

# *Data Pack*

## *MicroDIST*

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*June 2008*

## Introduction

MICRODIST® is a patented technology, developed by Lachat Instruments to improve the efficiency of sample preparation through distillation. MICRODIST® offers tremendous time and cost savings versus traditional MIDI distillation techniques. Additionally, MICRODIST® methods for Cyanide are USEPA approved for both NPDES and NPDWR. Other MICRODIST® parameters are equivalent for NPDES under the 2007 USEPA Method Update Rule.

This Data Pack contains application information on Lachat methods utilizing MICRODIST® distillation technology. Information on several methods for use with QuikChem® FIA instruments is included. Other concentration ranges for these analytes are also available. Additionally, Lachat Applications is continuously developing new methods, so contact Lachat for the most up-to-date Method's List.

This introduction gives an overview of Lachat's MICRODIST® methods and basic MICRODIST® procedure. More detailed information, including method support data, can be found in the remainder of this Data Pack.

**Table 1: MICRODIST® Method Summary**

MICRODIST Parameter	Matrix and Chemistry	Pre-Fill (21pk)	User-Fill (10pk)	User-Fill (50pk)	User-Fill (100pk)	Related Lachat QuikChem Method	EPA Approved (NPDES & NPDWR)	EPA Equivalent (NPDES)	Range	Method Detection Limit	Sample Throughput
<b>Cyanide-1</b>	Waters, solids, strong acid dissociable (SAD), total cyanide	A17001	A17017	A17517	A17117	10-204-00-1-X	335.4	NA	0.005 to 0.5 mg CN <sup>-</sup> /L	0.001 mg CN <sup>-</sup> /L	80 samples per hour
						10-204-00-1-X(2)	NA	335.4	0.002 to 0.5 mg CN <sup>-</sup> /L	0.38 µg CN <sup>-</sup> /L	144 samples per hour
<b>Cyanide-2</b>	Caustic extracts, SAD, total cyanide	A17001	A17017	A17517	A17117	10-204-00-1-W	NA	NA	0.5 to 100 mg CN <sup>-</sup> /L		120 samples per hour
<b>Cyanide-3</b>	Waters, solids, weak acid dissociable (WAD)	A17001	A17017	A17517	A17117	10-204-00-1-W	NA	NA	0.5 to 100 mg CN <sup>-</sup> /L		120 samples per hour
<b>Cyanide-5</b>	Waters, solids, SAD (total cyanide) in the presence of sulfide	A17011	A17017	A17517	A17117	10-204-00-1-X	335.4	NA	0.005 to 0.5 mg CN <sup>-</sup> /L	0.001 mg CN <sup>-</sup> /L	80 samples per hour
<b>Phenolics-1</b>	Waters, solids, 4-AATP	A17002	A17017	A17517	A17117	10-210-00-1-X	NA	420.1	0.005 to 0.2 mg phenol/L	0.0034 mg phenol/L	90 samples per hour
<b>Sulfide-1</b>	Acid Soluble Sulfides, waters, iodometric determination	A17003	A17017	A17517	A17117	none	NA	NA	Iodometric determination		
<b>Sulfide-2</b>	Waters/MTB colorimetric determination	A17001	A17017	A17517	A17117	10-116-29-1-A	NA	NA	0.02 to 2.0 mg S/L	0.005 mg S/L	72 samples per hour
<b>Ammonia-1</b>	Waters, phenate colorimetric or ISE determination	NA	A17017	A17517	A17117	10-107-06-1-X	NA	350.1	0.05 to 20.0 mg N/L	0.007 mg N/L	60 samples per hour
<b>Ammonia-2</b>	Waters, solids, nesslerization	NA	A17017	A17517	A17117	none	NA	NA	Nesslerization method		
<b>Tritium-1</b>	Contaminated waters, solids	A17014 (pkg of 10)	NA	NA	NA	none	NA	NA	Scintillation process		

\*Please be sure to check with local regulators to be sure these methods are in compliance with local regulations!

## General Overview of the use of MICRODIST<sup>®</sup>

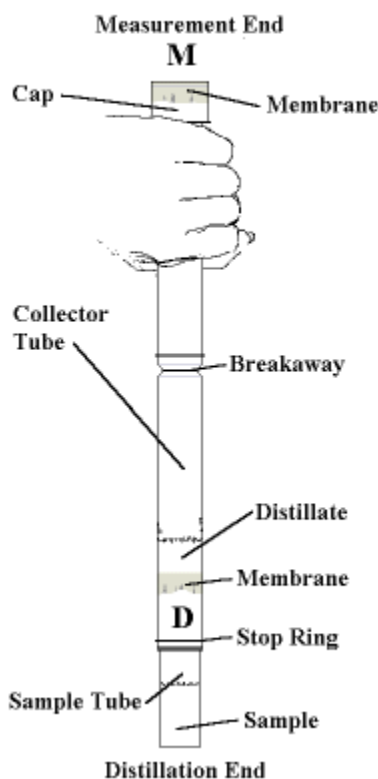


Figure 1: MICRODIST<sup>®</sup> tube.

**NOTE:** In the following procedure, **D** and **M** refer to the marks on the ends of the collector tube. **D** references the 'distillation' or 'discarded' end, while **M** references 'measuring' end. See [Figure 1](#).

### **General Procedure:**

1. Set the controller to the temperature specified in the method. Allow the heater block to warm up. This will take about 30 minutes. With the **m** end up, put collector tubes for the samples into the collector tube rack. Put the sample tubes into the sample tube rack (up to 21 for one block).
2. Pipet 6.0 mL of aqueous sample into each sample tube with an automatic pipet or weigh 6.0 grams of sample on a balance ([Figure 2](#)). When distilling solids, contaminated soils or sludge, place 0.5 g of sample, then 5.5 mL of deionized water into the sample tube.

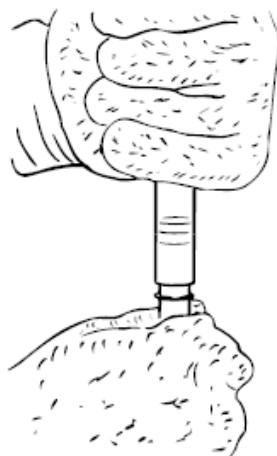


**Figure 2:** Placing samples in tubes.

In order to release the analyte from the matrix, it may be necessary to add a releasing solution. The releasing solution is added using a re-pipetor at a volume that is specified in the MICRODIST® method

3. Immediately push the  $\text{D}$  end of the collector tubes over the open end of each sample tube to start the seal. Place the assembly in the press, putting the sample tube through the hole in the white base. Before pressing, the user should grip the collector tube firmly at the breakaway point to keep the tube from shifting during the pressing procedure.
4. Put on the heat-resistant gloves. Push the sample tube and  $\text{D}$  end of each tube all the way into the preheated block so that the collector tube stop-ring touches the block. Placing 21 tubes should take less than one minute. Set the timer for the specified time in the method.
5. When time is up, put on the heat-resistant gloves and remove the first tube from the block. **Immediately** pull off the sample tube using a downward, twisting motion ([Figure 3](#)) as opposed to a sideways motion. The sample tube must be removed from the collector tube within 4 seconds of removal from the block or a backwards vacuum of the sample may occur – causing low recoveries. Dispose of the sample tube and any remaining solution.

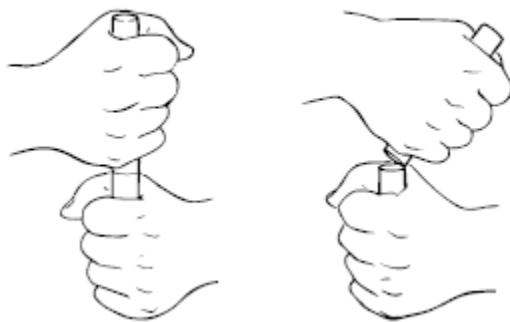
Place each collector tube into the collector tube rack, with the  $\text{D}$  end up. It should take less than two minutes to pull and separate all 21 tubes. Allow the tubes to cool for at least 10 minutes.



**Figure 3:** Separating the Collector and Sample tubes

6. With the **D** end still up, “break” the collector tube in half by pulling the **D** end hard towards the user to break it, then twisting and tearing off the **D** end (Figure 4). (Prevent lost droplets by holding the **M** end on a firm surface and cracking off the **D** end.) Discard the **D** end.

**NOTE:** Parafilm both ends of the remaining **M** portion of the tube if analysis will not occur immediately after “breaking” the tube.



**Figure 4:** “Breaking” the Collector Tube

7. In the remaining **m** portion of the collector tube, dilute to the 6.0 mL mark with deionized water. This results in the original sample volume. Analyze the solution in the tube with the corresponding Lachat QuikChem Method. There is enough distillate for two 25-second sample periods when an FIA method is used. Seal both ends of the tube with Parafilm if the sample will not be analyzed immediately.

