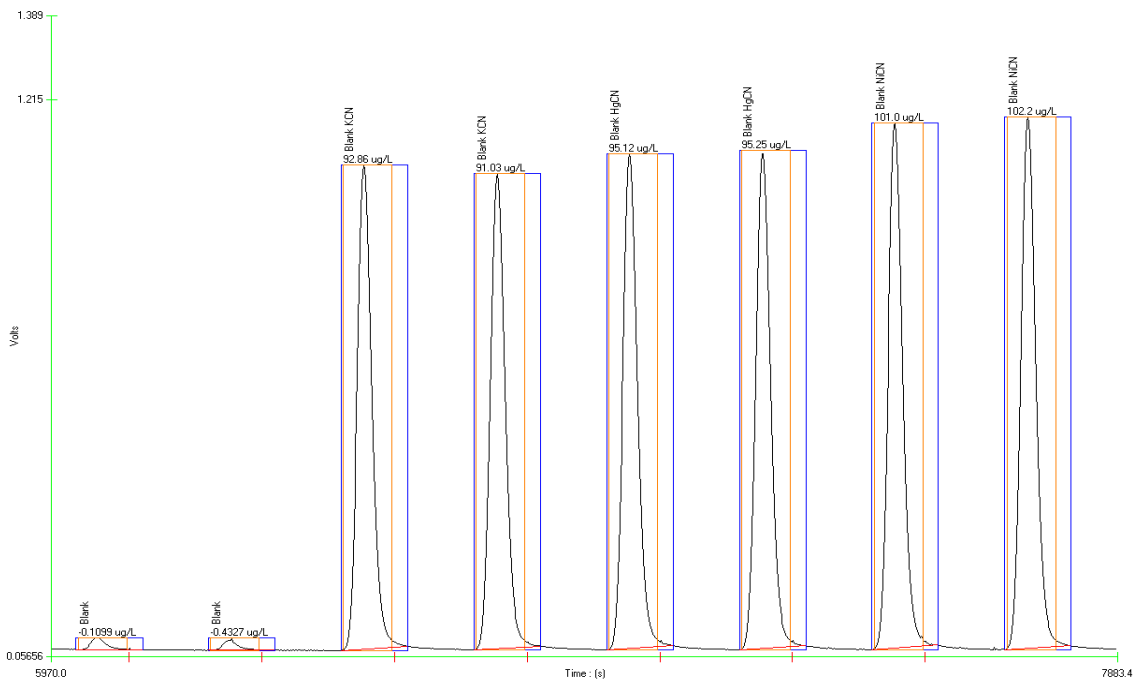
Cyanide Spike Recoveries in Loveland, CO Tap Water



Sample ID	Average spike recoveries ($\mu\text{g CN/L}$)	Spike Level ($\mu\text{g CN/L}$)	% Recovery
Loveland Tap Water	-0.368	100	---
Loveland Tap Water KCN	104.4	100	104.8
Loveland Tap Water $\text{K}_2\text{Ni}(\text{CN})_4$	92.94	100	93.31
Loveland Tap Water $\text{Hg}(\text{CN})_2$	98.65	100	99.02

Conclusion: Potassium, Nickel and Mercury cyanide are recovered at levels greater than 93%.

