Data Pack

Methods for the Determination of Cyanide Using Amperometric Detection

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June 2008

LACHAT INSTRUMENTS
A Hach Company Brand

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This Data Pack contains support data and apparatus information on three Lachat methods for the determination of cyanide using amperometric detection. This allows for measurement of cyanide without the use of hazardous chemicals like pyridine and barbituric acid, meeting a sizable demand by customers in the Automated Photometry market.

Method Descriptions

Lachat QuikChem Method number 10-204-00-5-A utilizes Ligand Exchange reagents to free easily liberated cyanides from methyl complexes. The range for this method is 2-400 µg CN^-/L. This method is equivalent to EPA method number OIA1677.

Lachat QuikChem Method number 10-204-00-5-B utilizes heat, acid, and UV light to digest cyanide complexes in-line. The digested sample then passes through a diffusion block, where the cyanide (now present as $HCN_{(g)}$) is trapped in dilute NaOH. The cyanide present in the trapping solution is separated utilizing gas diffusion, then measured amperometrically.

Lachat QuikChem method number 10-204-00-5-X allows samples distilled utilizing the MicroDIST disposable distillation tubes to be measured through amperometric detection as well.

Special Apparatus

In addition to the QuikChem Flow Injection Analyzer and Method manifold, special apparatus is required to use the Lachat QuikChem amperometric methods:

- Amperometric Detector apparatus (Lachat part no. 84920)
- Heating Unit (Lachat part no. A85100)
- Direct Voltage Detector (Lachat part no. 85272)

Users of the amperometric methods need a good understanding of the Omnion software, flow injection analysis principles, and valve timing. Detailed instructions on how to install the required apparatus and copies of the published methods are required for installation and successful analysis.

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

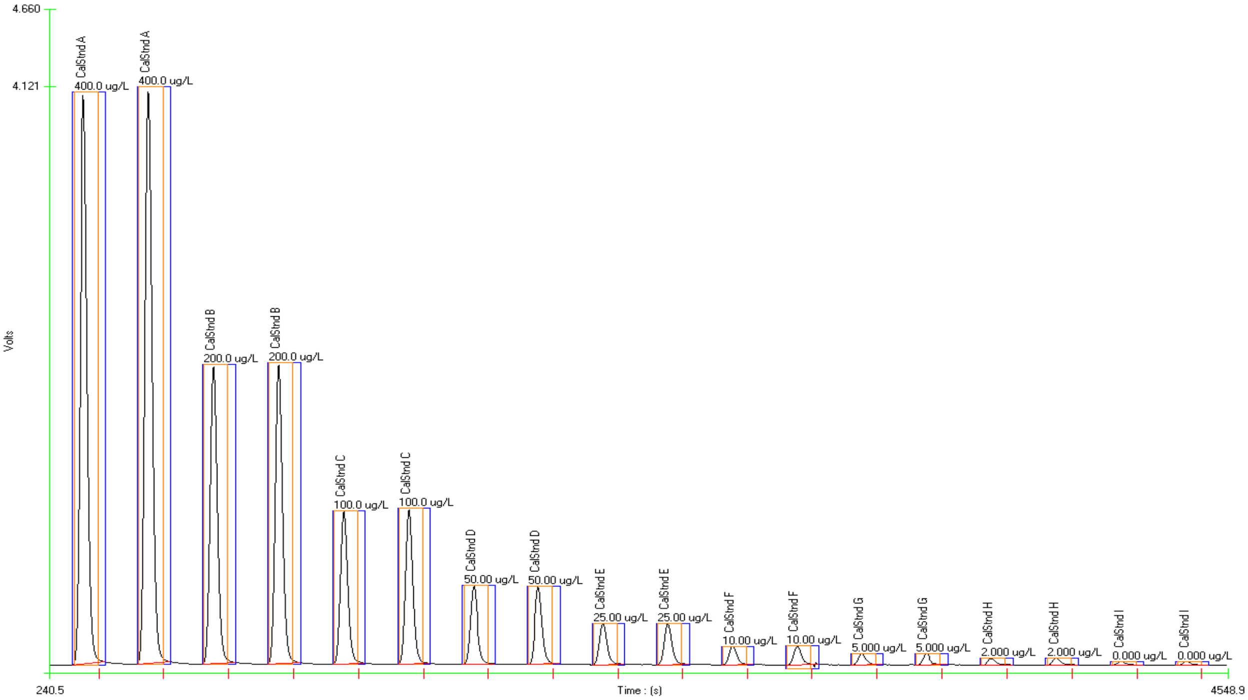
– Principle –

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 –

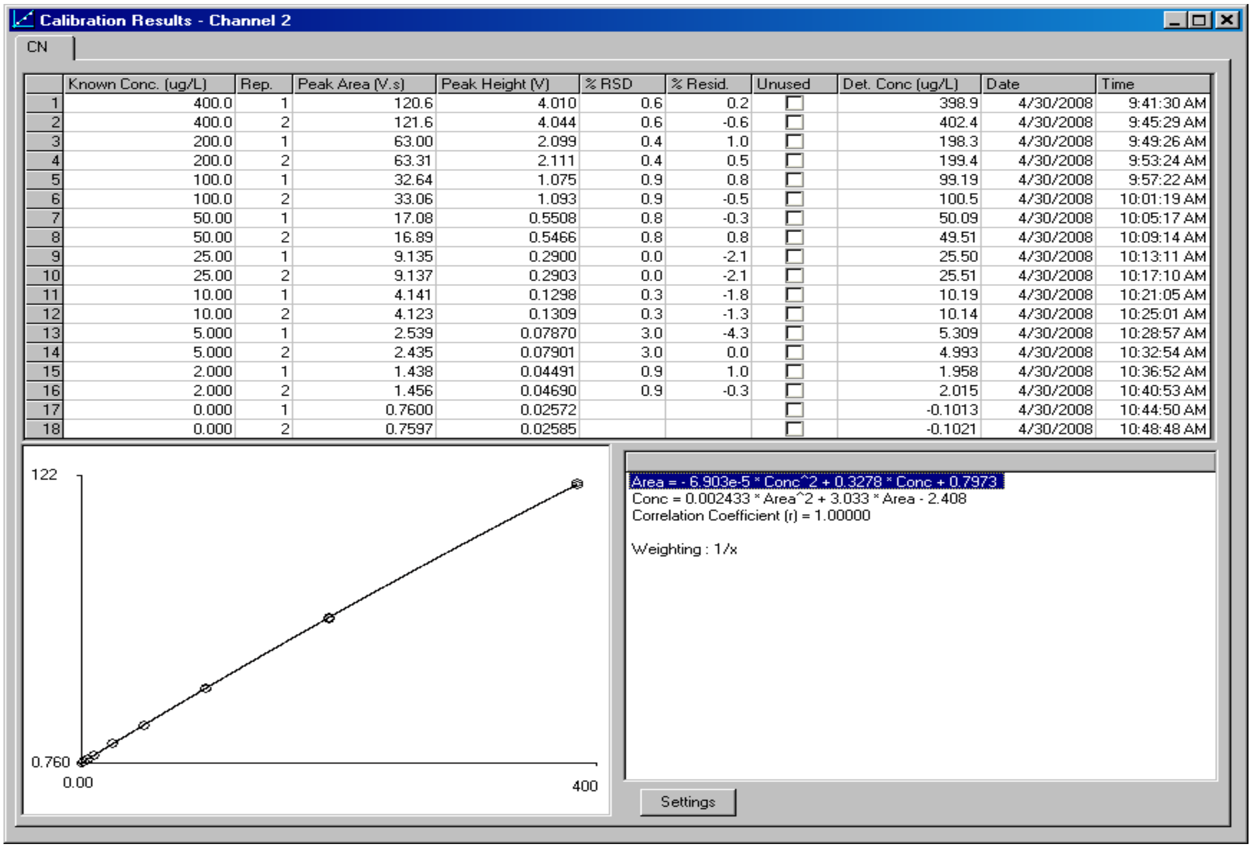
1. High levels of carbonate can release CO_2 into the acceptor stream and cause an interference with the amperometric detector that results in a slight masking effect (15 percent negative bias with 20 ppb cyanide in 1500 ppm carbonate).
2. Sulfide will diffuse through the gas diffusion membrane and can 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.

Calibration Data for Amperometric Cyanide



File Name: 4-30 cal support.OMN
Acq. Date: 30 April 2008

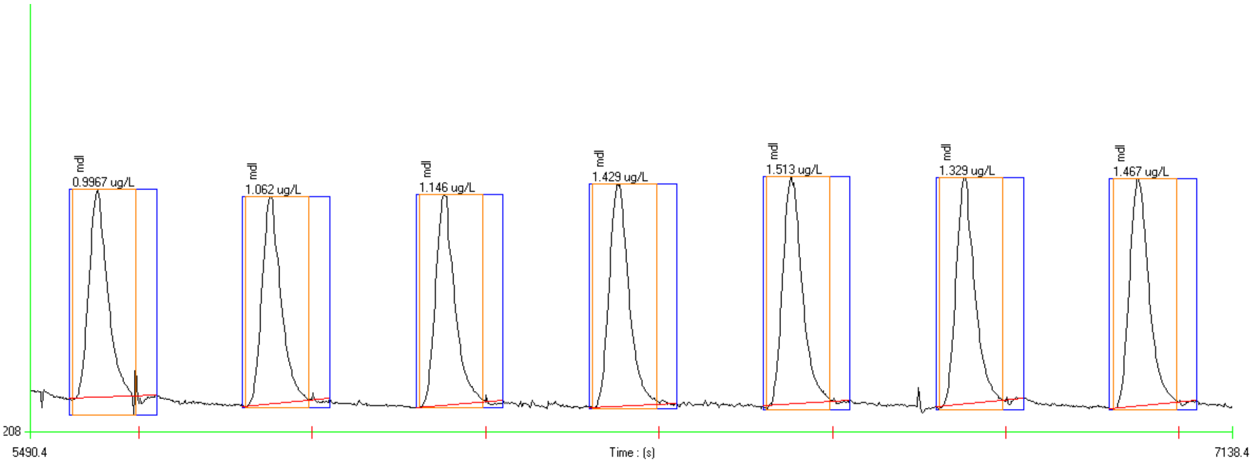
Calibration Graph and Statistics



	Known Conc. (ug/L)	Rep.	Peak Area (V.s)	Peak Height (V)	% RSD	% Resid.	Unused	Det. Conc (ug/L)	Date	Time
1	400.0	1	120.6	4.010	0.6	0.2	☐	398.9	4/30/2008	9:41:30 AM
2	400.0	2	121.6	4.044	0.6	-0.6	☐	402.4	4/30/2008	9:45:29 AM
3	200.0	1	63.00	2.099	0.4	1.0	☐	198.3	4/30/2008	9:49:26 AM
4	200.0	2	63.31	2.111	0.4	0.5	☐	199.4	4/30/2008	9:53:24 AM
5	100.0	1	32.64	1.075	0.9	0.8	☐	99.19	4/30/2008	9:57:22 AM
6	100.0	2	33.06	1.093	0.9	-0.5	☐	100.5	4/30/2008	10:01:19 AM
7	50.00	1	17.08	0.5508	0.8	-0.3	☐	50.09	4/30/2008	10:05:17 AM
8	50.00	2	16.89	0.5466	0.8	0.8	☐	49.51	4/30/2008	10:09:14 AM
9	25.00	1	9.135	0.2900	0.0	-2.1	☐	25.50	4/30/2008	10:13:11 AM
10	25.00	2	9.137	0.2903	0.0	-2.1	☐	25.51	4/30/2008	10:17:10 AM
11	10.00	1	4.141	0.1298	0.3	-1.8	☐	10.19	4/30/2008	10:21:05 AM
12	10.00	2	4.123	0.1309	0.3	-1.3	☐	10.14	4/30/2008	10:25:01 AM
13	5.000	1	2.539	0.07870	3.0	-4.3	☐	5.309	4/30/2008	10:28:57 AM
14	5.000	2	2.435	0.07901	3.0	0.0	☐	4.993	4/30/2008	10:32:54 AM
15	2.000	1	1.438	0.04491	0.9	1.0	☐	1.958	4/30/2008	10:36:52 AM
16	2.000	2	1.456	0.04690	0.9	-0.3	☐	2.015	4/30/2008	10:40:53 AM
17	0.000	1	0.7600	0.02572			☐	-0.1013	4/30/2008	10:44:50 AM
18	0.000	2	0.7597	0.02585			☐	-0.1021	4/30/2008	10:48:48 AM

Area = - 6.903e-5 * Conc^2 + 0.3278 * Conc + 0.7973
Conc = 0.002433 * Area^2 + 3.033 * Area - 2.408
Correlation Coefficient (r) = 1.00000