**QuikChem<sup>®</sup> Method 10-107-06-1-X**

# Ammonia in Waters and Wastewaters

**0.05 to 20.0 mg N/L as NH<sub>3</sub>**

**– Principle –**

Samples are predistilled using the Lachat MICRO DIST<sup>®</sup> apparatus. Ammonia is then determined on the distillate. This method is based on the Berthelot reaction where ammonia reacts with alkaline phenol and sodium hypochlorite to form indophenol blue. Sodium nitroprusside (nitroferricyanide) is added to enhance sensitivity. The absorbance of the reaction product measured at 630 nm is directly proportional to the original ammonia concentration.

**– Interferences –**

1. Calcium and magnesium ions may precipitate if present in sufficient concentration. EDTA is added to the sample in-line to prevent this problem.
2. Color, turbidity and certain organic species may interfere. Turbidity is removed by manual filtration. Sample color may be corrected for by running the samples through the manifold without color formation.

**– Special Apparatus –**

Please see Parts and Price list for Ordering Information

1. Heating Unit Lachat Part No. A85X00 (X=1 for 110V, X=2 for 220V)
2. MICRO DIST<sup>®</sup>, Lachat Part No. A17X02 (X=1 for 110V, X=2 for 220V)
3. MICRO DIST<sup>®</sup>, tubes for Ammonia

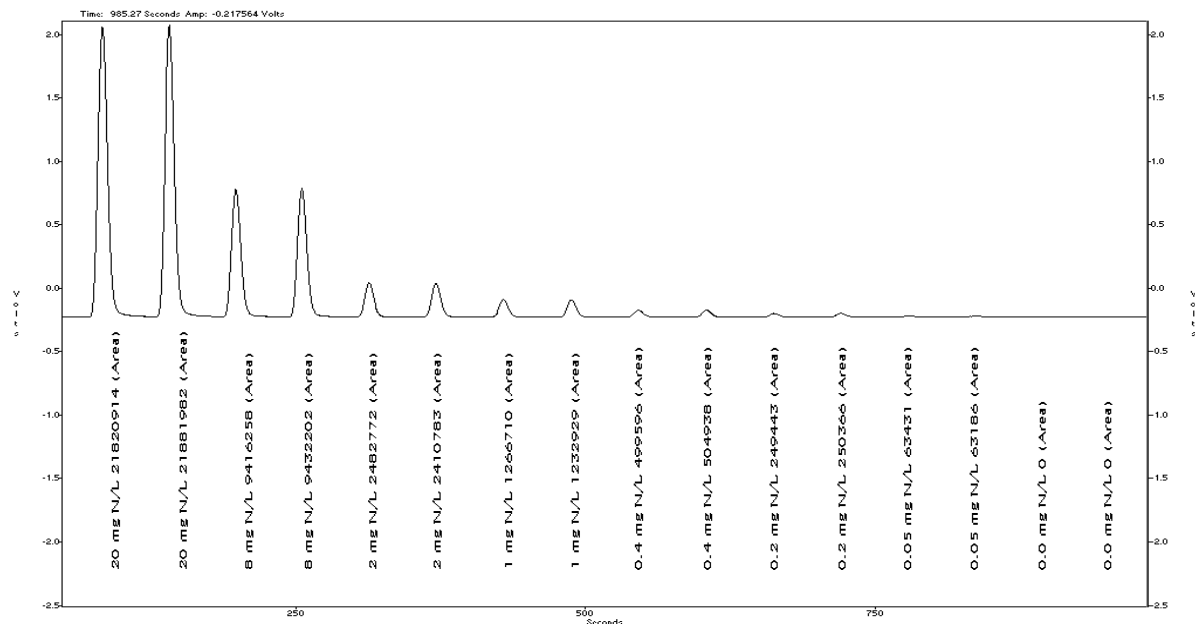
Lachat part no.	Quantity/pack	Type
A17017A	10	User fill option
A17517A	50	User fill option
A17117A	100	User fill option

4. PVC PUMP TUBES MUST BE USED FOR THIS METHOD

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Revised by K. Bogren on August 2003. Written and copyrighted © by A. Huberty 28 May 1996 by **Lachat Instruments, 5600 Lindbergh Drive, Loveland Colorado 80539 USA**. Phone: 970-663-1377 FAX: 970-962-6710. This document is the property of Lachat Instruments. Unauthorized copying of this document is prohibited.

## Calibration Data for Ammonia

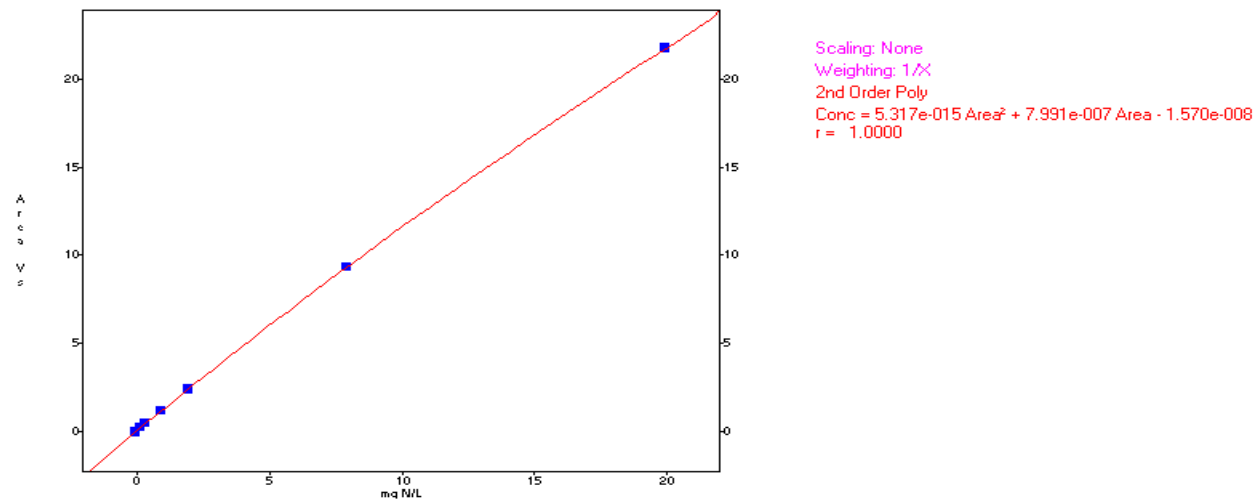


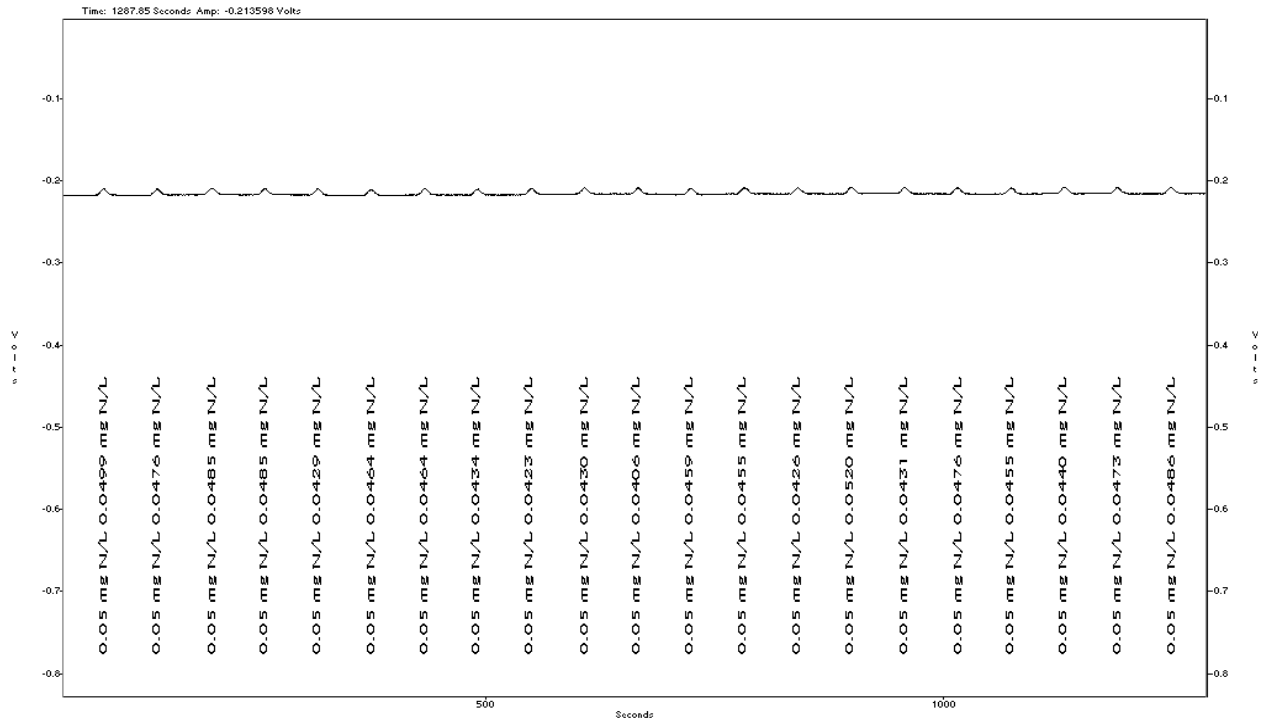
Method file name: 042600c1.fdt

Acq. Date: 26 April 2000

## Calibration Graph and Statistics

Level	Average	mg/L	Determined	Replicate %RSD	% residual
1	21851448	20.0	20.0	0.2	0.0
2	9424230	8.00	8.0	0.1	-0.0
3	2446778	2.00	1.988	2.1	0.6
4	1249820	1.00	0.999	1.9	-0.7
5	502267	0.40	0.397	0.8	-0.7
6	249905	0.20	0.20	0.3	-0.0
7	63309	0.05	0.049	0.3	-1.2
8	0	0.00	0.00	0.00	---