Cyanide Spike Recoveries in 0.025 N NaOH

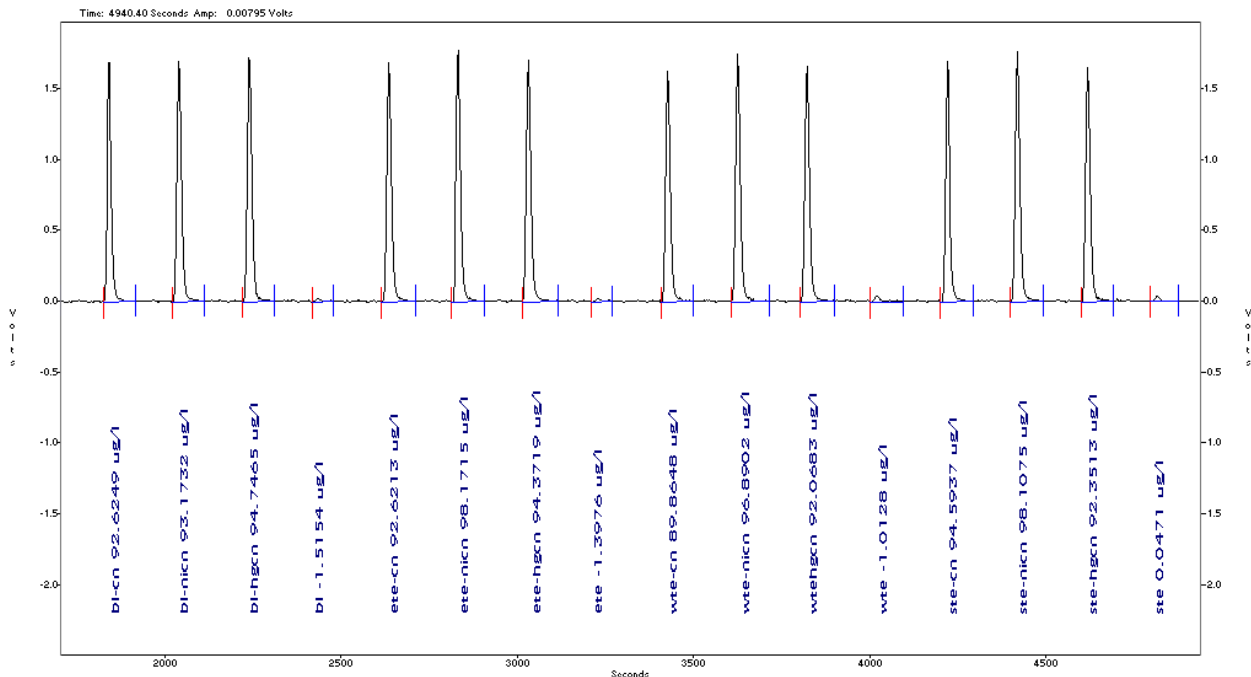


Sample ID	Average spike recoveries ($\mu\text{g CN/L}$)	Spike Level ($\mu\text{g CN/L}$)	% Recovery
0.025 N NaOH	-0.271	100	---

0.025 N NaOH KCN	92.22	100	92.22
0.025 N NaOH K ₂ Ni(CN) ₄	95.18	100	95.46
0.025 N NaOH Hg(CN) ₂	101.6	100	101.9

Conclusion: Potassium, Nickel and Mercury cyanide are recovered at levels greater than 92%.

Spike Recoveries from Previously Reported Data



Sample ID	Sample (spike level 94.34 µg CN/L)	EXP. Results (µg CN/L)	*Results (µg CN/L)	**Recovery (%)
bl-cn	KCN spike into blank	92.62	94.1	100
bl-nicn	(K ₂ Ni(CN) ₄ H ₂ O) spike into blank	93.17	94.7	100.6
bl-hgcn	(Hg(CN) ₂) spike into blank	94.75	96.3	102.3
bl	Blank (0.025M NaOH solution)	-1.52		
ete-cn	KCN spike into ETE sample	92.62	94.0	99.9
ete-nicn	(K ₂ Ni(CN) ₄ H ₂ O) spike into ETE sample	98.17	99.6	105.8
ete-hgcn	(Hg(CN) ₂) spike into ETE sample	94.37	95.8	101.7
ete	ETE sample	-1.40		
wte-cn	KCN spike into WTE sample	89.86	90.9	96.5
wte-nicn	(K ₂ Ni(CN) ₄ H ₂ O) spike into WTE sample	96.89	97.9	104.0
wtehgcn	(Hg(CN) ₂) spike into WTE sample	92.07	93.1	98.9
wte	WTE sample	-1.01		
ste-cn	KCN spike into STE sample	94.59	94.5	100.4
ste-nicn	(K ₂ Ni(CN) ₄ H ₂ O) spike into STE sample	98.11	98.1	104.2
ste-hgcn	(Hg(CN) ₂) spike into STE sample	92.35	92.3	98.0
ste	STE sample	0.05		

* Results = (EXP results – Blank)

** Recovery: Assume that the simple cyanide in blank are recovered 100%.

Conclusion:

- 1) Ligand exchange solution is working. Both nickel cyanide and mercury cyanide in blank are recovered 100%.
- 2) There is no sample matrix effect. All of the simple and complex cyanide tested were recovered at greater than 95%.

Available Amperometric Methods for Cyanide**			
Method Number	Method Type	Range	Comments
10-204-00-5-A[^]	Ligand Exchange	2-400 µg CN ⁻ /L	Settleable particulates not suitable for the method. Detector can be sold in the US only.
10-204-00-5-B	In-line Total CN	2-500 µg CN ⁻ /L	Settleable particulates not suitable for the method. Acidic, UV digestion. Selective against thiocyanate. Recovers Ferricyanide nearly quantitatively. Detector can be sold in the US only.
10-204-00-5-X	Micro Dist TM	µg CN ⁻ /L	Can be sold in the US only. The cyanide ion is trapped in a 0.1 M sodium hydroxide absorbing solution, which is diluted to 0.025 M solution during the distillation. Detector can be sold in the US only

****Available for sale in the U.S. only at this time.**



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