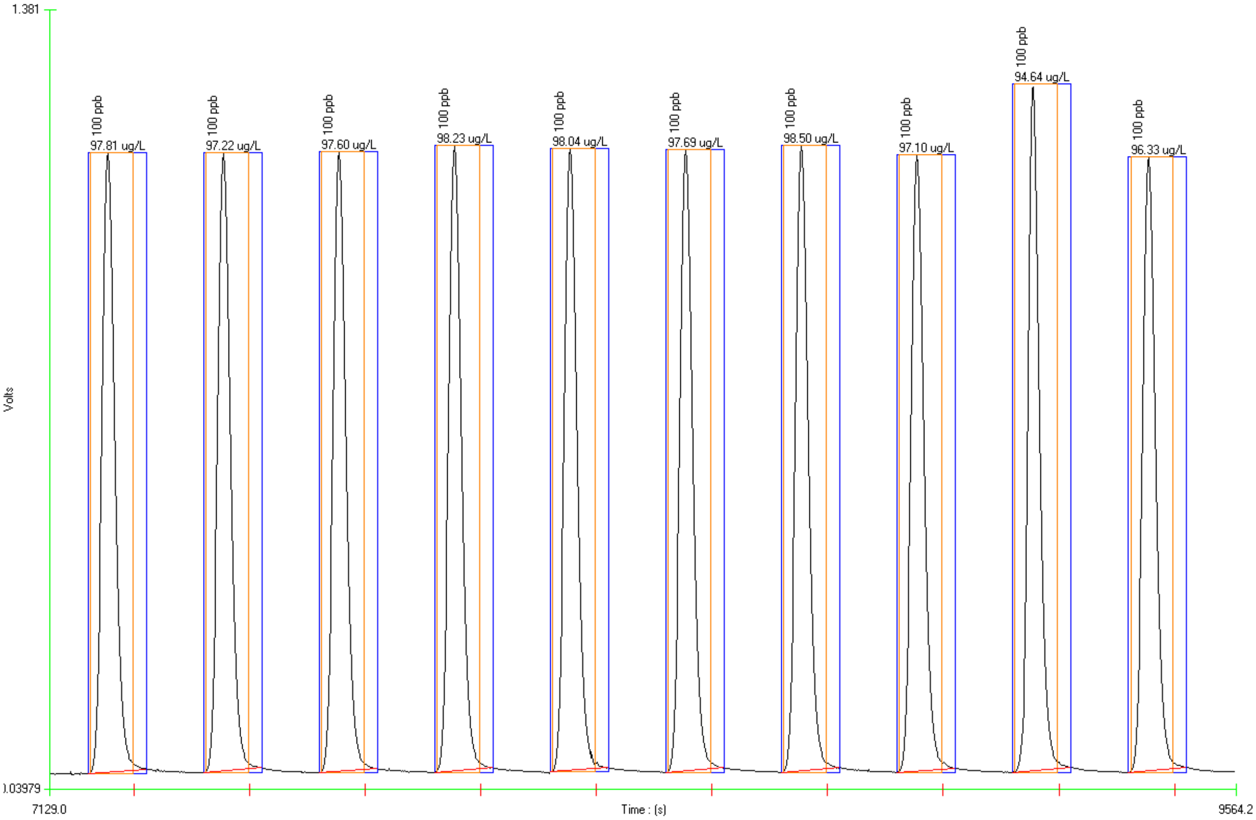
Weighting : 1/x



Method Detection Limit for cyanide using a 1.0 µg CN$^-$/L standard

MDL= 0.65 µg CN$^-$/L

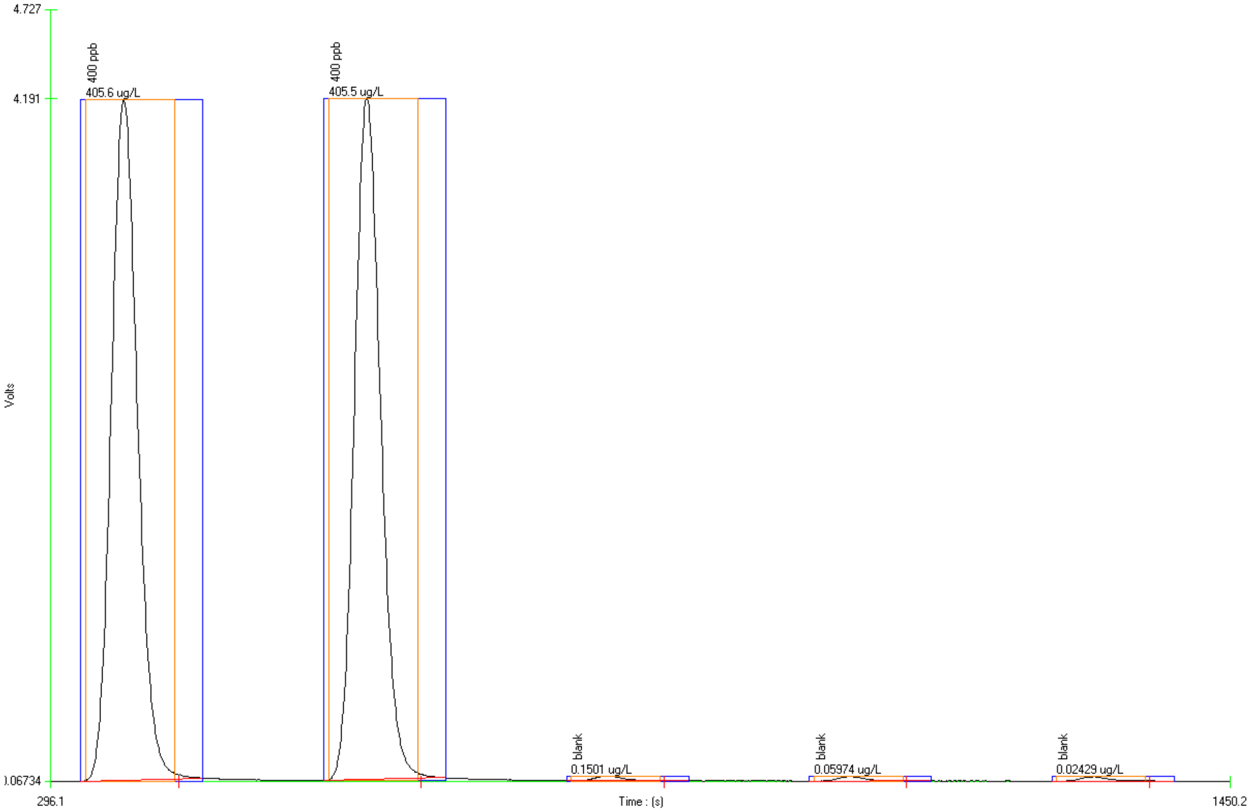
Standard Deviation (s) = 0.208 µg CN$^-$/L, Mean (x) = 1.28 µg CN$^-$/L, Known value = 1.0 µg CN$^-$/L
File Name: 4-30 cal support.OMN
Acq. Date: 30 April 2008



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

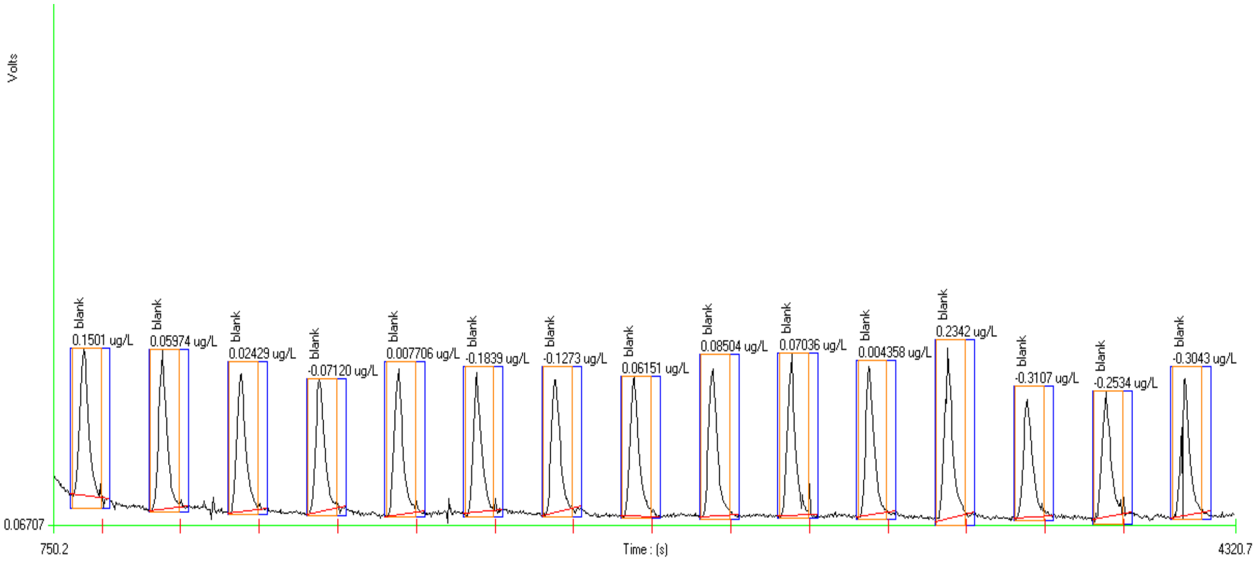


Carryover Study: 400 µg CN$^-$/L standard followed by 3 blanks

Carryover Passed

File Name: 4-30 CO DIN.omn

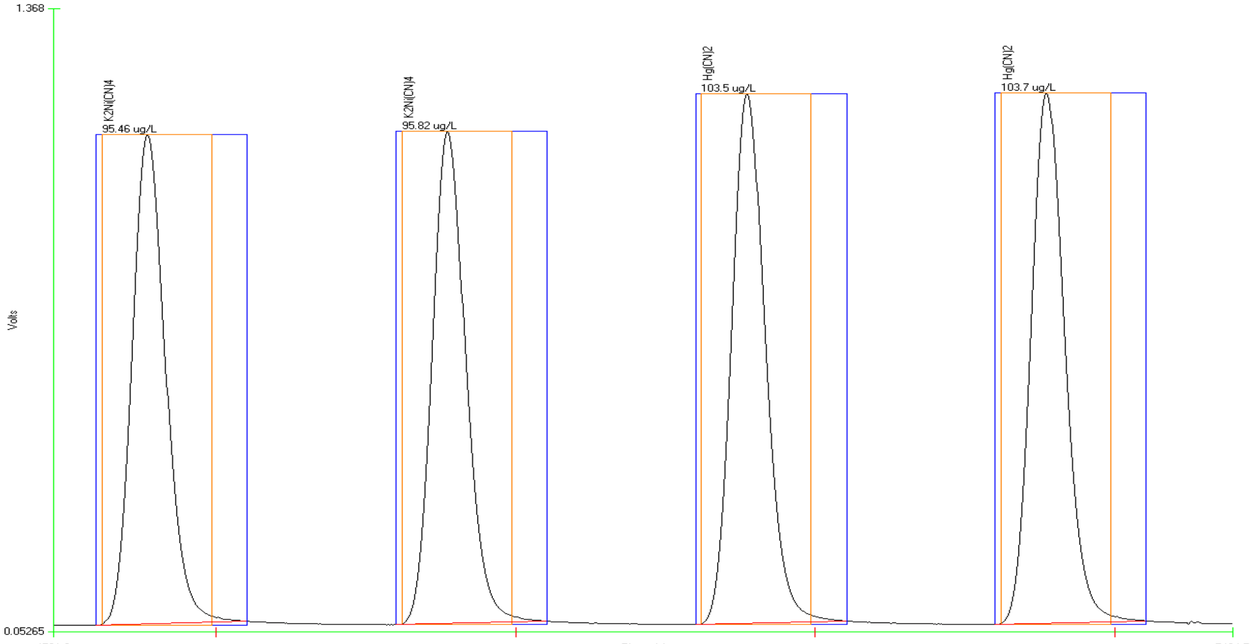
Acq. Date: 30 April 2008



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

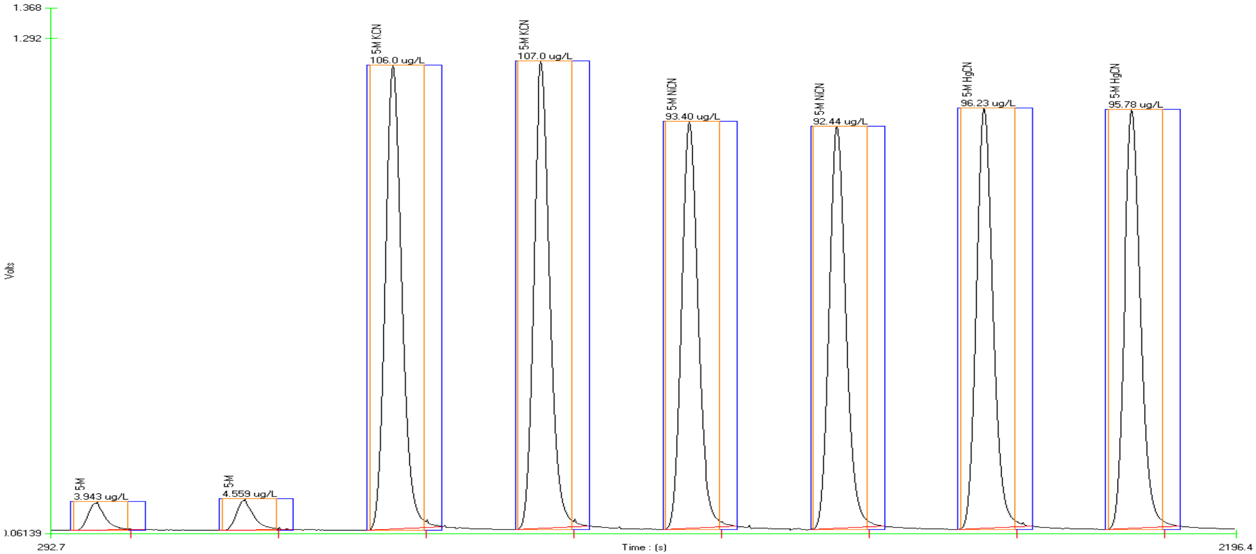


Compound	Amount in Sample	Average Value Obtained	% Recovery *
Nickel cyanide	100.0 µg CN-/L	95.64 µg CN^-/L	95.64
Mercury cyanide	100.0 µg CN-/L	103.6 µg CN^-/L	103.6

*(determined/known) * 100

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

Cyanide Spike Recoveries in 5-Mile Effluent

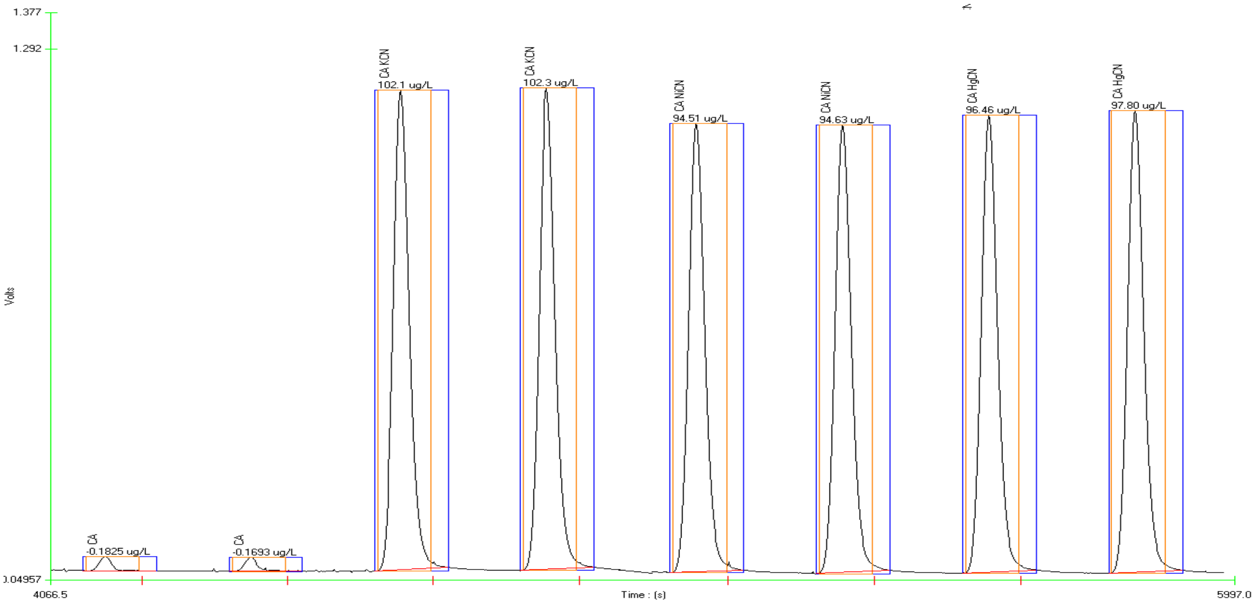


Sample ID	Average spike recoveries (µg CN^-/L)	Spike Level (µg CN^-/L)	% Recovery
5-M effluent	4.25	100	---
5-M KCN	106.5	100	102.25
5-M $K_2Ni(CN)_4$	92.92	100	88.67