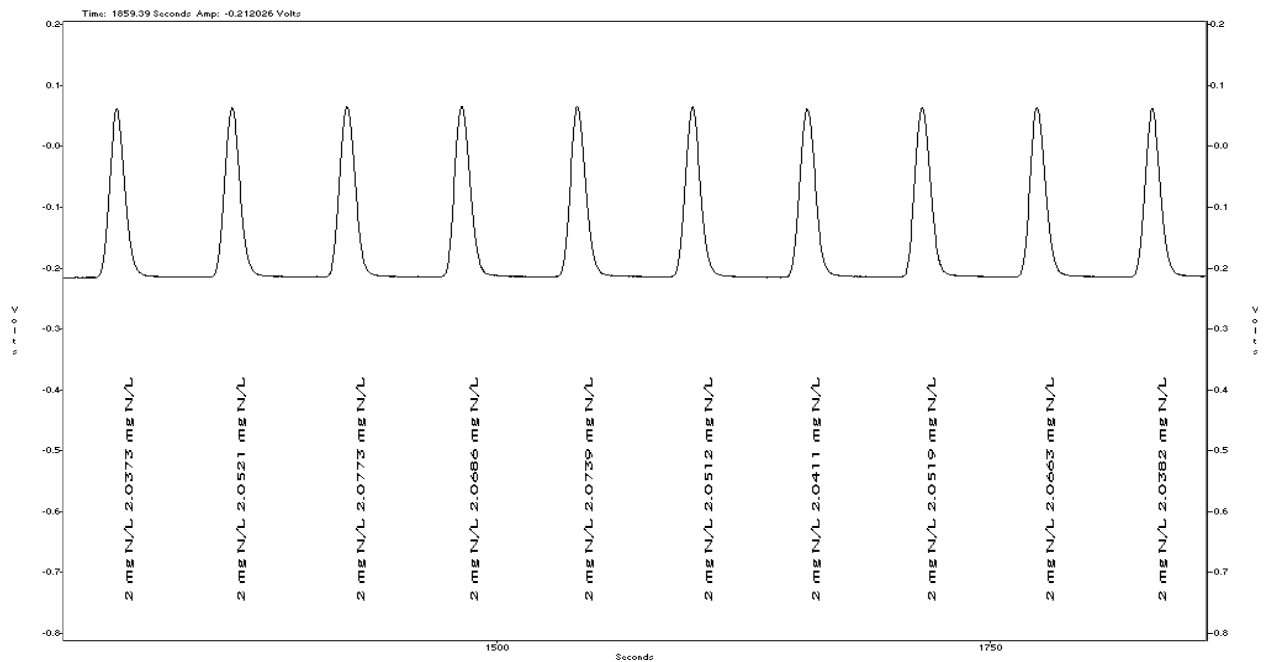


**Method Detection Limit for ammonia, using a 0.05 mg N/L Standard**

**MDL = 0.007 mg N/L**

Standard Deviation (s) = 0.0029 mg N/L Mean (X) = 0.0458 mg N/L, Known Value = 0.05 mg N/L

ACQ. Date: 26 April 2000



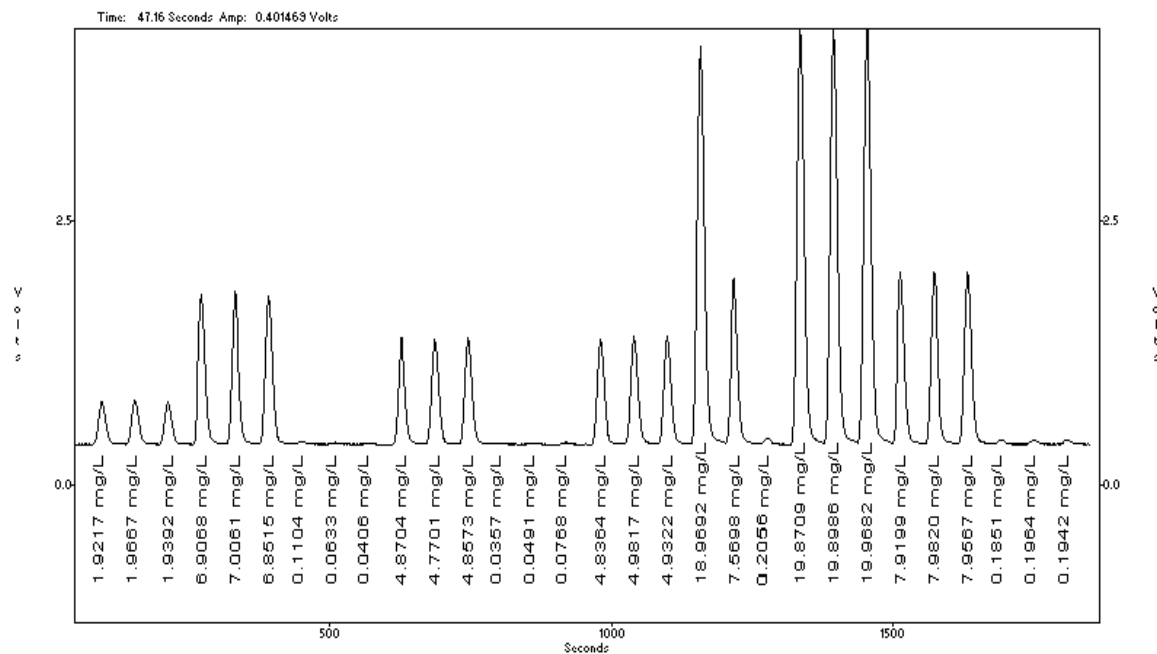
**Precision data for ammonia, using a 2.0 mg N/L Standard**

**%RSD = 0.72%**

Standard Deviation (s) = 0.0148 mg N/L, Mean (X) = 2.0558 mg N/L, Known Value = 2.0 mg N/L

ACQ. Date: 26 April 2000

### Spike Recoveries for Method 10-107-06-1-X



Recoveries of ammonia in inorganic soil, Lake Michigan surface water, and New Berlin (WI) well water  
 Data Filename: 960329s2.fdt  
 Acquisition time: 29 March 1996, 14:47:04

Cup Numbers	Sample Type	Average (mg/L)	Spike Level (mg/L)	Recovery
1-3	inorganic soil	1.9425	---	---
4-6	inorganic soil - spiked	6.9212	5	99.6%
7-9	Lake Michigan	0.0714	---	---
10-12	Lake Michigan - spiked	4.8326	5	95.2%
13-15	New Berlin well	0.0539	---	---
16-18	New Berlin well - spiked	4.9168	5	97.3%

### Recoveries of ammonia in distilled samples compared to undistilled samples

Cup Number	Known Value (mg/L)	Determined Average Concentration (mg/L)	Recovery
19	20 distilled	18.9692	95.3%
22 (3 reps)	20 undistilled	19.9126	
20	8 distilled	7.5698	95.2%
23 (3 reps)	8 undistilled	7.9529	
21	0.2 distilled	0.2056	107.4%
24 (3 reps)	0.2 undistilled	0.1919	

**QuikChem® Method 10-204-00-1-X**

**Cyanide in drinking and wastewaters using  
MICRODIST®**

**0.005 to 0.5 mg CN<sup>-</sup>/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. See method for information on interference handling.