5-M $Hg(CN)_2$	96.00	100	91.75

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

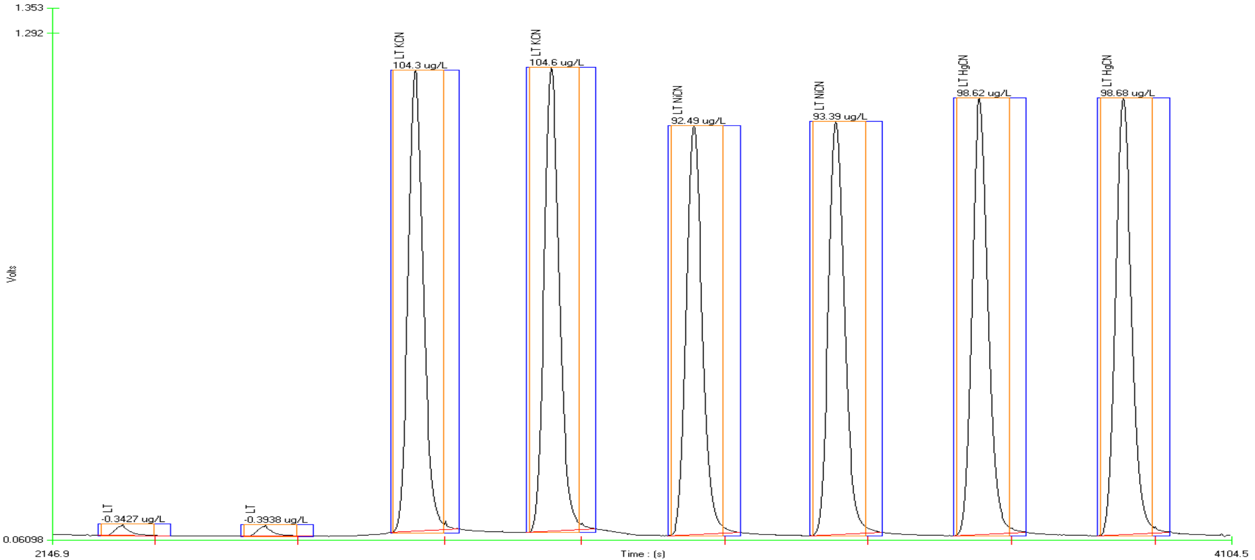
Cyanide Spike Recoveries in California Tap Water



Sample ID	Average spike recoveries ($\mu g\ CN^-/L$)	Spike Level ($\mu g\ CN^-/L$)	% Recovery
CA Tap Water	-0.176	100	---
CA Tap Water KCN	102.2	100	102.4
CA Tap Water $K_2Ni(CN)_4$	94.75	100	94.75
CA Tap Water $Hg(CN)_2$	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

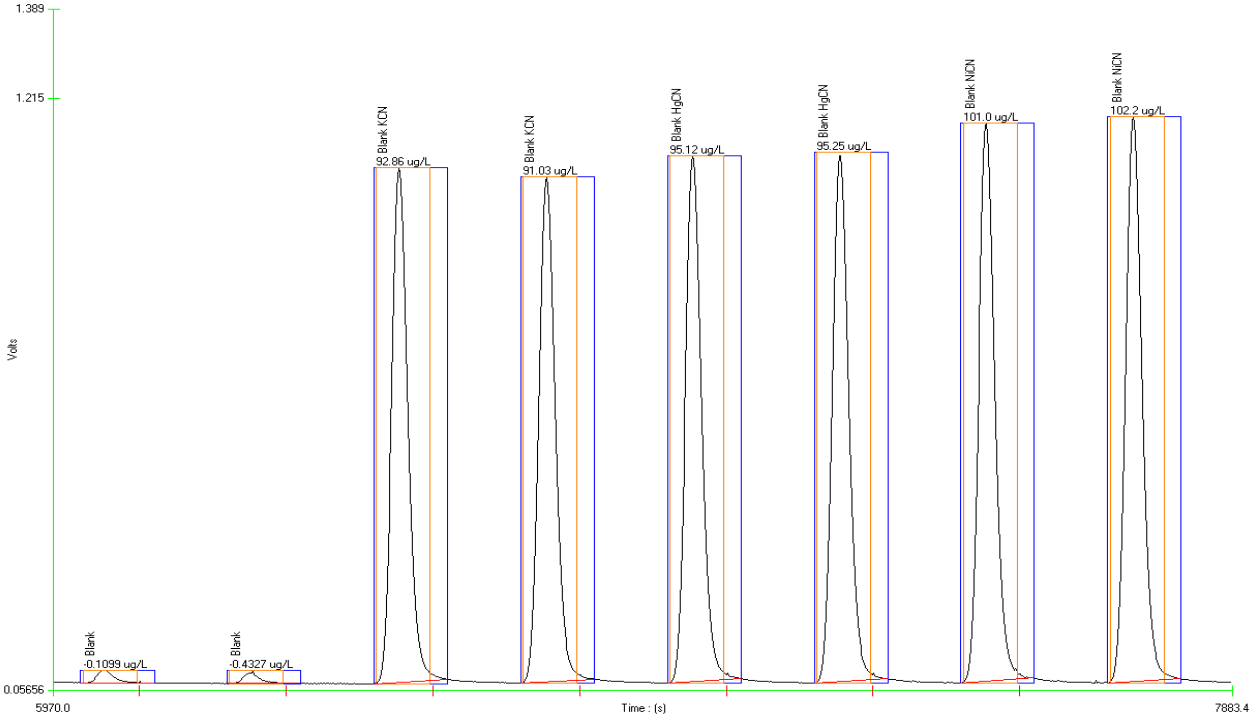


Sample ID	Average spike recoveries ($\mu g\ CN^-/L$)	Spike Level ($\mu g\ CN^-/L$)	% Recovery

Loveland Tap Water	-0.368	100	---
Loveland Tap Water KCN	104.4	100	104.8
Loveland Tap Water $K_2Ni(CN)_4$	92.94	100	93.31
Loveland Tap Water $Hg(CN)_2$	98.65	100	99.02

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

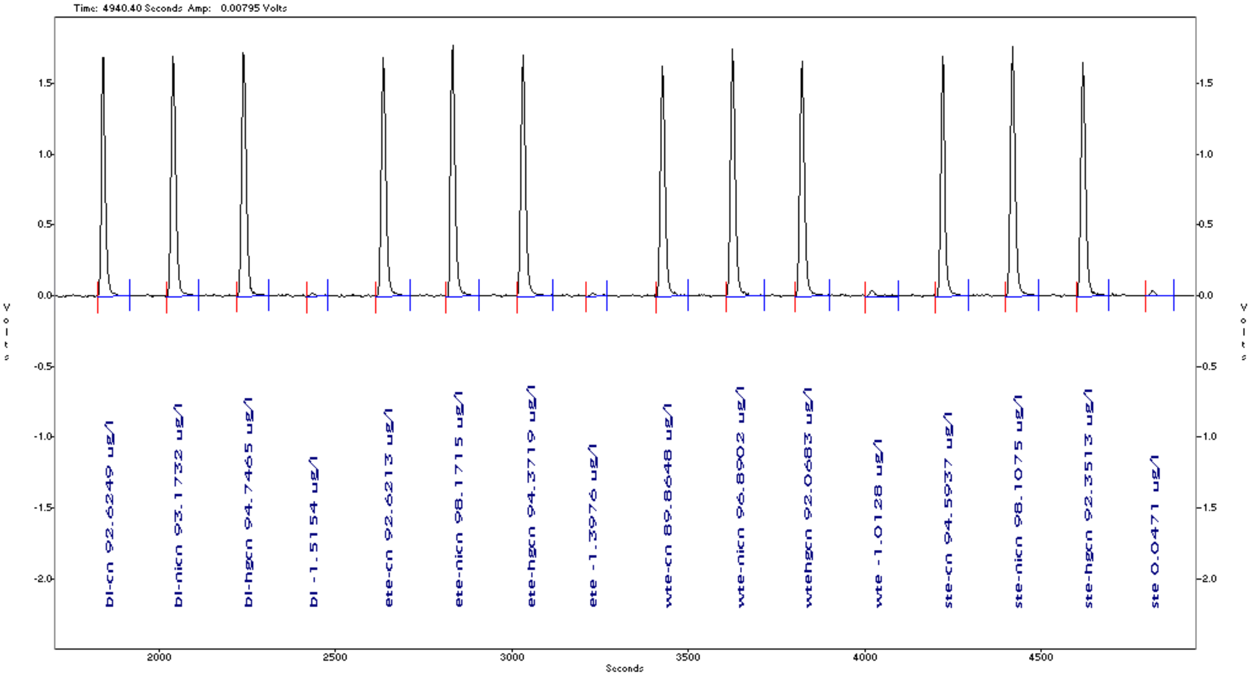
Cyanide Spike Recoveries in 0.025 N NaOH



Sample ID	Average spike recoveries ($\mu g\ CN^-/L$)	Spike Level ($\mu g\ CN^-/L$)	% Recovery
0.025 N NaOH	-0.271	100	---
0.025 N NaOH KCN	92.22	100	92.22
0.025 N NaOH $K_2Ni(CN)_4$	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	($K_2Ni(CN)_4$ H2O) 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	($K_2Ni(CN)_4$ H2O) 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	($K_2Ni(CN)_4$ H2O) 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	($K_2Ni(CN)_4$ H2O) 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%.

QuikChem® Method 10-204-00-5-B

DETERMINATION OF TOTAL CYANIDE BY FLOW INJECTION ANALYSIS UTILIZING IN-LINE DIGESTION, GAS DIFFUSION SEPARATION AND AMPEROMETRIC DETECTION

2.0 to 500 μg CN^-/L

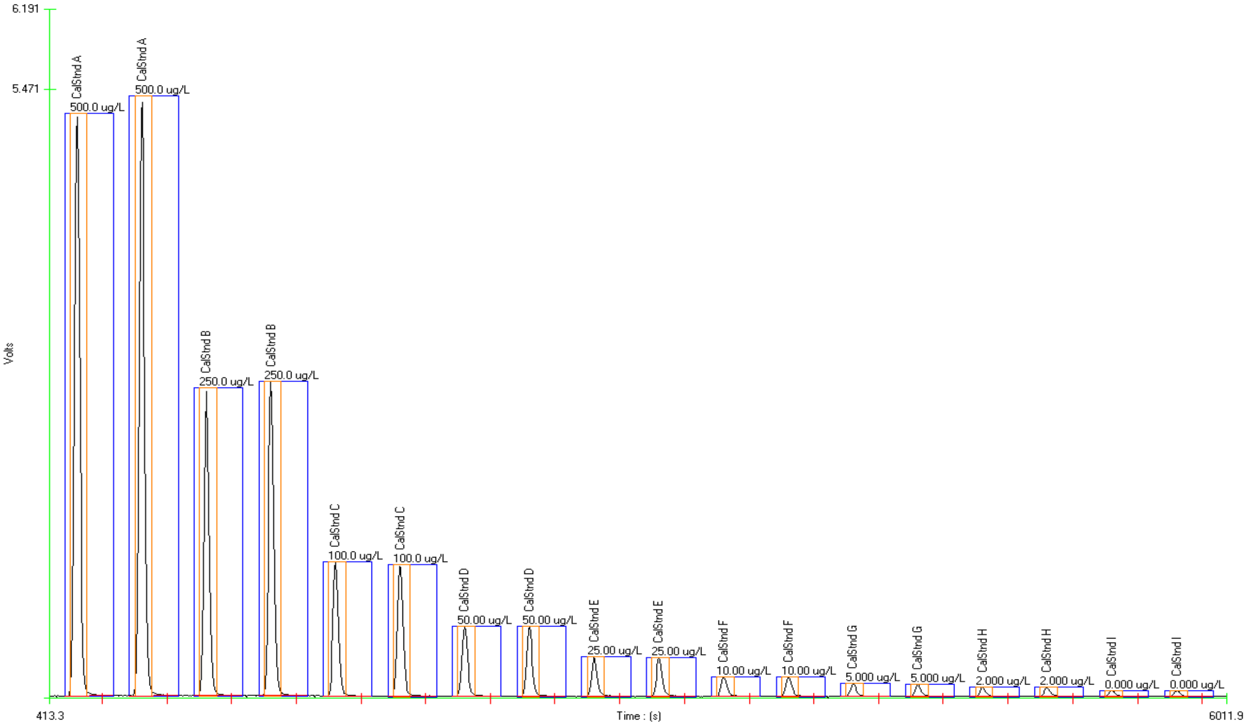
– Principle –

Liquid samples are first mixed with phosphoric acid, and then introduced into an in-line cyanide distillation unit. In this unit, the samples are first heated to 135 °C and then passed over a Black-Light Blue UV lamp (radiation peaks at 352 nm and 368 nm) to cleave metal-CN complexes. The CN^- released from these complexes during the heating and irradiation combines with protons to form HCN(g). The sample then exits the distillation unit and passes through a diffusion block where the HCN(g) diffuses across a Teflon membrane and is trapped as CN^- in a sodium hydroxide solution flowing across the bottom of the membrane (note: pKa of HCN is ~ 9.2-9.3). 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 –

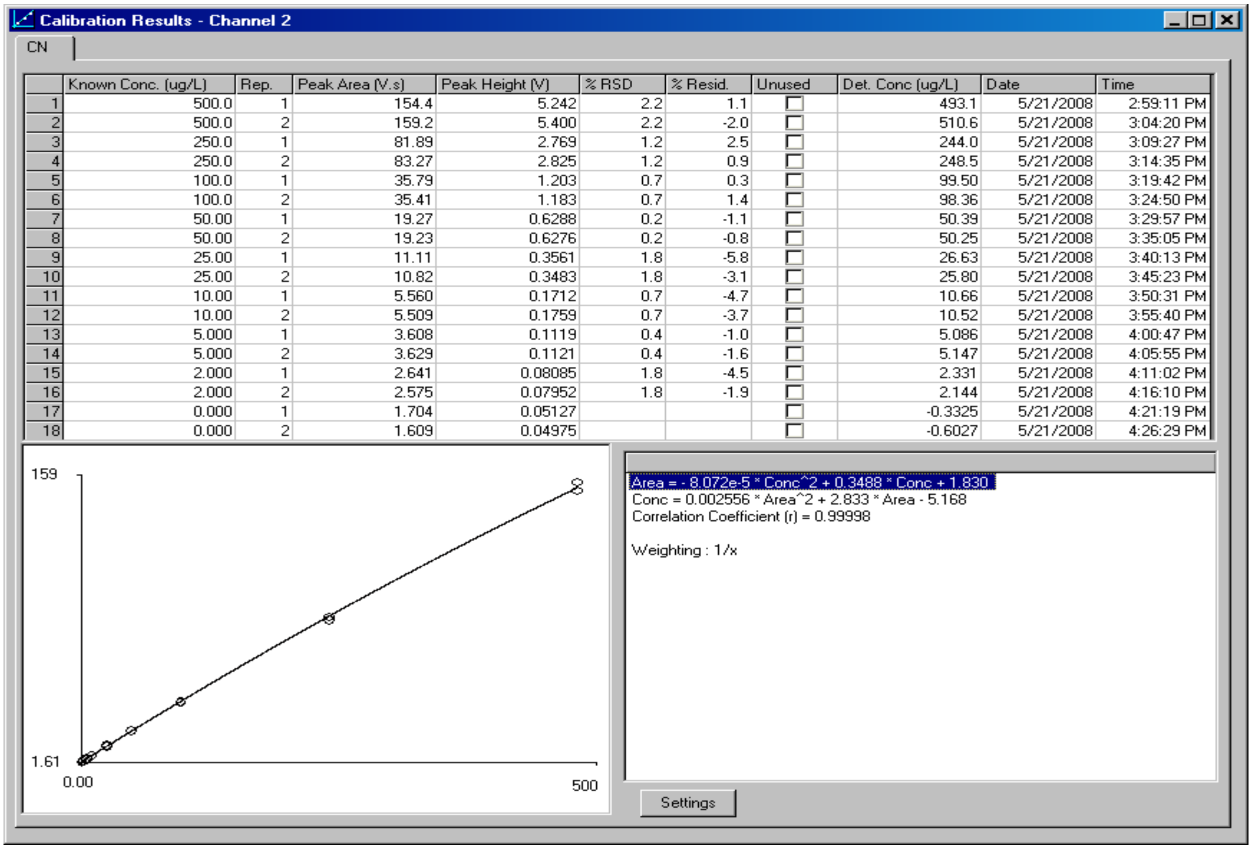
1. High levels of carbonate can release CO_2 into the acceptor stream and cause an interference with the amperometric detector that results in a slight masking effect (15 percent negative bias with 20 ppb cyanide in 1500 ppm carbonate).
2. Sulfide will diffuse through the gas diffusion membrane and can 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.
4. Thiocyanate is not a significant interference in this method. A 100 μg/L SCN^- standard gave an average CN^- response of –1.12 μg CN^- /L.