**– Special Apparatus –**

Please see Parts and Price list for Ordering Information

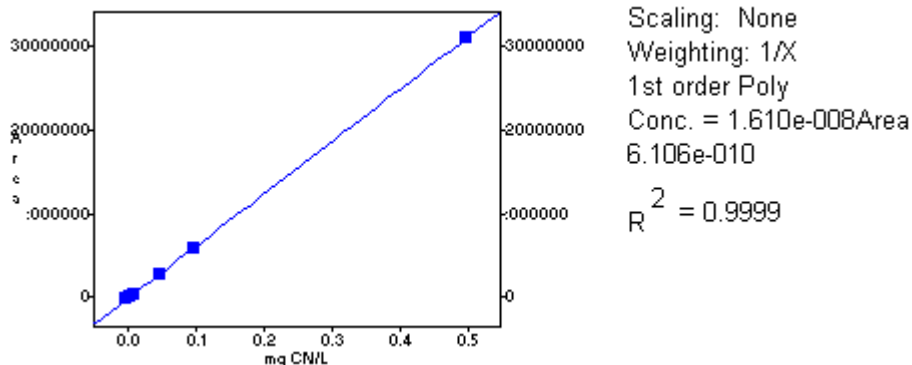
1. Heating Unit
2. MICRO DIST® Block and Cyanide-1 tubes. The MICRO DIST block is available from Lachat Instruments, Loveland, Colorado as Lachat part number A17100. The Cyanide-1 tubes are available as Lachat part number A17001
3. Filter paper, No. 2

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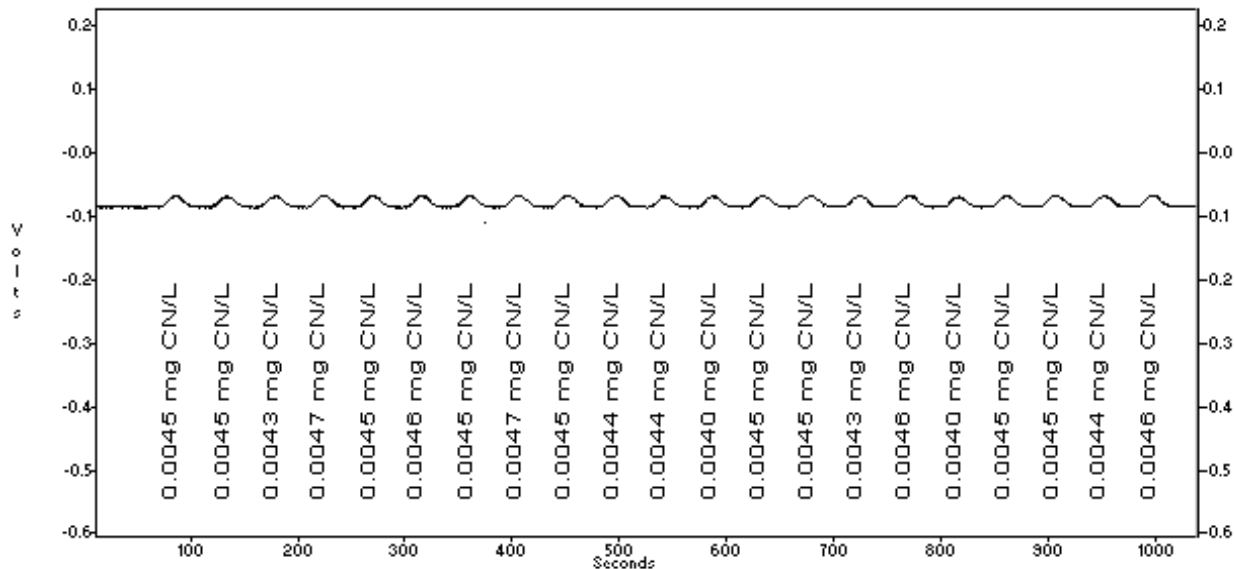
Revised by L. Egan 29 November 2007. Written and copyrighted © by K. Bogren on 22 September 1999 by **Lachat Instruments, 5600 Lindbergh Drive, Loveland, CO 80539 USA**. Phone: 970-663-1377 FAX: 970-962-6710. This document is the property of Lachat Instruments. Unauthorized copying of this document is prohibited.

### Calibration Graph and Statistics

Level	Area	mg CN <sup>-</sup> /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	---



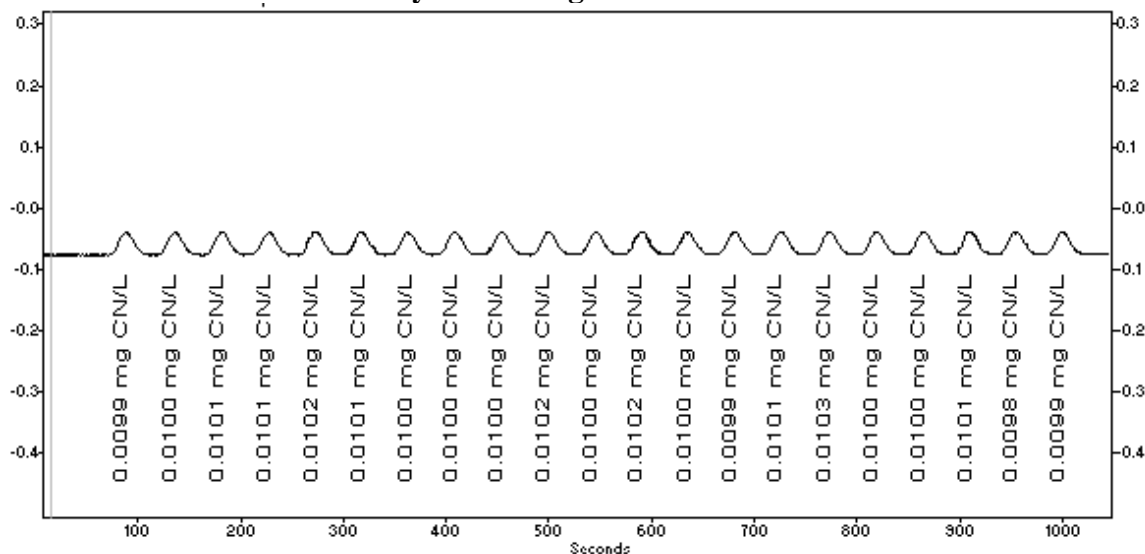
### Method Detection Limit using Distilled Standards



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



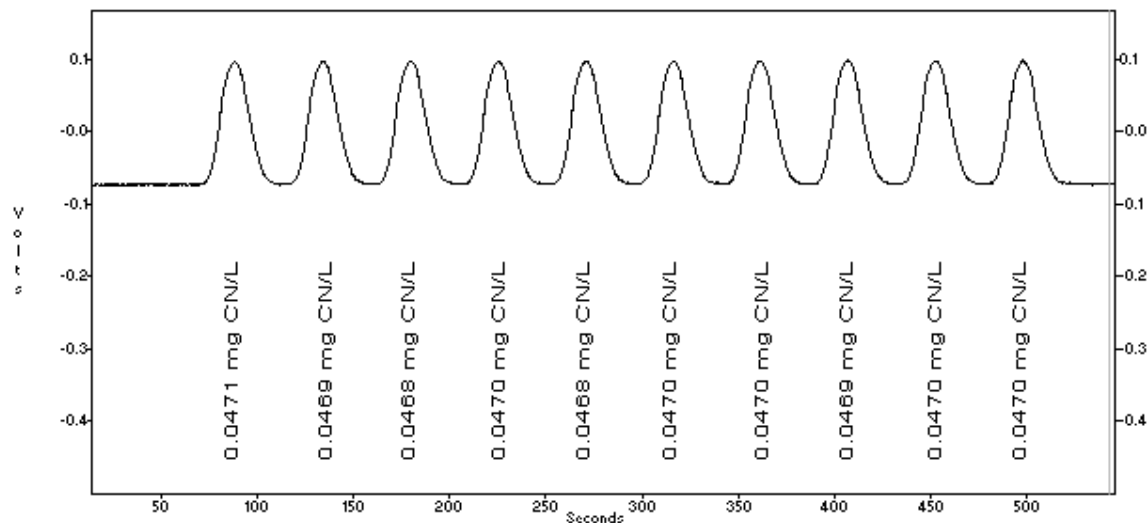
### Method Detection Limit for cyanide using 0.01 mg/L standard

**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



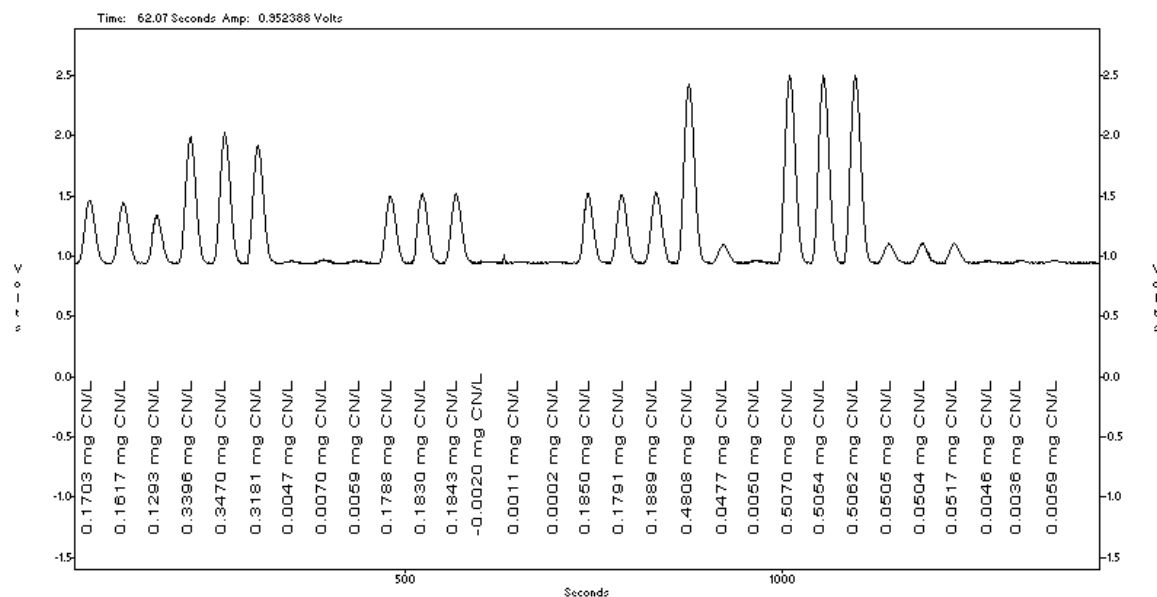
### Precision data for Cyanide using 0.05 mg/L standard

**% 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



ERA water and soil sample spikes; Lake Michigan sample spikes

Data Filename: 102095s1.fdt

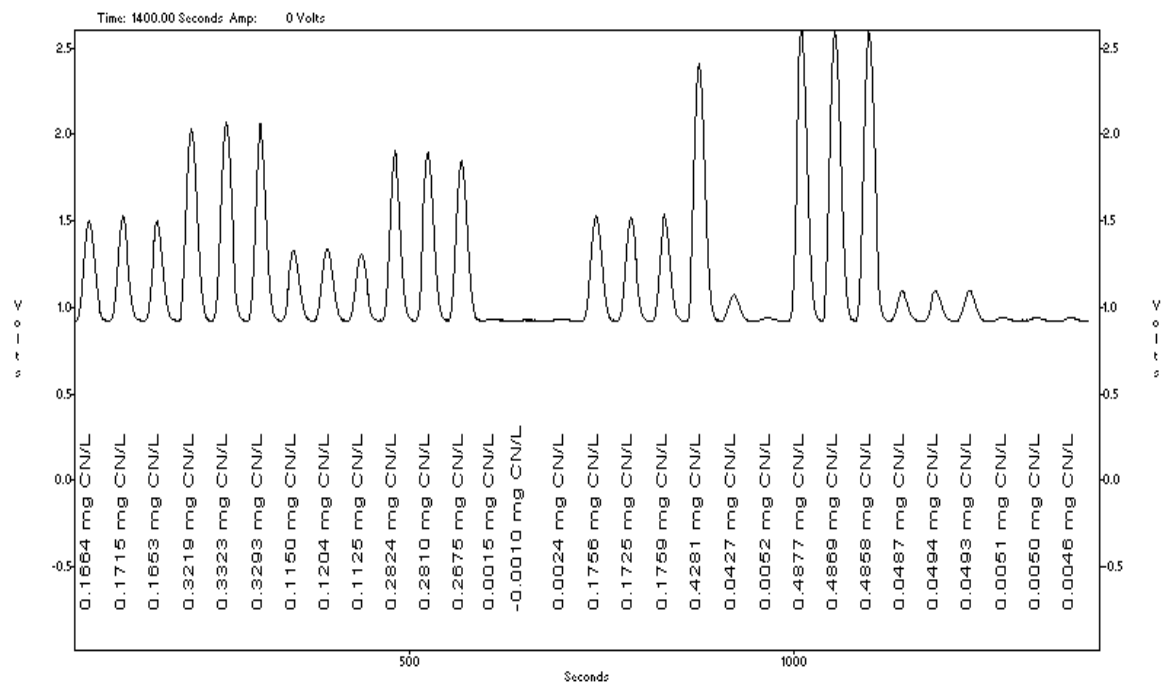
Acquisition Time: 20 October 1995

Recoveries of cyanide in ERA and Lake Michigan sample spikes

Cup Numbers	Average (mg CN <sup>-</sup> /L)	Spike Level (mg CN <sup>-</sup> /L)	Recovery
1-2	0.1660	---	---
4-5	0.3433	0.181	97.96%
7-9	0.0059	---	---
10-12	0.1820	0.181	97.3%
13-15	0.0004	---	---
16-18	0.1843	0.181	101.6%

Recoveries of cyanide in distilled samples compared to undistilled samples

Cup Number	Known Value (mg CN <sup>-</sup> /L)	Determined average Concentration (mg CN <sup>-</sup> /L)	Recovery
19	0.500 distilled	0.4808	94.98 %
22 (3 reps)	0.500 undistilled	0.5062	
20	0.050 distilled	0.0477	93.7 %
23 (3 reps)	0.050 undistilled	0.0509	
21	0.005 distilled	0.0050	106.4 %
24 (3 reps)	0.005 undistilled	0.0047	



Recovery data of cyanide in ERA water, refinery sludge and New Berlin well water samples  
Data Filename: 102695s1.fdt  
Acquisition Time: 26 Oct 1995

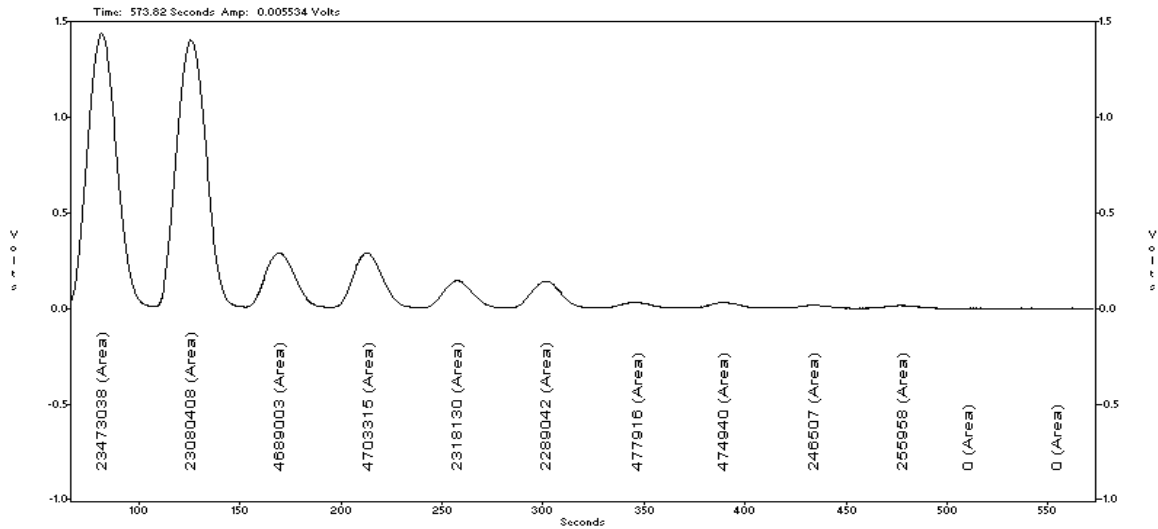
#### Recoveries of cyanide in ERA and Lake Michigan sample spikes

Cup Numbers	Sample Type	Average (mg CN/L)	Spike Level (mg CN/L)	Recovery
1-3	ERA	0.1677	---	---
4-6	ERA	0.3278	0.181	88.5%
7-9	Refinery sludge	0.1160	---	---
10-12	Refinery sludge	0.2770	0.181	88.9%
13-15	New Berlin	0.0013	---	---
16-18	New Berlin	0.1747	0.181	95.8%

#### Recoveries of cyanide in distilled samples compared to undistilled samples

Cup Number	Known Value (mg/L)	Determined average Concentration (mg/L)	Recovery
19	0.500 distilled	0.4281	87.92 %
22 (3 reps)	0.500 undistilled	0.4868	
20	0.050 distilled	0.0427	86.96 %
23 (3 reps)	0.050 undistilled	0.0491	
21	0.005 distilled	0.0052	106.1 %
24 (3 reps)	0.005 undistilled	0.0049	