Calibration Data for Total Cyanide



File Name: 5-21 cal support.omn
Acq. Date: 21 May 2008

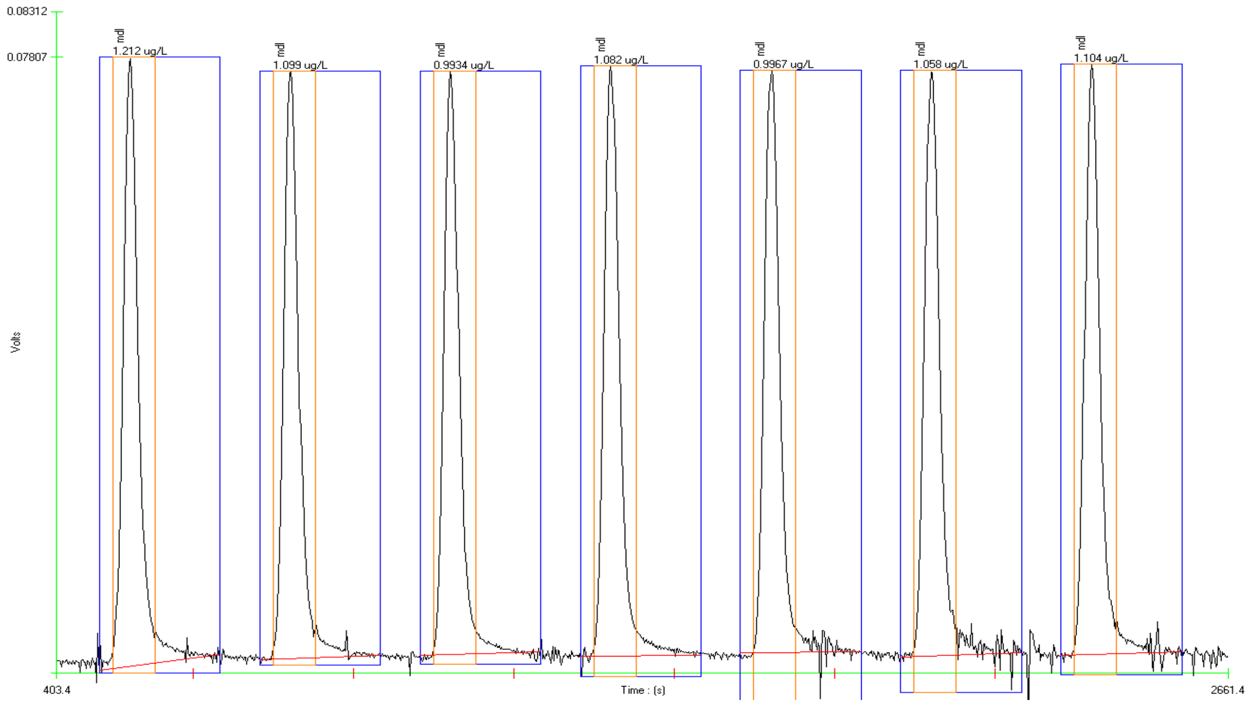
Calibration Graph and Statistics



	Known Conc. (ug/L)	Rep.	Peak Area (V.s)	Peak Height (V)	% RSD	% Resid.	Unused	Det. Conc (ug/L)	Date	Time
1	500.0	1	154.4	5.242	2.2	1.1	☐	493.1	5/21/2008	2:59:11 PM
2	500.0	2	159.2	5.400	2.2	-2.0	☐	510.6	5/21/2008	3:04:20 PM
3	250.0	1	81.89	2.769	1.2	2.5	☐	244.0	5/21/2008	3:09:27 PM
4	250.0	2	83.27	2.825	1.2	0.9	☐	248.5	5/21/2008	3:14:35 PM
5	100.0	1	35.79	1.203	0.7	0.3	☐	99.50	5/21/2008	3:19:42 PM
6	100.0	2	35.41	1.183	0.7	1.4	☐	98.36	5/21/2008	3:24:50 PM
7	50.00	1	19.27	0.6288	0.2	-1.1	☐	50.39	5/21/2008	3:29:57 PM
8	50.00	2	19.23	0.6276	0.2	-0.8	☐	50.25	5/21/2008	3:35:05 PM
9	25.00	1	11.11	0.3561	1.8	-5.8	☐	26.63	5/21/2008	3:40:13 PM
10	25.00	2	10.82	0.3483	1.8	-3.1	☐	25.80	5/21/2008	3:45:23 PM
11	10.00	1	5.560	0.1712	0.7	-4.7	☐	10.66	5/21/2008	3:50:31 PM
12	10.00	2	5.509	0.1759	0.7	-3.7	☐	10.52	5/21/2008	3:55:40 PM
13	5.000	1	3.608	0.1119	0.4	-1.0	☐	5.086	5/21/2008	4:00:47 PM
14	5.000	2	3.629	0.1121	0.4	-1.6	☐	5.147	5/21/2008	4:05:55 PM
15	2.000	1	2.641	0.08085	1.8	-4.5	☐	2.331	5/21/2008	4:11:02 PM
16	2.000	2	2.575	0.07952	1.8	-1.9	☐	2.144	5/21/2008	4:16:10 PM
17	0.000	1	1.704	0.05127			☐	-0.3325	5/21/2008	4:21:19 PM
18	0.000	2	1.609	0.04975			☐	-0.6027	5/21/2008	4:26:29 PM

Area = - 8.072e-5 * Conc^2 + 0.3488 * Conc + 1.830
Conc = 0.002556 * Area^2 + 2.833 * Area - 5.168
Correlation Coefficient (r) = 0.99998

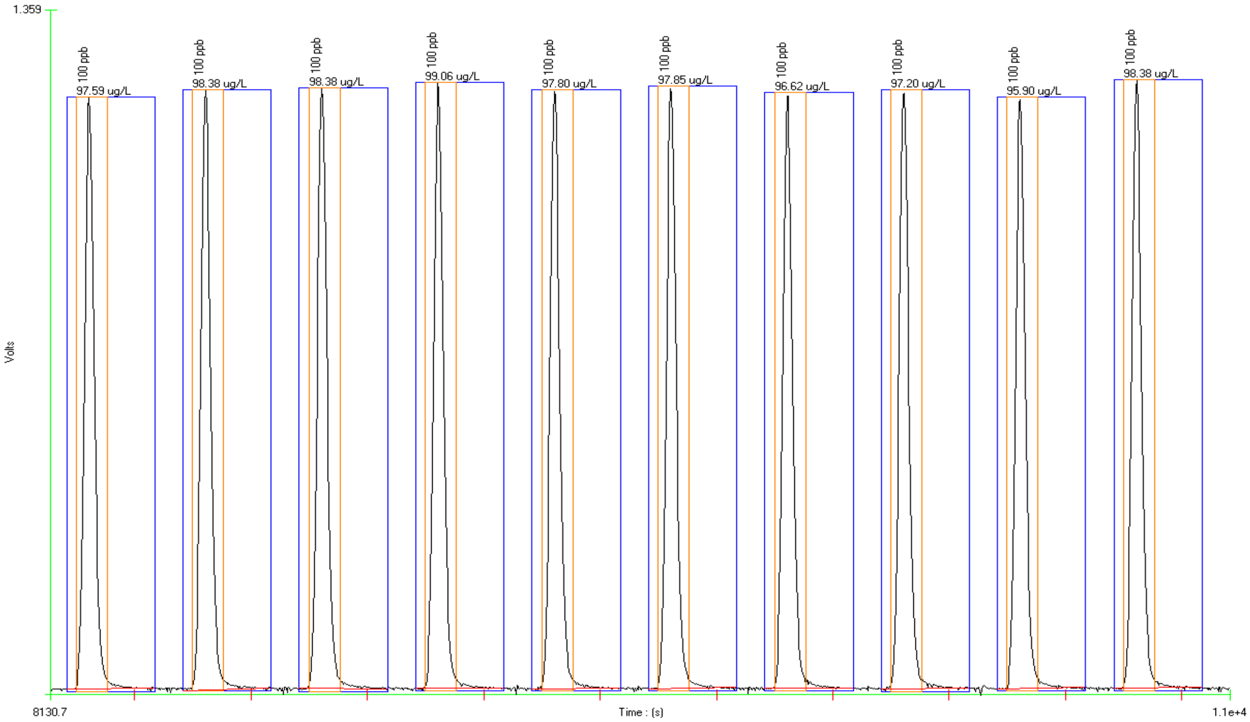
Weighting : 1/x



Method Detection Limit for cyanide using a 1.0 µg CN$^-$/L standard

MDL= 0.234 µg CN$^-$/L, Reporting 0.914 µg CN$^-$/L due to carry over.

Standard Deviation (s) = 0.074 µg CN$^-$/L, Mean (x) = 1.08 µg CN$^-$/L, Known value = 1.0 µg CN$^-$/L
File Name: 5-22 mdl.omn
Acq. Date: 22 May 2008

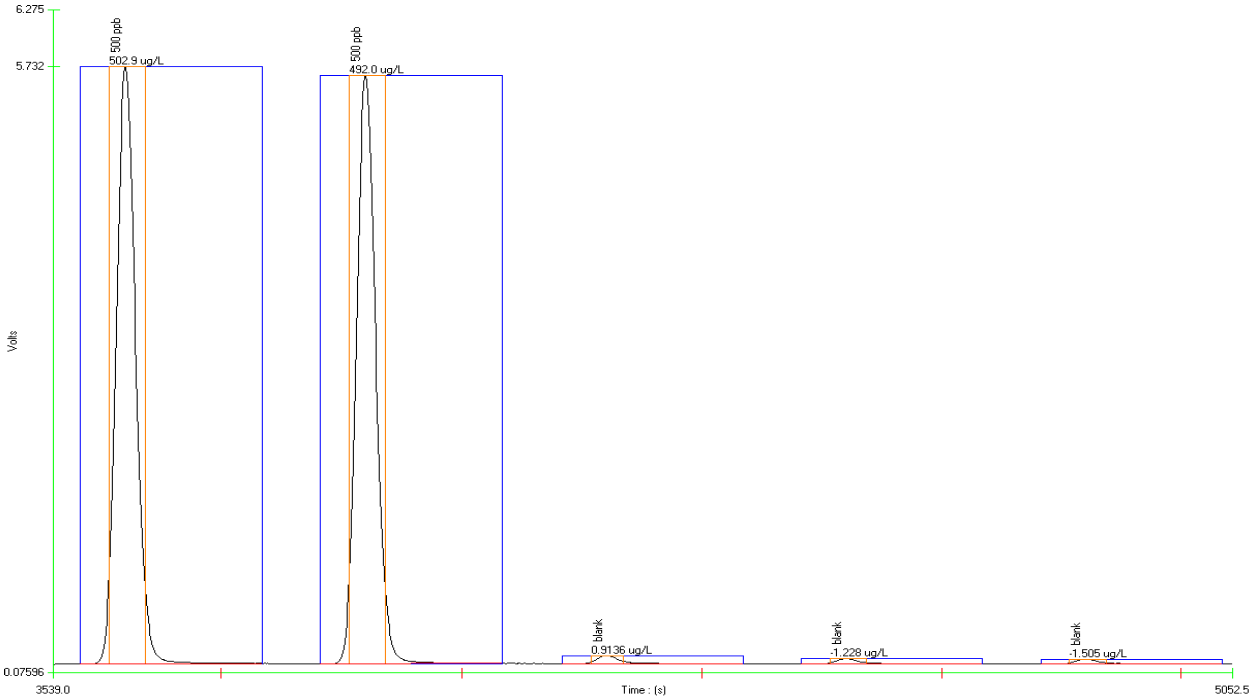


Precision data for cyanide using a 100 µg CN$^-$/L standard

% RSD = 0.96 %

Standard Deviation (s) = 0.94 µg CN$^-$/L, Mean (x) = 97.72 µg CN$^-$/L, Known value = 100 µg CN$^-$/L

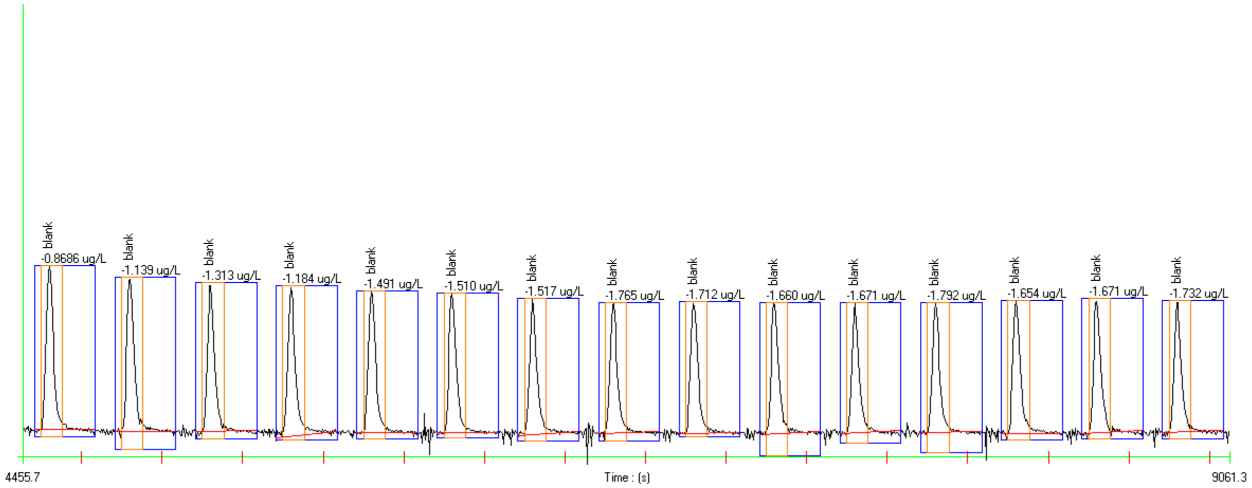
File Name: 5-21 cal support.omn
Acq. Date: 21 May 2008



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

Carryover Failed, Reporting MDL as 0.914 µg CN^-/L

File Name: 5-22 CO.omn
Acq. Date: 22 May 2008

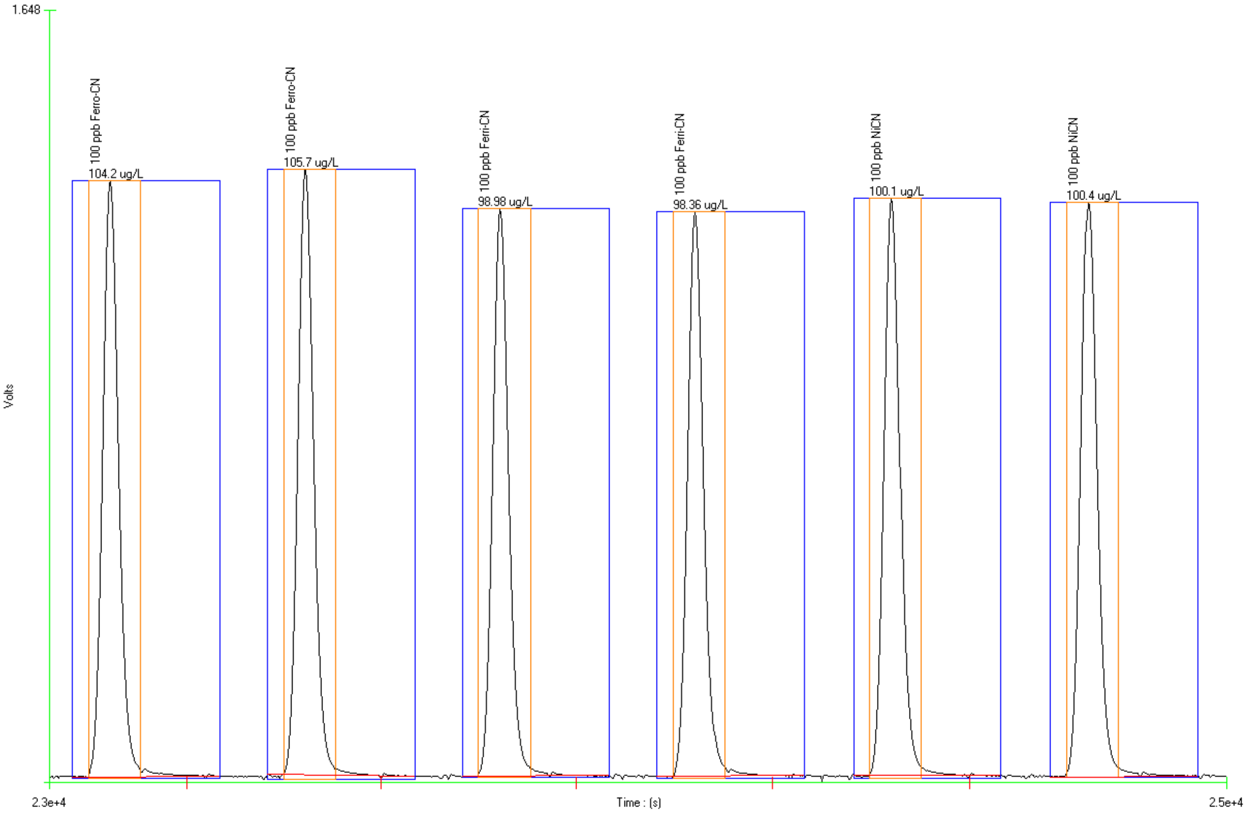


DIN Blanks

Average: -1.512 µg CN^-/L, SD = 0.270 µg CN^-/L. Calculated DIN Limits: Detection Limit = 0.811 µg CN^-/L, Decision Limit = 1.623 µg CN^-/L, Determination Limit = 2.434 µg CN^-/L;

File Name: 5-22 DIN.omn
Acq. Date: 22 May 2008

Recovery of Ferro, Ferri and Nickel cyanide complexes

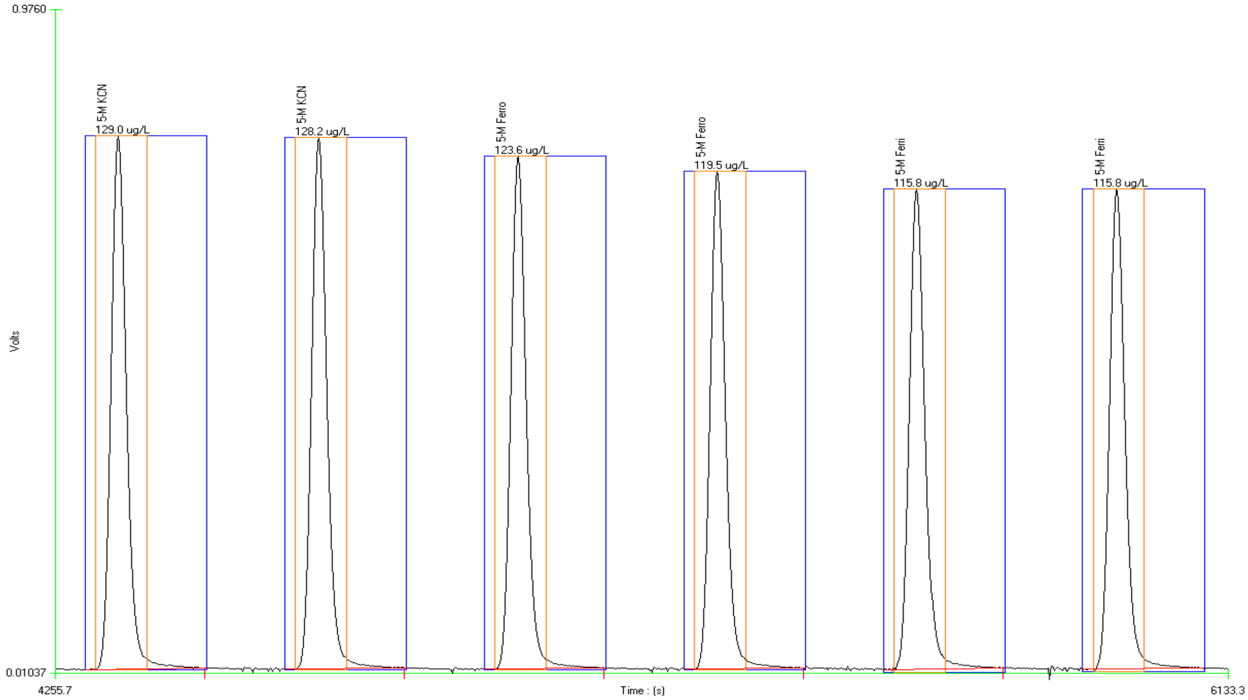


Compound	**Amount in Sample**	**Average Value Obtained**	**% Recovery** *
Ferro cyanide	100.0 µg CN-/L	104.95 µg CN^-/L	104.95
Ferri cyanide	100.0 µg CN-/L	98.67 µg CN^-/L	98.67
Nickel cyanide	100.0 µg CN-/L	100.25 µg CN^-/L	100.25

*(determined/known) * 100

Conclusion: Ferro, Ferri, and Nickel cyanide are recovered at levels greater than 98%.

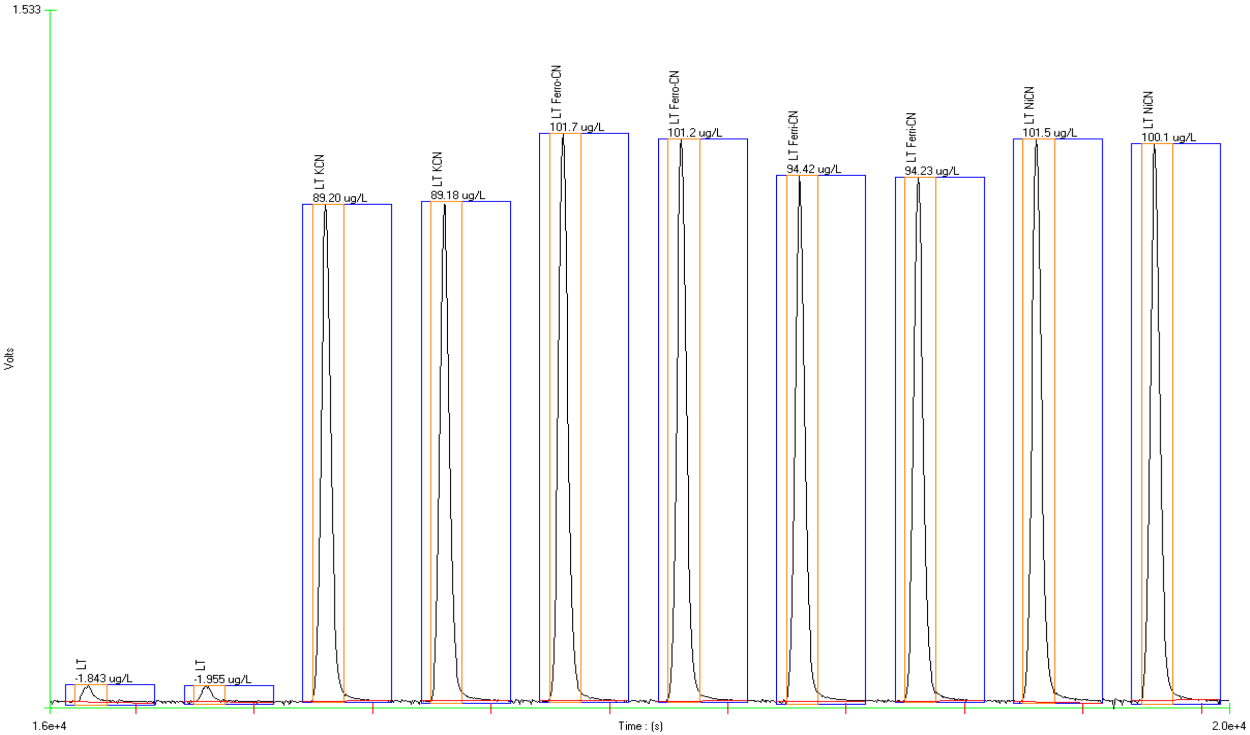
Cyanide Spike Recoveries in 5-Mile Effluent



Sample ID	**Average spike recoveries (µg CN^-/L)**	**Spike Level (µg CN^-/L)**	**% Recovery**
5-M effluent	25.92	100	---
5-M KCN	128.6	100	102.7
5-M Ferro-CN	121.6	100	95.63
5-M Ferri-CN	115.8	100	89.88

Conclusion: Potassium, Ferro, and Ferri cyanide are recovered at levels greater than 90%. Nickle cyanide only recovered at 50% in the effluent sample.

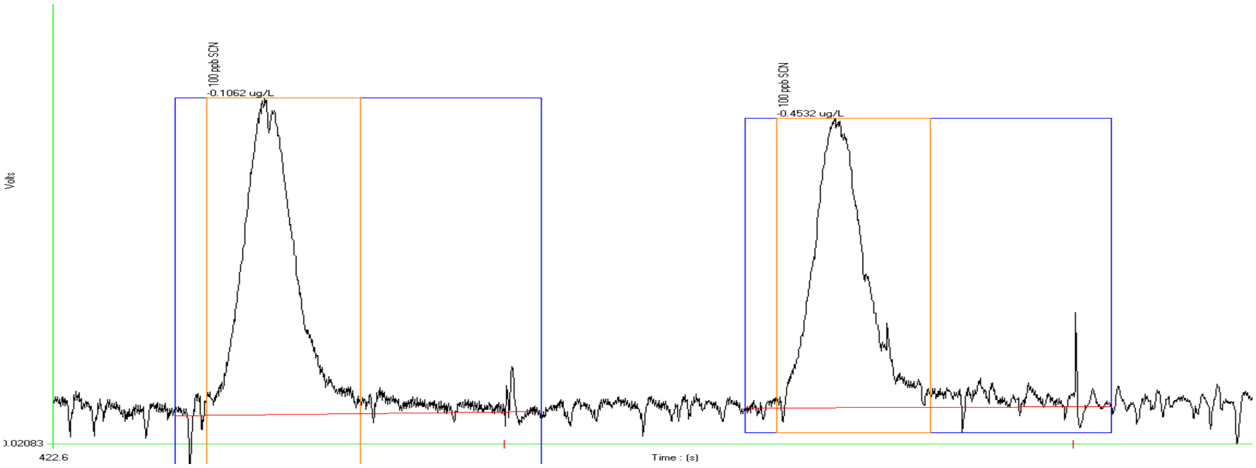
Cyanide Spike Recoveries in Loveland, CO Tap Water



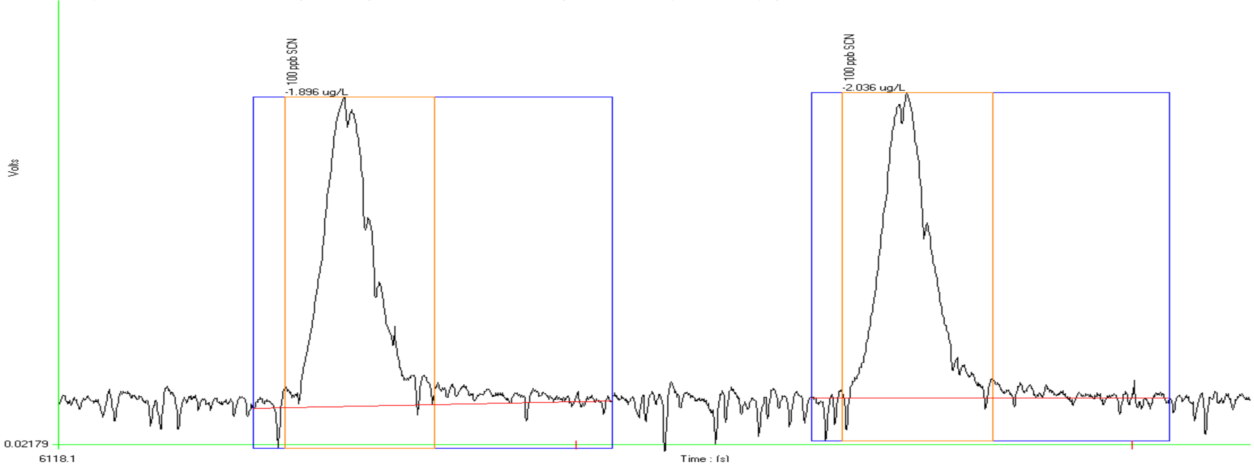
Sample ID	**Average spike recoveries (µg CN^-/L)**	**Spike Level (µg CN^-/L)**	**% Recovery**
Loveland Tap Water	-1.90	100	---
Loveland Tap Water KCN	89.19	100	91.09
Loveland Tap Water Ferro-CN	101.4	100	103.4
Loveland Tap Water Ferri-CN	94.32	100	96.22
Loveland Tap Water $K_2Ni(CN)_4$	100.8	100	102.7

Conclusion: Potassium, Ferro, Ferri, and Nickel cyanide are recovered at levels greater than 91%.

Interference due to Thiocyanate (SCN^-)



Recovery of SCN^- at the beginning of the run, average recovery –0.28 µg CN-/L (SCN^- as CN^-)



Recovery of SCN^- at the end of the run, average recovery –1.97 µg CN-/L (SCN^- as CN^-)

Conclusion: The interference from Thiocyanate is negligible, with an average of –1.12 µg CN-/L throughout the course of the analytical run. It is suggested that during the analytical run the analyst inject a 100 ppb SCN^- standard to ensure that the membrane has retained its hydrophobic nature, see section 11.3.4 for more information.

QuikChem® Method 10-204-00-5-X

Cyanide in Drinking and Wastewaters using the MICRO DIST^{TM} Distillation and Utilizing Gas Diffusion Separation and Amperometric Detection

5.0 to 400 μg 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 0.1 M sodium hydroxide absorbing solution which is diluted to 0.025 M solution during the distillation. By means of flow injection, the hydrocyanic acid (HCN) 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. Most non-volatile interferences are eliminated or minimized by the distillation procedure. Some of the known interferences are aldehydes, nitrate-nitrite, and oxidizing agents, such as chlorine, 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.
2. Thiocyanate will interfere if present. This method should not be used if sample thiocyanate concentrations exceed 0.002 mg/L. Instead, a method based on weak acid dissociable, or ligand-exchange digestion should be used.
3. Sulfides adversely affect the procedure by producing hydrogen sulfide during distillation. If a drop of the sample on lad acetate test paper indicates the presence of sulfide, treat 25 mL more than the stabilized sample ($pH \geq 12$) than that required for the cyanide determination with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate, measure the sample to be used for analysis. Avoid a large

excess of cadmium and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material.