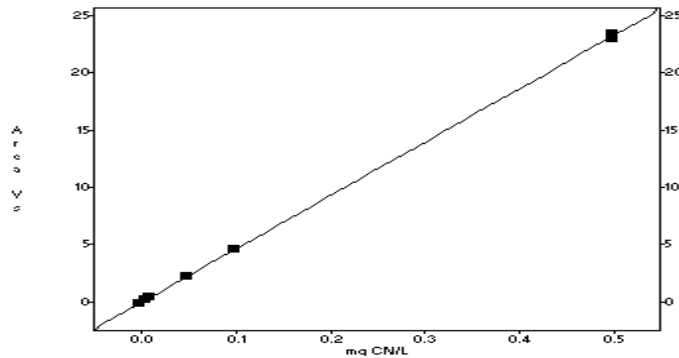
**SUPPORT DATA FOR WEAK ACID DISSOCIABLE CYANIDE  
(USING 10-204-00-1-X; not part of ATP Case Study)**



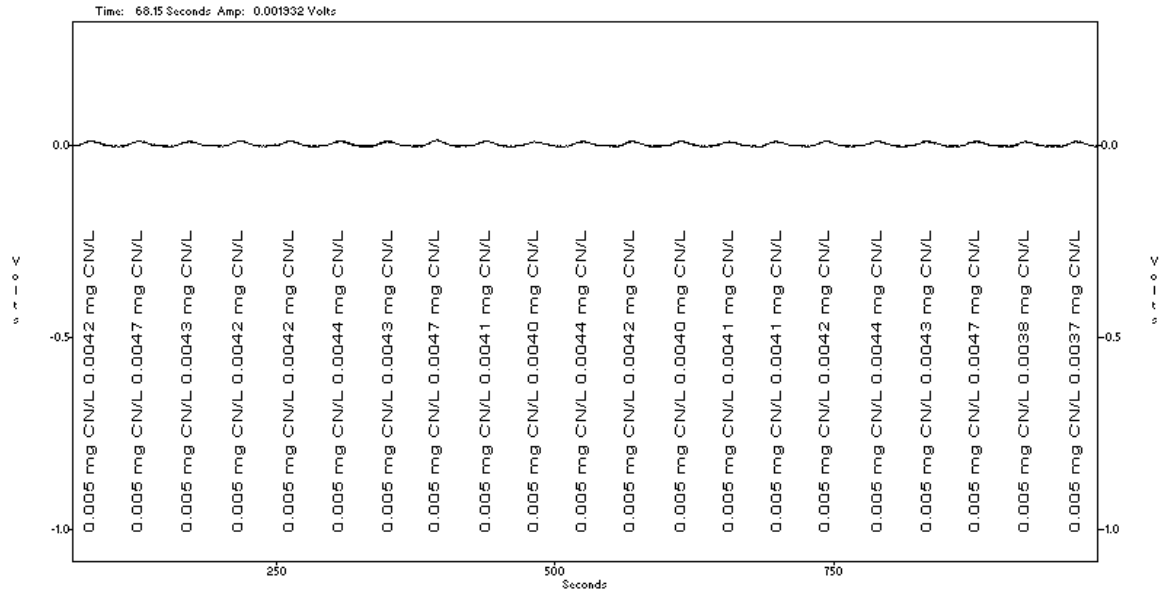
Data Filename: 960207c1.fdt  
Acquisition Time: 7 February 1996

**WAD Cyanide Calibration Graph and Statistics**

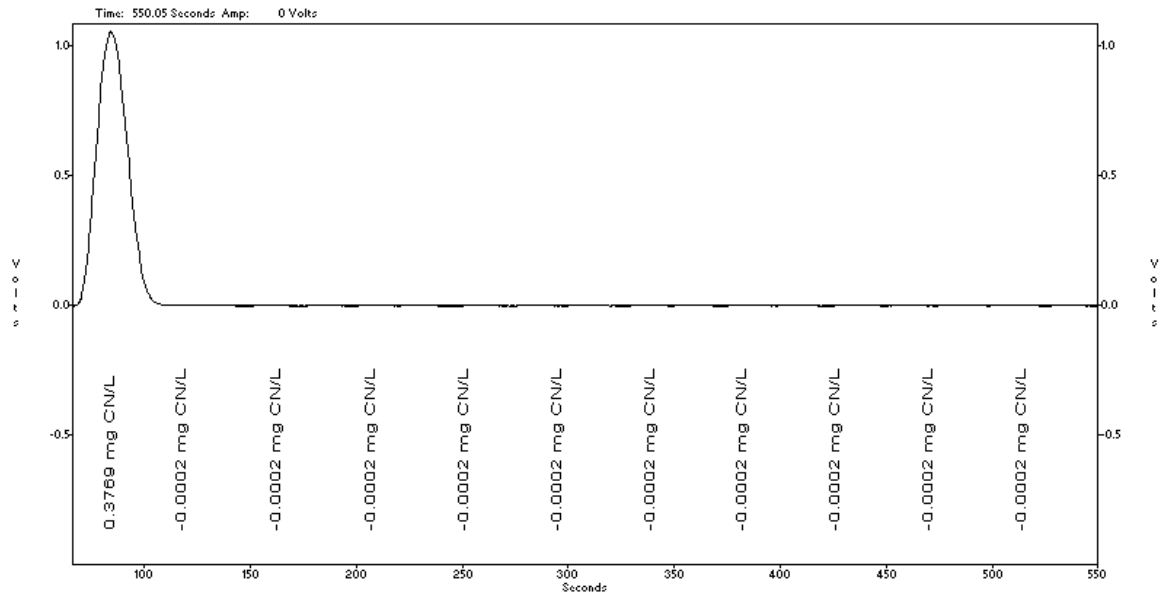
Level	Area	Known (mg CN <sup>-</sup> /L)	Determined (mg CN <sup>-</sup> /L)	Replicate %RSD	% residual
1	23276724	05	0.5	1.2	0.0
2	4696159	0.1	0.1007	0.2	-0.7
3	2303586	0.05	0.0493	0.9	1.4
4	476428	0.01	0.0101	0.4	-0.5
5	251233	0.005	0.0052	2.7	-4.2
6	0	0	--	0	--



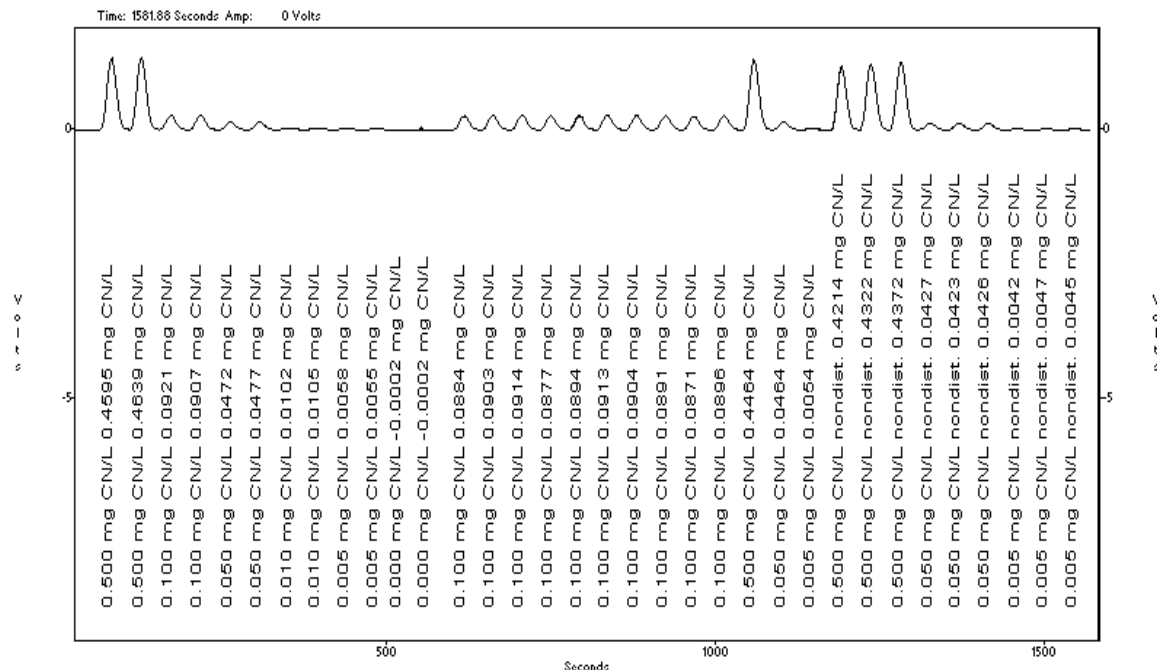
Scaling: None  
Weighting: None  
1st Order Poly  
Conc = 2.149e-008 Area - 1.900e-004  
R<sup>2</sup> = 1.0000



WAD Cyanide Method Detection Limit block using 0.005 mg CN/L distilled standards  
**MDL = 0.0066 mg CN/L**  
 Mean (x) = 0.00424 mg CN/L, Standard Deviation = 0.0026 mg CN/L, Known Value = 0.005 mg CN/L  
 Data Filename: 960207s2.fdt  
 Acquisition time: 7 February 1996