4. Studies have shown that sulfide concentrations of up to 10 mg S^{2-}/L in the distillate can be tolerated. However, when is it expected that hydrogen sulfide will be generated from the distilled sample during the distillation, every effort should be made to analyze distillates within 2 hours from the start of distillation.

   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.

5. If exceptionally high concentrations of sulfide (>> 10 mg S^{2-}/L) are generated during distillation they could significantly bias results, violating the QC criteria in Section 9 of this method. Should this situation occur, the distillate should be treated with cadmium carbonate and filtered per Section 11.1.1, or a MICRO DIST tube containing the lead cation, as described in other approved distillation methods, should be used. When using a sulfide removal procedure, make sure to initially analyze QC samples to ensure criteria are being met with the modified procedure.

   Note: If samples contain particulate that would be removed upon filtration, the samples must be filtered prior to treatment with cadmium carbonate. The collected particulate must be saved, and the filtrate then treated using the sulfide removal procedure above. The collected particulate and treated filtrate must be recombined, homogenized, and included in the total cyanide distillation.

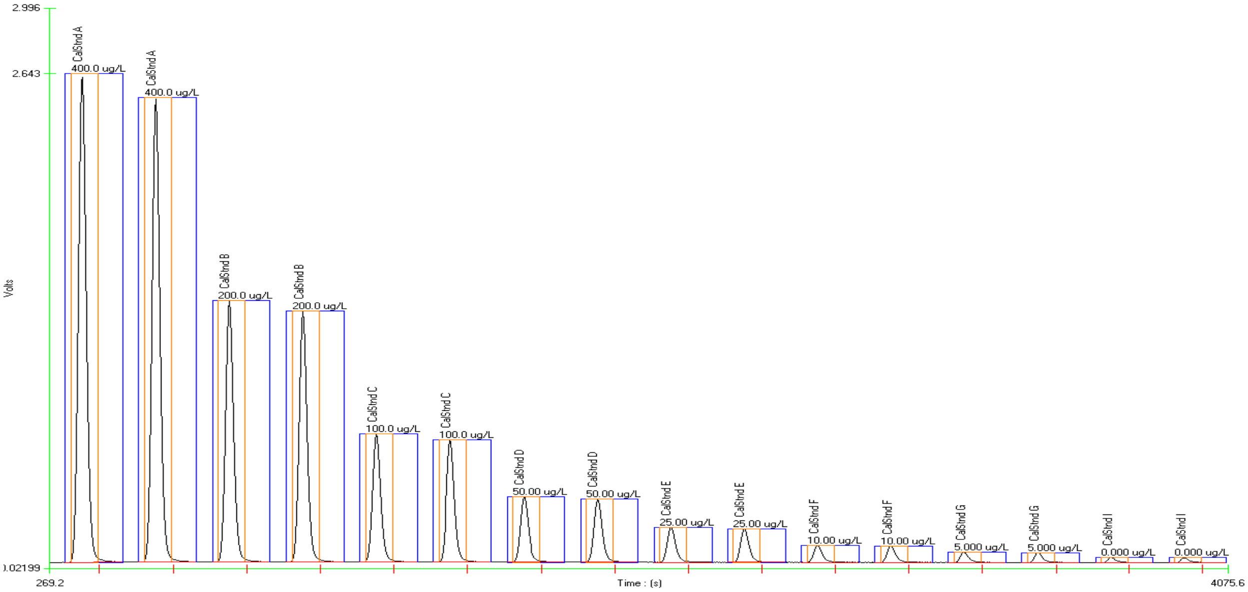
6. High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation nitrate and nitrite will form nitrous acid that will react with some organic compounds to form oximes. These oximes will decompose under test conditions to generate HCN. The interference of nitrate and nitrite is eliminated by pretreatment with sulfamic acid.

7. Oxidizing agents, such as residual chlorine, decompose most of the cyanides. Test a drop of the sample with potassium iodide (KI)-starch paper at time of collection; a blue color indicates the need for treatment. Add ascorbic acid, a few crystals (about 0.6 g each) at a time, until a drop of sample produces no color in the indicator paper; then add an additional 0.06h g of ascorbic acid for each liter of sample volume. Sodium arsenite has also been employed to remove oxidizing agents.

8. High levels of carbonate can release CO_2 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).

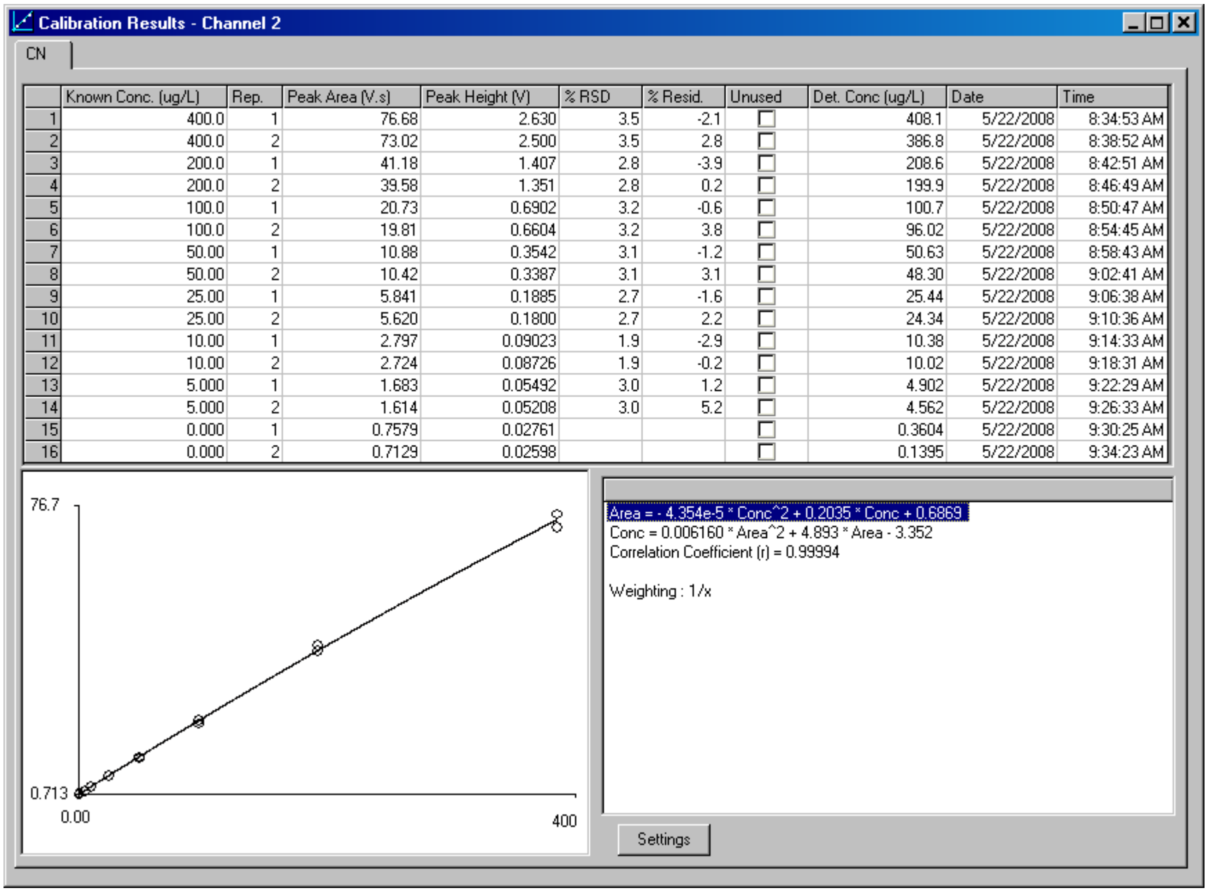
9. Method interferences may be caused by contaminants in the reagent water, reagents, and sample processing apparatus that bias response.

Calibration Data for Cyanide (MicroDist^{TM} Distilled Standards)



File Name: 5-22 Cal spikes.omn
Acq. Date: 22 May 2008

Calibration Graph and Statistics (Distilled Standards)

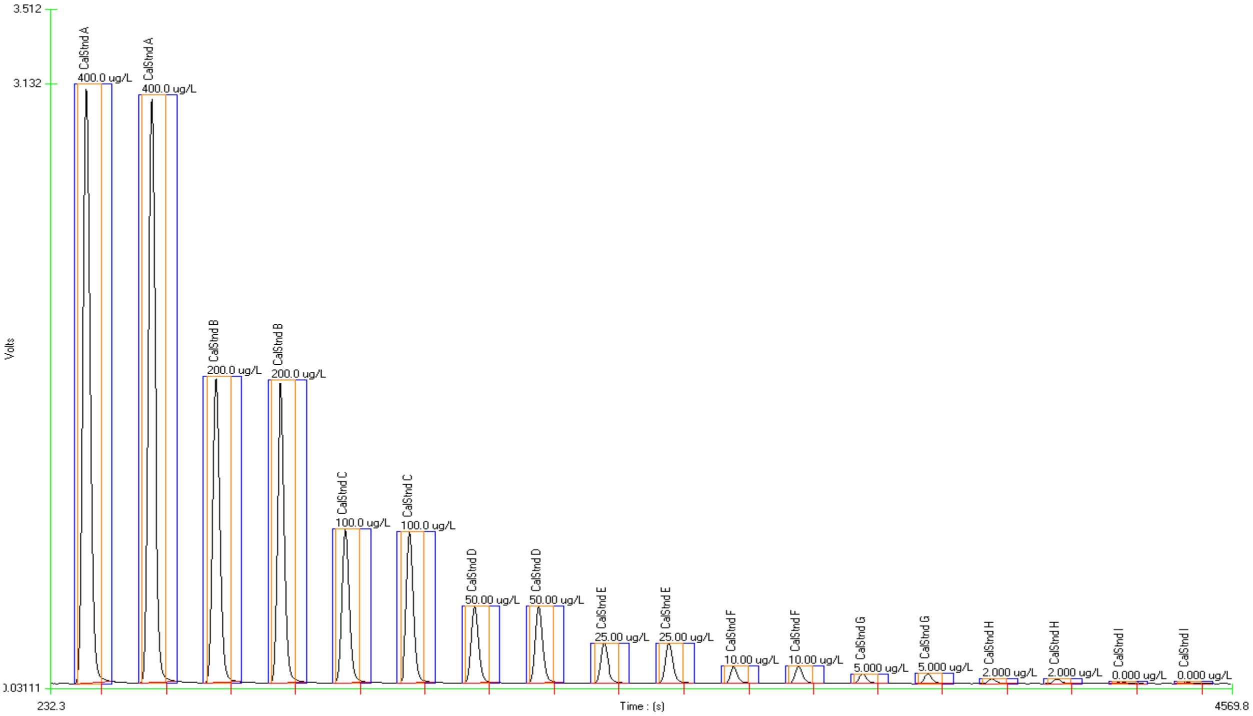


	Known Conc. (ug/L)	Rep.	Peak Area (V.s)	Peak Height (V)	% RSD	% Resid.	Unused	Det. Conc (ug/L)	Date	Time
1	400.0	1	76.68	2.630	3.5	-2.1	☐	408.1	5/22/2008	8:34:53 AM
2	400.0	2	73.02	2.500	3.5	2.8	☐	386.8	5/22/2008	8:38:52 AM
3	200.0	1	41.18	1.407	2.8	-3.9	☐	208.6	5/22/2008	8:42:51 AM
4	200.0	2	39.58	1.351	2.8	0.2	☐	199.9	5/22/2008	8:46:49 AM
5	100.0	1	20.73	0.6902	3.2	-0.6	☐	100.7	5/22/2008	8:50:47 AM
6	100.0	2	19.81	0.6604	3.2	3.8	☐	96.02	5/22/2008	8:54:45 AM
7	50.00	1	10.88	0.3542	3.1	-1.2	☐	50.63	5/22/2008	8:58:43 AM
8	50.00	2	10.42	0.3387	3.1	3.1	☐	48.30	5/22/2008	9:02:41 AM
9	25.00	1	5.841	0.1885	2.7	-1.6	☐	25.44	5/22/2008	9:06:38 AM
10	25.00	2	5.620	0.1800	2.7	2.2	☐	24.34	5/22/2008	9:10:36 AM
11	10.00	1	2.797	0.09023	1.9	-2.9	☐	10.38	5/22/2008	9:14:33 AM
12	10.00	2	2.724	0.08726	1.9	-0.2	☐	10.02	5/22/2008	9:18:31 AM
13	5.000	1	1.683	0.05492	3.0	1.2	☐	4.902	5/22/2008	9:22:29 AM
14	5.000	2	1.614	0.05208	3.0	5.2	☐	4.562	5/22/2008	9:26:33 AM
15	0.000	1	0.7579	0.02761			☐	0.3604	5/22/2008	9:30:25 AM
16	0.000	2	0.7129	0.02598			☐	0.1395	5/22/2008	9:34:23 AM

Area = - 4.354e-5 * Conc^2 + 0.2035 * Conc + 0.6869
Conc = 0.006160 * Area^2 + 4.893 * Area - 3.352
Correlation Coefficient (r) = 0.99994

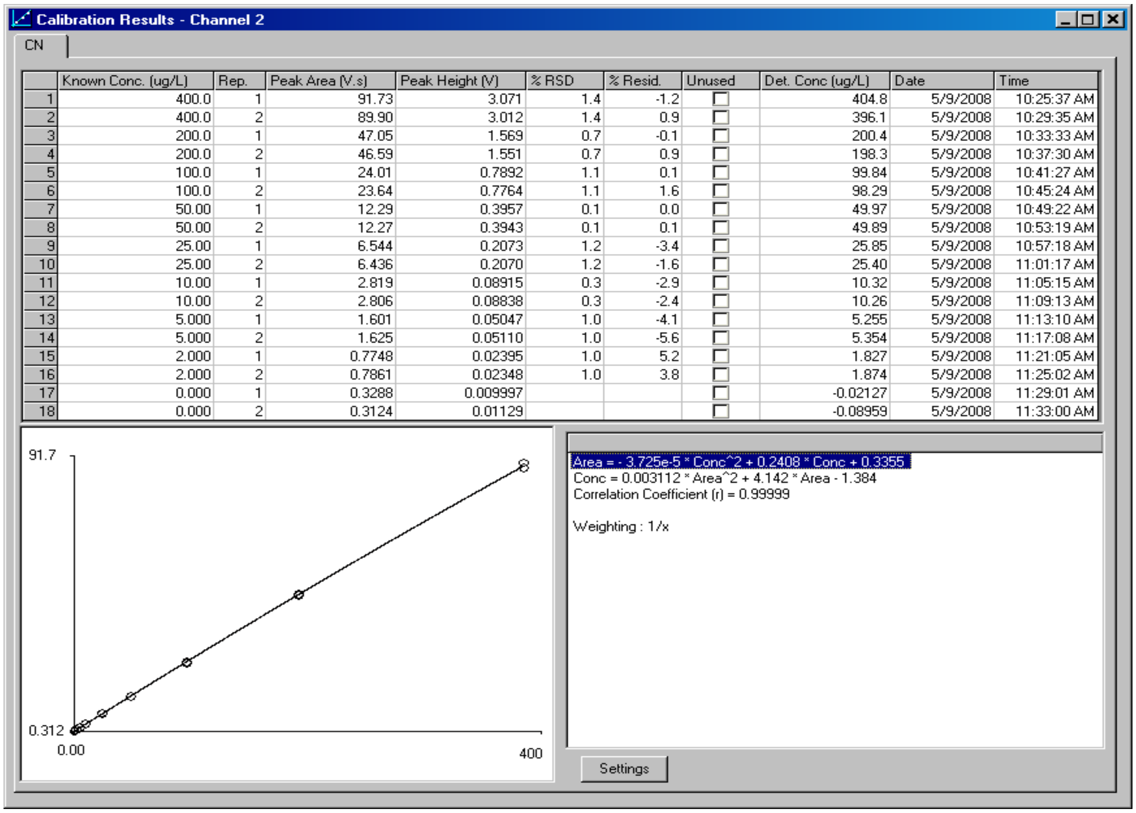
Weighting : 1/x

Calibration Data for Cyanide (Non-Distilled Standards)



File Name: 5-9 cal support no dist.OMN
Acq. Date: 9 May 2008

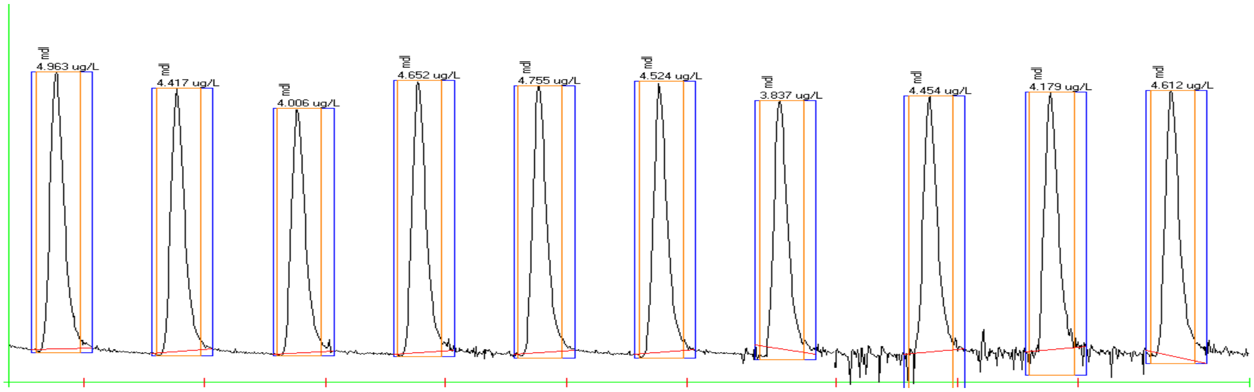
Calibration Graph and Statistics (Non-Distilled Standards)



	Known Conc. (ug/L)	Rep.	Peak Area (V.s)	Peak Height (V)	% RSD	% Resid.	Unused	Det. Conc (ug/L)	Date	Time
1	400.0	1	91.73	3.071	1.4	-1.2	☐	404.8	5/9/2008	10:25:37 AM
2	400.0	2	89.90	3.012	1.4	0.9	☐	396.1	5/9/2008	10:29:35 AM
3	200.0	1	47.05	1.569	0.7	-0.1	☐	200.4	5/9/2008	10:33:33 AM
4	200.0	2	46.59	1.551	0.7	0.9	☐	198.3	5/9/2008	10:37:30 AM
5	100.0	1	24.01	0.7892	1.1	0.1	☐	99.84	5/9/2008	10:41:27 AM
6	100.0	2	23.64	0.7764	1.1	1.6	☐	98.29	5/9/2008	10:45:24 AM
7	50.00	1	12.29	0.3957	0.1	0.0	☐	49.97	5/9/2008	10:49:22 AM
8	50.00	2	12.27	0.3943	0.1	0.1	☐	49.89	5/9/2008	10:53:19 AM
9	25.00	1	6.544	0.2073	1.2	-3.4	☐	25.85	5/9/2008	10:57:18 AM
10	25.00	2	6.436	0.2070	1.2	-1.6	☐	25.40	5/9/2008	11:01:17 AM
11	10.00	1	2.819	0.08915	0.3	-2.9	☐	10.32	5/9/2008	11:05:15 AM
12	10.00	2	2.806	0.08838	0.3	-2.4	☐	10.26	5/9/2008	11:09:13 AM
13	5.000	1	1.601	0.05047	1.0	-4.1	☐	5.255	5/9/2008	11:13:10 AM
14	5.000	2	1.625	0.05110	1.0	-5.6	☐	5.354	5/9/2008	11:17:08 AM
15	2.000	1	0.7748	0.02395	1.0	5.2	☐	1.827	5/9/2008	11:21:05 AM
16	2.000	2	0.7861	0.02348	1.0	3.8	☐	1.874	5/9/2008	11:25:02 AM
17	0.000	1	0.3288	0.009997			☐	-0.02127	5/9/2008	11:29:01 AM
18	0.000	2	0.3124	0.01129			☐	-0.08959	5/9/2008	11:33:00 AM

Area = - 3.725e-5 * Conc^2 + 0.2408 * Conc + 0.3355
Conc = 0.003112 * Area^2 + 4.142 * Area - 1.384
Correlation Coefficient (r) = 0.99999

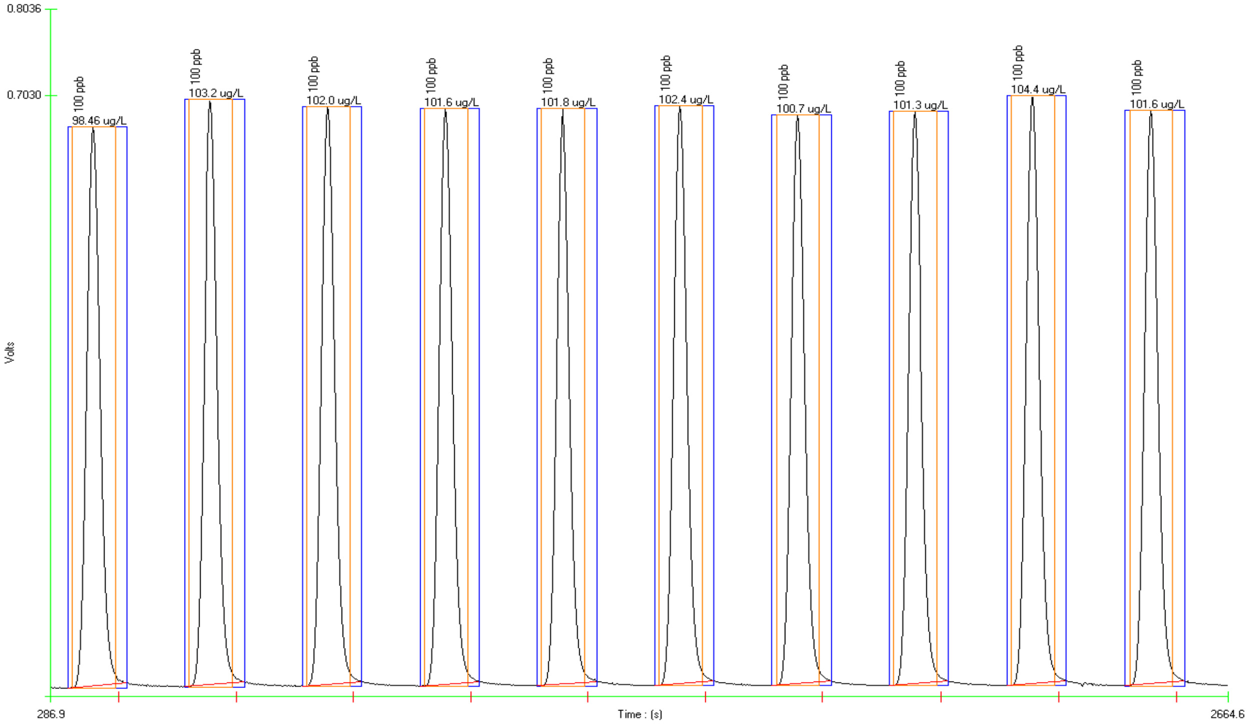
Weighting : 1/x



Method Detection Limit for cyanide using a 4.0 µg CN^-/L standard **(Distilled Standards)**

MDL= 0.975 µg CN^-/L

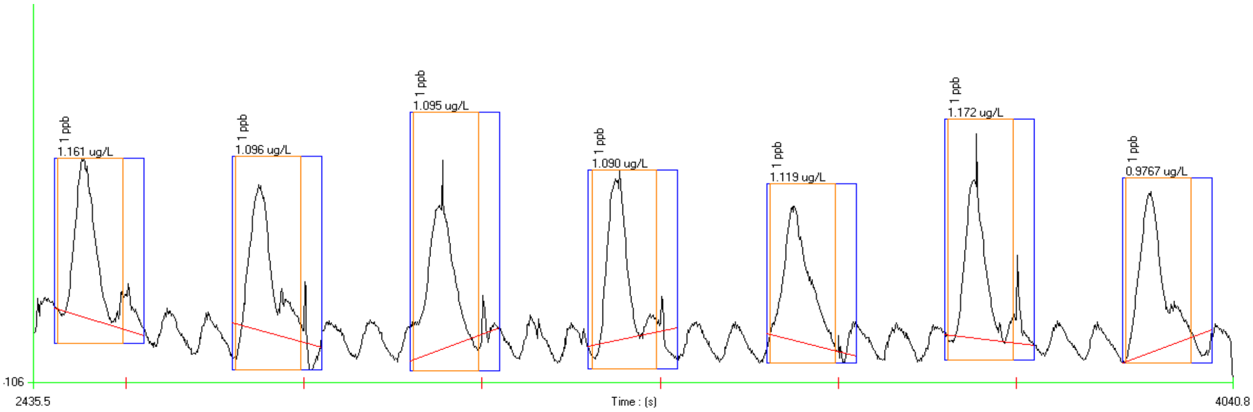
Standard Deviation (s) = 0.346 µg CN^-/L, Mean (x) = 4.44 µg CN^-/L, Known value = 4.0 µg CN^-/L
File Name: 5-22 Prec mdl.omn
Acq. Date: 22 May 2008



Precision data for cyanide using a 100 µg CN^-/L standard **(Distilled Standards)**

% RSD = 1.53 %

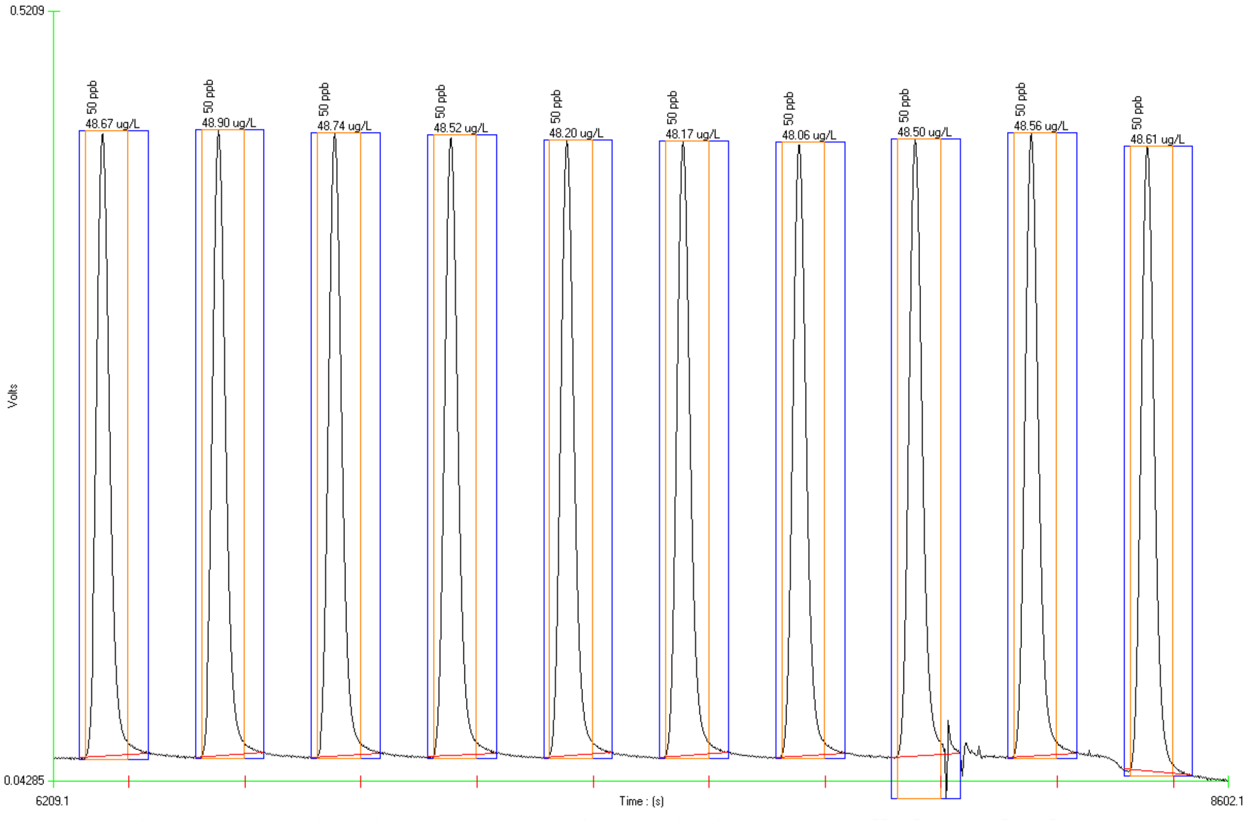
Standard Deviation (s) = 1.56 µg CN^-/L, Mean (x) = 101.75 µg CN^-/L, Known value = 100 µg CN^-/L
File Name: 5-22 Prec mdl.omn
Acq. Date: 22 May 2008



Method Detection Limit for cyanide using a 1.0 µg CN$^-$/L standard **(Non-Distilled Standards)**

MDL= 0.20 µg CN$^-$/L

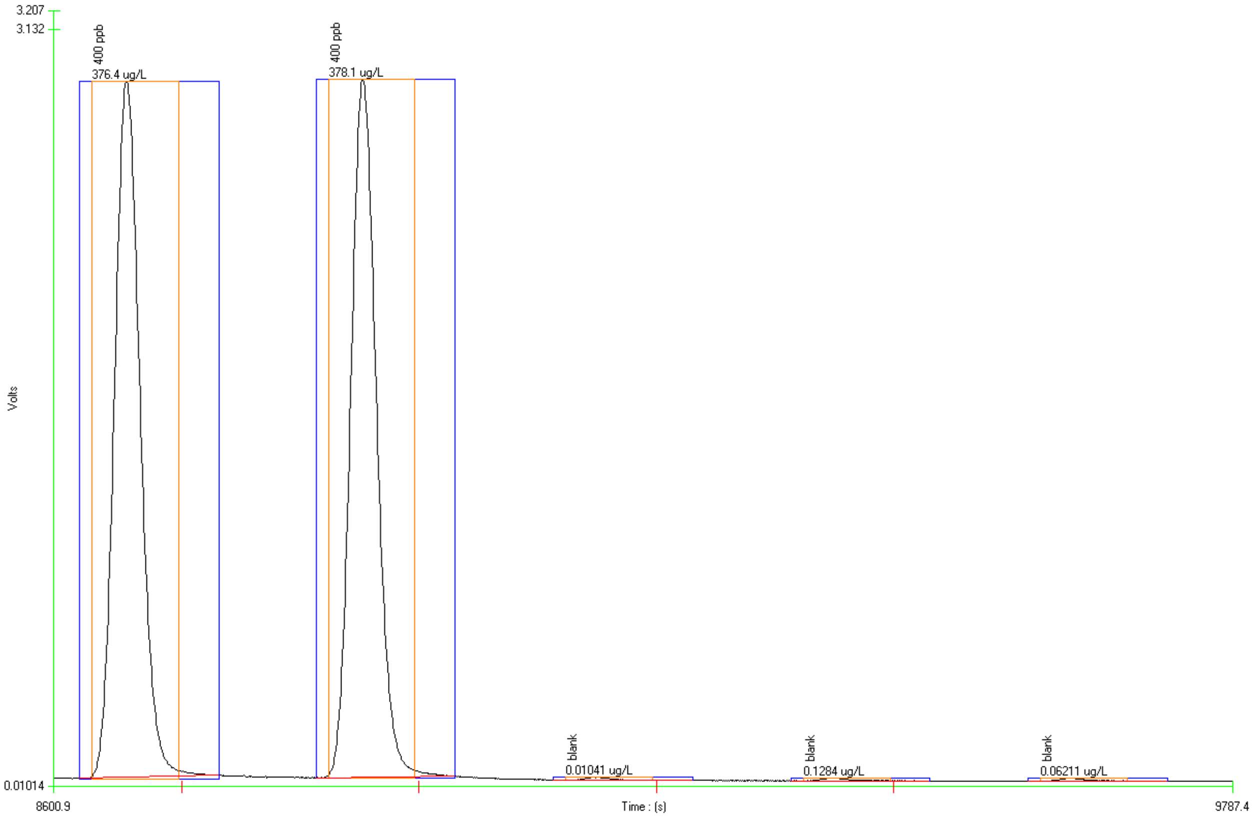
Standard Deviation (s) = 0.064 µg CN$^-$/L, Mean (x) = 1.10 µg CN$^-$/L, Known value = 1.0 µg CN$^-$/L
File Name: 5-8 cal mdl no dist.OMN
Acq. Date: 8 May 2008