Cyanide carryover on nondistilled standards: 1.0 mg CN/L followed by 10 blanks  
 Carryover passed  
 Data Filename: 960207r1.fdt  
 Acquisition time: 7 February 1996



WAD Cyanide precision block and recoveries on 0.5, 0.05, and 0.005 mg CN<sup>-</sup>/L distilled standards

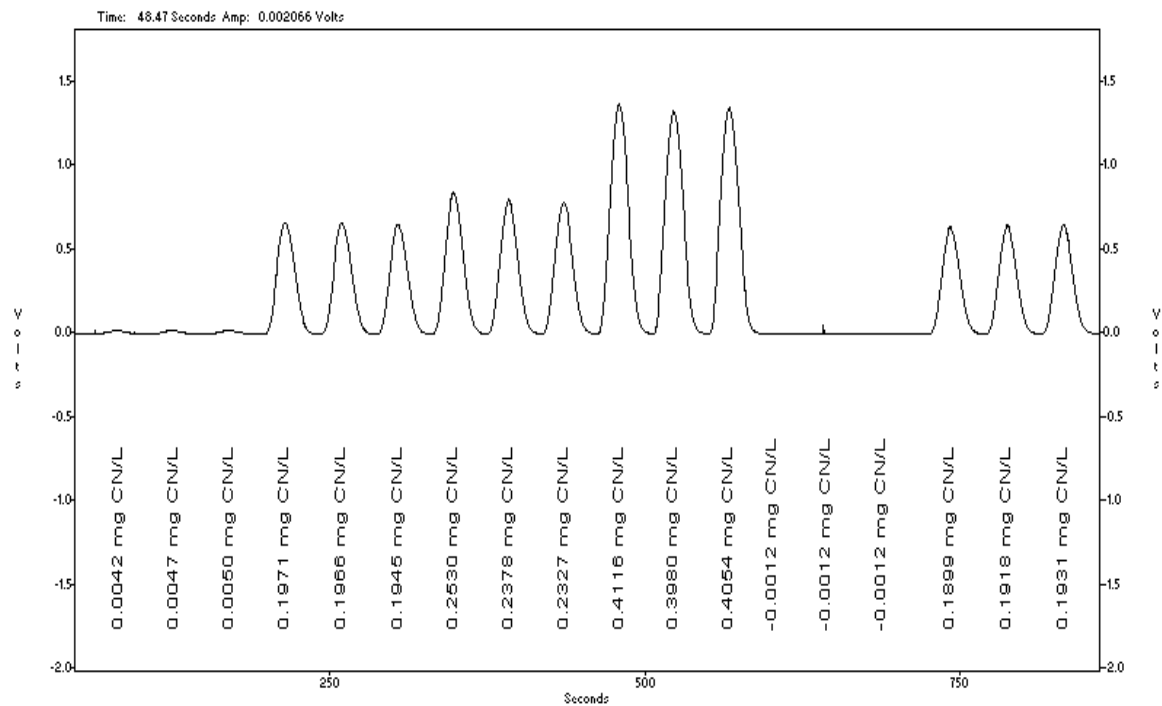
**Precision for 0.1 mg CN<sup>-</sup>/L standards: %RSD = 1.53**

Mean (x) = 0.0895 mg CN<sup>-</sup>/L, Standard Deviation (s) = 0.00137 mg CN<sup>-</sup>/L, Known Value = 0.1 mg CN<sup>-</sup>/L

Data Filename: 960207s1.fdt

**Recoveries of WAD cyanide in distilled samples compared to undistilled samples**

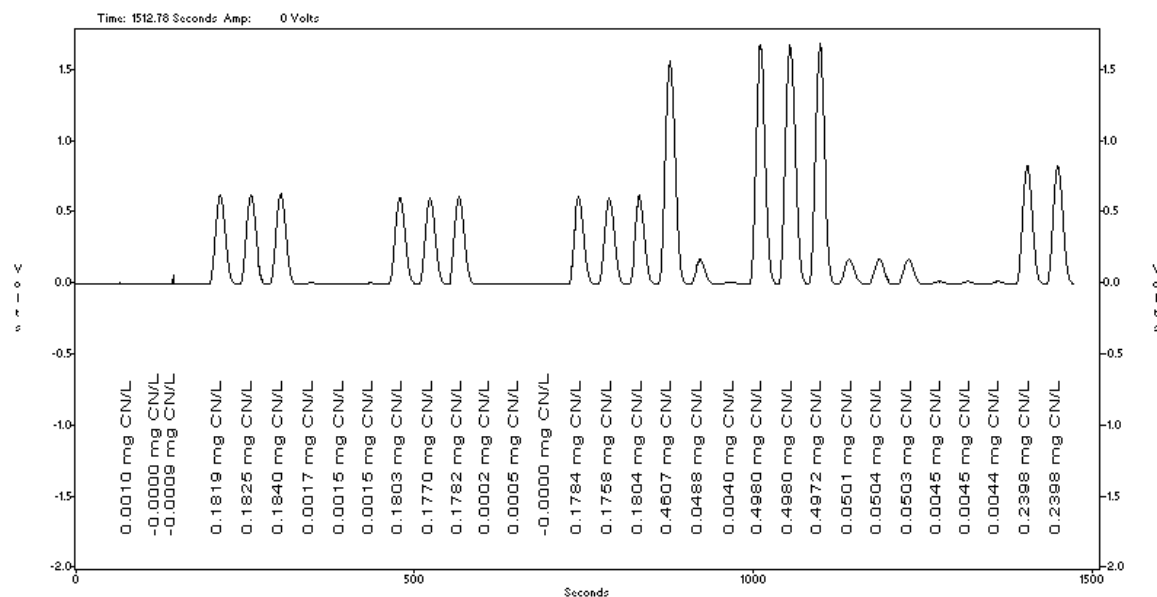
Cup Number	Known Value (mg CN <sup>-</sup> /L)	Determined average Concentration (mg CN <sup>-</sup> /L)	Recovery
17	0.500 distilled	0.4464	103.7 %
20 (3 reps)	0.500 undistilled	0.4303	
18	0.050 distilled	0.0464	109.2 %
21 (3 reps)	0.050 undistilled	0.0425	
19	0.005 distilled	0.0054	120 %
22 (3 reps)	0.005 undistilled	0.0045	



Recoveries of WAD cyanide in ERA water, ERA soil, and Lake Michigan water samples  
Data Filename: 960208s2.fdt  
Acquisition time: 8 Feb 1996

Recoveries of WAD cyanide in ERA and Lake Michigan sample spikes

Cup Numbers	Sample Type	Average (mg/L)	Spike Level (mg/L)	Recovery
1-3	ERA water	0.0046	---	---
4-6	ERA water - spiked	0.1961	0.181	105.8%
7-9	ERA soil	0.2412	---	---
10-12	ERA soil - spiked	0.4050	0.181	95.9%
13-15	Lake Michigan	0.0000	---	---
16-18	Lake Michigan - spiked	0.1916	0.181	105.8%



Recoveries of cyanide in New Berlin well water, refinery sludge, and Milwaukee STP effluent samples  
Data Filename: 960209s1.fdt  
Acquisition time: 9 February 1996, 15:45:38

Recoveries of cyanide in New Berlin well, refinery sludge, and Milwaukee STP samples

Cup Numbers	Sample Type	Average (mg CN <sup>-</sup> /L)	Spike Level (mg CN <sup>-</sup> /L)	Recovery
1-3	New Berlin well	0.0000	---	---
4-6	New Berlin well - spiked	0.1828	0.181	101.0%
7-9	refinery sludge	0.0016	---	---
10-12	refinery sludge - spiked	0.1785	0.181	97.7%
13-15	Milwaukee STP effluent	0.0002	---	---
16-18	Milw. STP effluent -spiked	0.1782	0.181	98.3%

Recoveries of cyanide in distilled samples compared to undistilled samples

Cup Number	Known Value (mg CN <sup>-</sup> /L)	Determined average Concentration (mg CN <sup>-</sup> /L)	Recovery
19	0.500 distilled	0.4607	92.6 %
22 (3 reps)	0.500 undistilled	0.4977	
20	0.050 distilled	0.0488	97.0 %
23 (3 reps)	0.050 undistilled	0.0503	
21	0.005 distilled	0.0040	88.9 %
24 (3 reps)	0.005 undistilled	0.0045	

**QuikChem<sup>®</sup> Method 10-204-00-5-X**

# **Cyanide in drinking and wastewaters using the MICRO DIST distillation system 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.