Precision data for cyanide using a 50 µg CN$^-$/L standard **(Non-Distilled Standards)**

% RSD = 0.56 %

Standard Deviation (s) = 0.27 µg CN$^-$/L, Mean (x) = 48.49 µg CN$^-$/L, Known value = 50 µg CN$^-$/L
File Name: 5-9 cal support no dist.OMN
Acq. Date: 9 May 2008

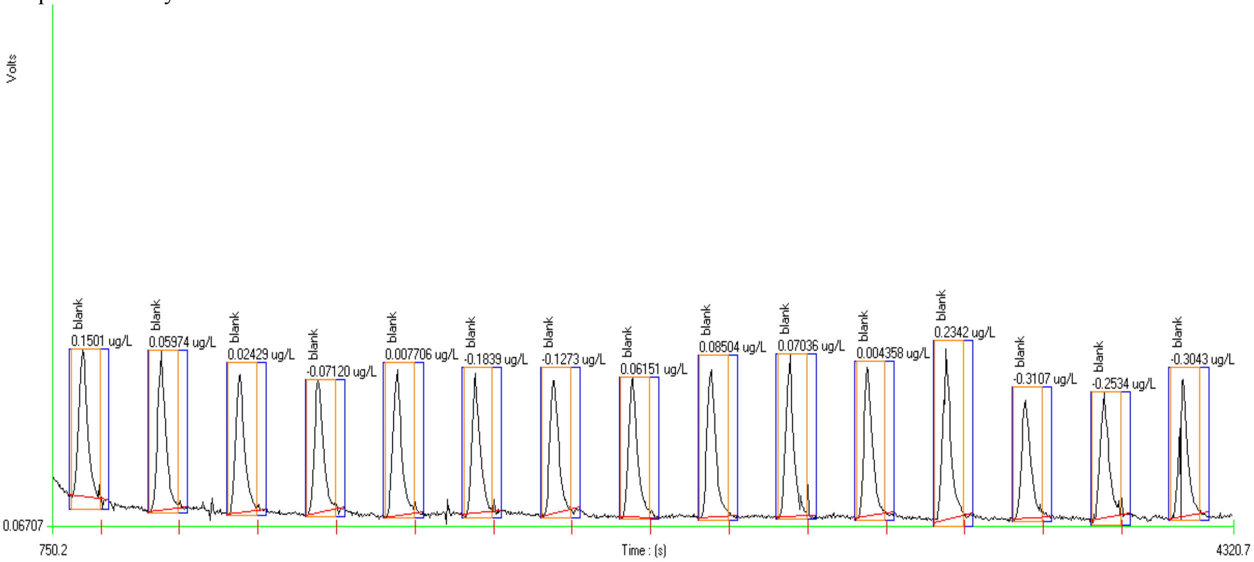


Carryover Study: 400 µg CN$^-$/L standard followed by 3 blanks (Non-Distilled Standards)

Carryover Passed

File Name: 5-9 cal support no dist.OMN

Acq. Date: 9 May 2008



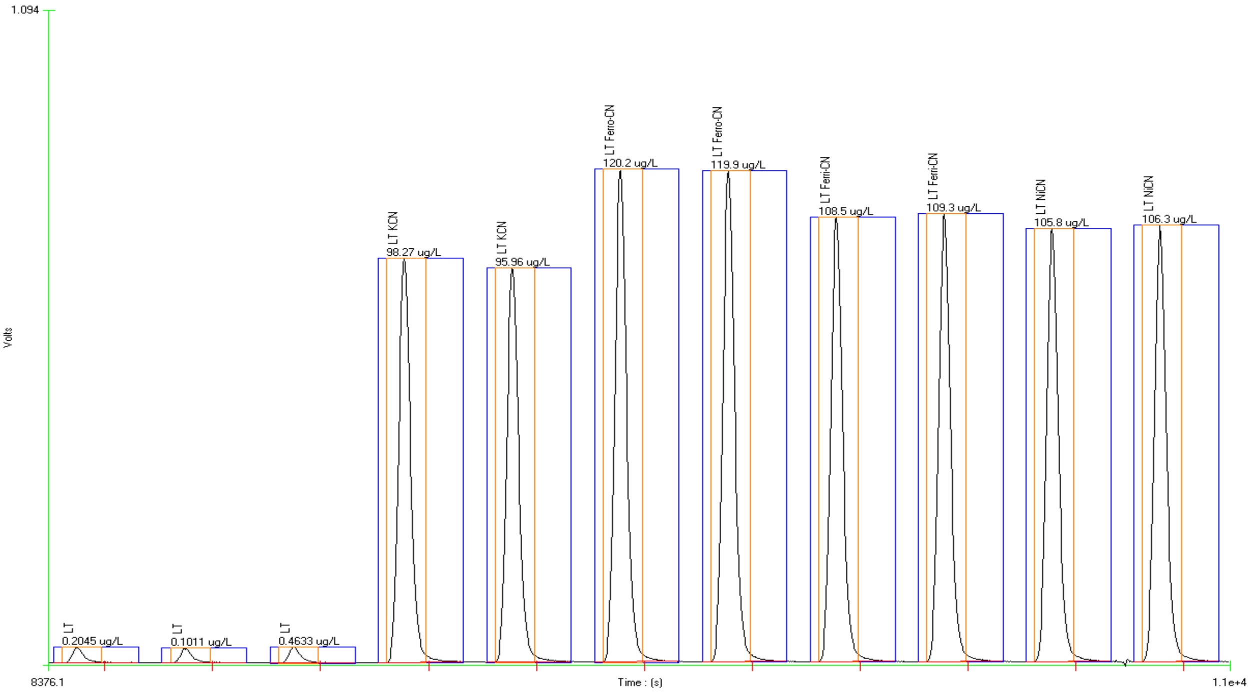
DIN Blanks (Non-Distilled Standards)

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

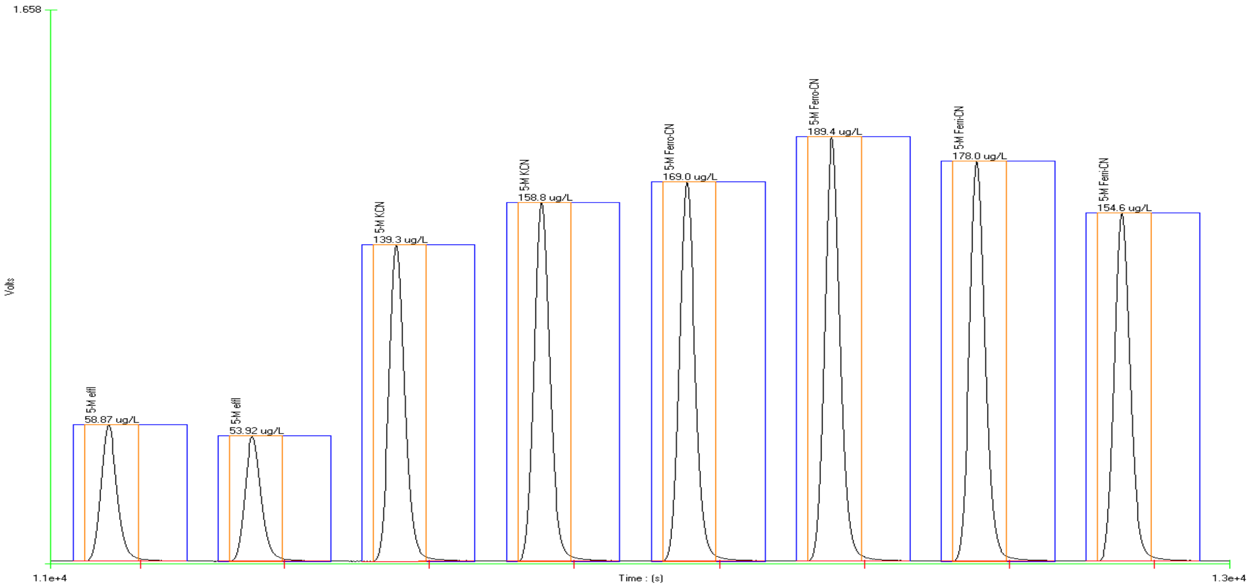
Cyanide Spike Recoveries in Loveland, CO Tap Water

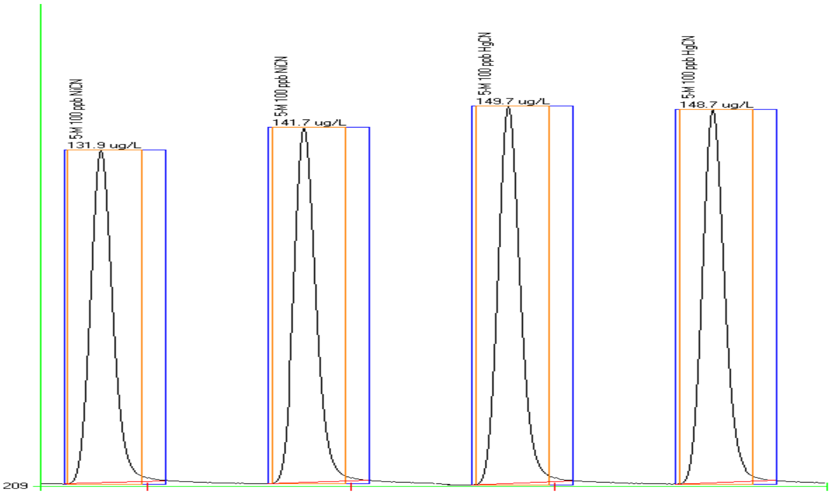


Sample ID	**Average spike recoveries (µg CN^-/L)**	**Spike Level (µg CN^-/L)**	**% Recovery**
Loveland Tap Water	0.256	100	---
Loveland Tap Water KCN	97.12	100	96.86
Loveland Tap Water Ferro-CN	120.1	100	119.8
Loveland Tap Water Ferri-CN	108.9	100	108.6
Loveland Tap Water $K_2Ni(CN)_4$	106.1	100	105.8

Conclusion: Potassium, Ferro, Ferri and Nickel cyanide are recovered at levels greater than 96%.

Cyanide Spike Recoveries in 5-Mile Effluent

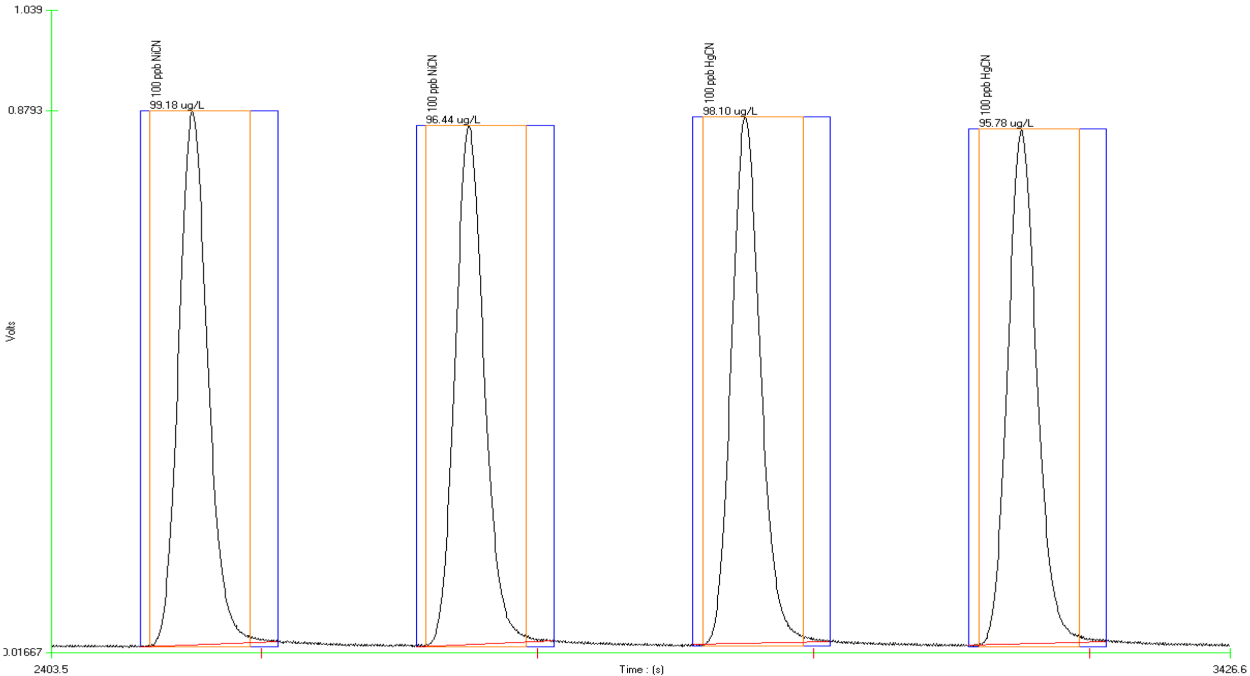




Sample ID	Average spike recoveries (μg CN^-/L)	Spike Level (μg CN^-/L)	% Recovery
5-M effluent	56.40	---	---
5-M KCN	149.1	100	92.66
5-M Ferro-CN	179.2	100	122.8
5-M Ferri-CN	166.3	100	109.9
5-M $K_2Ni(CN)_4$	136.8	100	80.41
5-M $Hg(CN)_2$	149.2	100	92.81

Conclusion: Potassium, Ferro, Ferri, Nickel and Mercury cyanide are recovered at levels greater than 80%.

Recovery of Nickel cyanide and Mercury cyanide complexes



Compound	Amount in Sample	Average Value Obtained	% Recovery *
Nickel cyanide	100.0 µg CN-/L	97.81 µg CN^-/L	97.81
Mercury cyanide	100.0 µg CN-/L	96.94 µg CN^-/L	96.84

*(determined/known) * 100

Conclusion: Nickel and Mercury cyanide are recovered at levels greater than 96%.