**– Special Apparatus –**

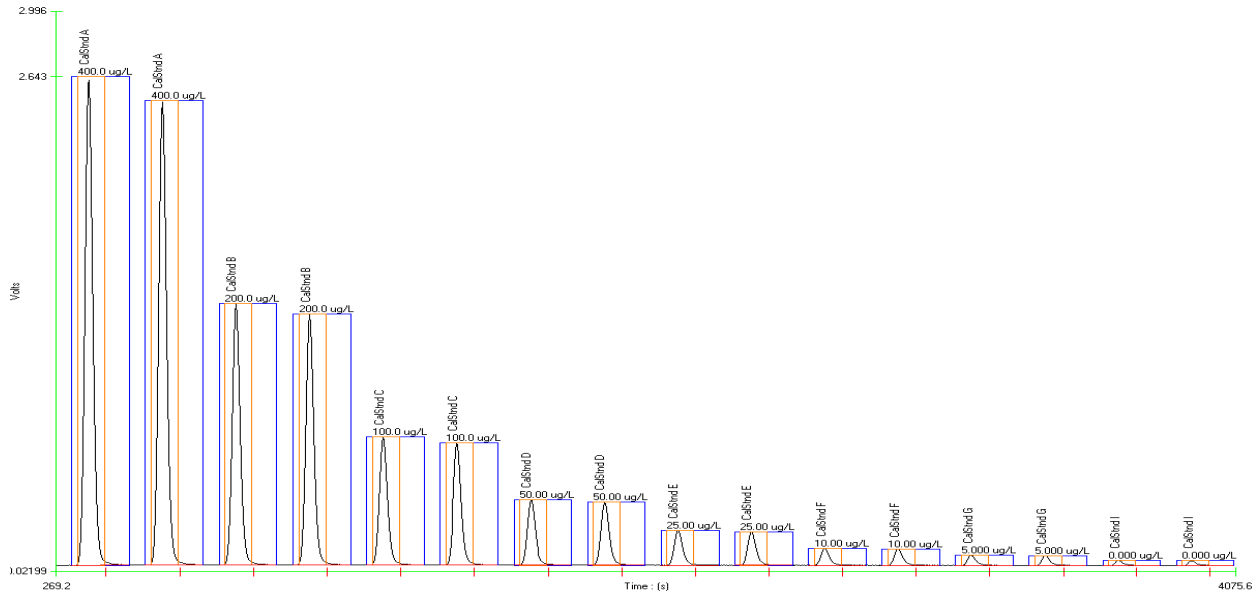
Please see Parts and Price list for Ordering Information

1. Heating Unit.
2. MICRO DIST Block and User Filled cyanide tubes. The MICRO DIST<sup>®</sup> block is available from Lachat Instruments, Loveland, Colorado as Lachat part number A17100. The user filled tubes are available as Lachat part number A17117 (pkg of 100), A17017 (pkg of 10), A17517 (pkg of 50).
3. Filter paper, No. 2
4. Amperometric Detector apparatus (Lachat Part No. 84920)
5. Direct Voltage Detector (Lachat Part No. 84967 for the QC8000; 85272 for the QC8500)

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Written and copyrighted © by Scott Tucker 30 May, 2008 by **Lachat Instruments, 5600 Lindbergh Drive, Loveland, CO 80539 USA**. Phone: 970-663-1377 FAX: 970-962-6710. This document is the property of Lachat Instruments. Unauthorized copying of this document is prohibited.

### Calibration Data for Cyanide (Distilled Standards)



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

### Calibration Graph and Statistics (Distilled Standards)

**Calibration Results - Channel 2**

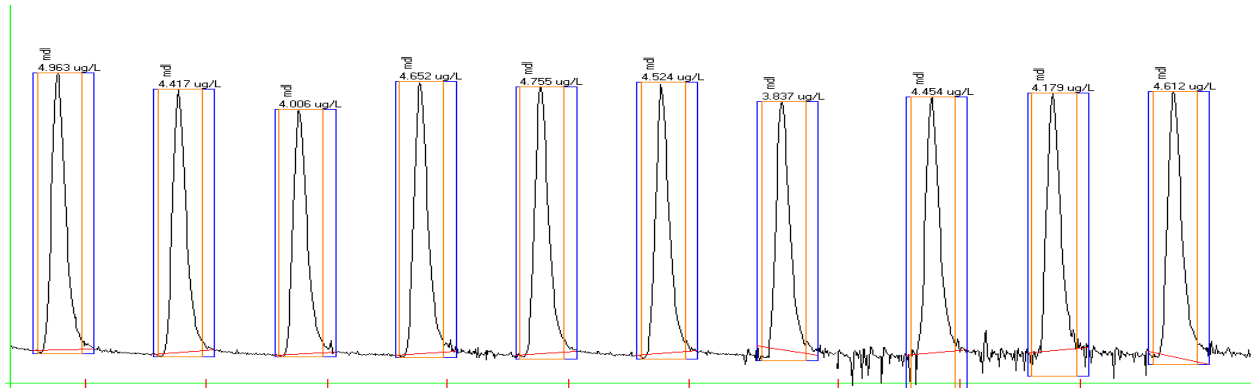
	Known Conc. (ug/L)	Rep.	Peak Area (V.s)	Peak Height (V)	% RSD	% Resid.	Unused	Det. Conc (ug/L)	Date	Time
1	400.0	1	76.68	2.630	3.5	-2.1	<input type="checkbox"/>	408.1	5/22/2008	8:34:53 AM
2	400.0	2	73.02	2.500	3.5	2.8	<input type="checkbox"/>	386.8	5/22/2008	8:38:52 AM
3	200.0	1	41.18	1.407	2.8	-3.9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

Settings



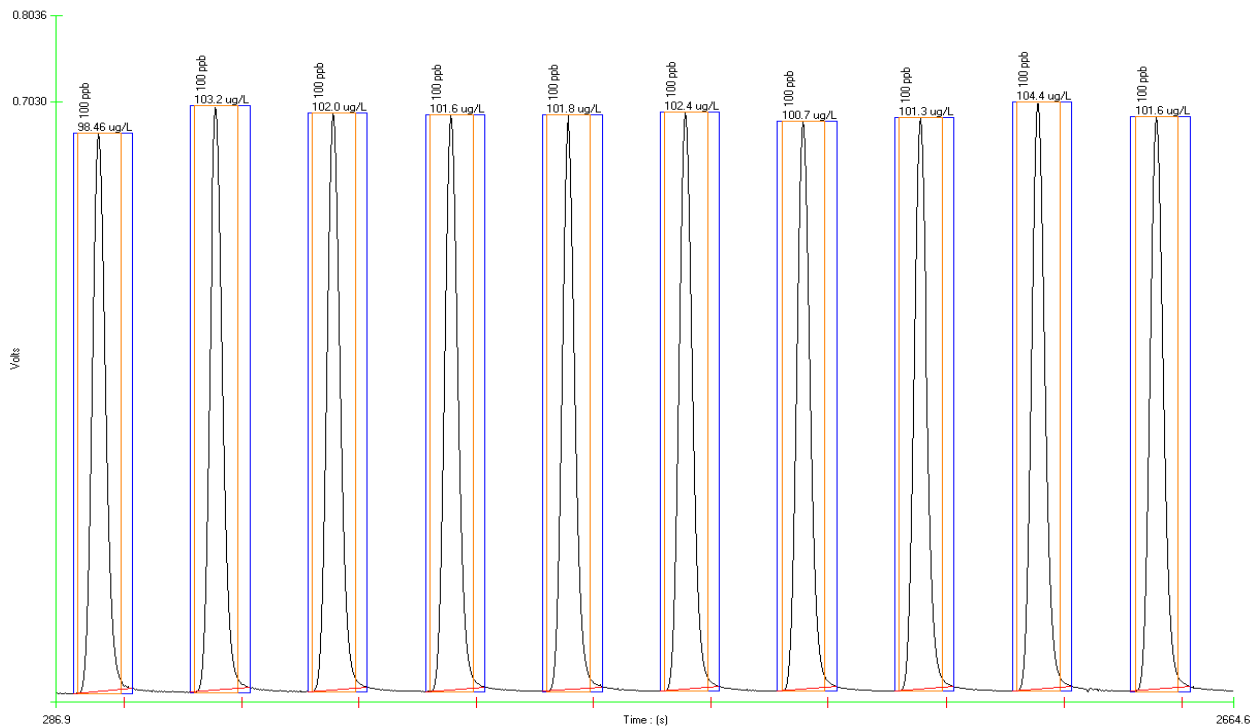
**Method Detection Limit for cyanide using a 4.0 µg CN<sup>-</sup>/L standard (Distilled Standards)**

**MDL = 0.975 µg CN<sup>-</sup>/L**

Standard Deviation (s) = 0.346 µg CN<sup>-</sup>/L, Mean (x) = 4.44 µg CN<sup>-</sup>/L, Known value = 4.0 µg CN<sup>-</sup>/L

File Name: 5-22 Prec mdl.omn

Acq. Date: 22 May 2008



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

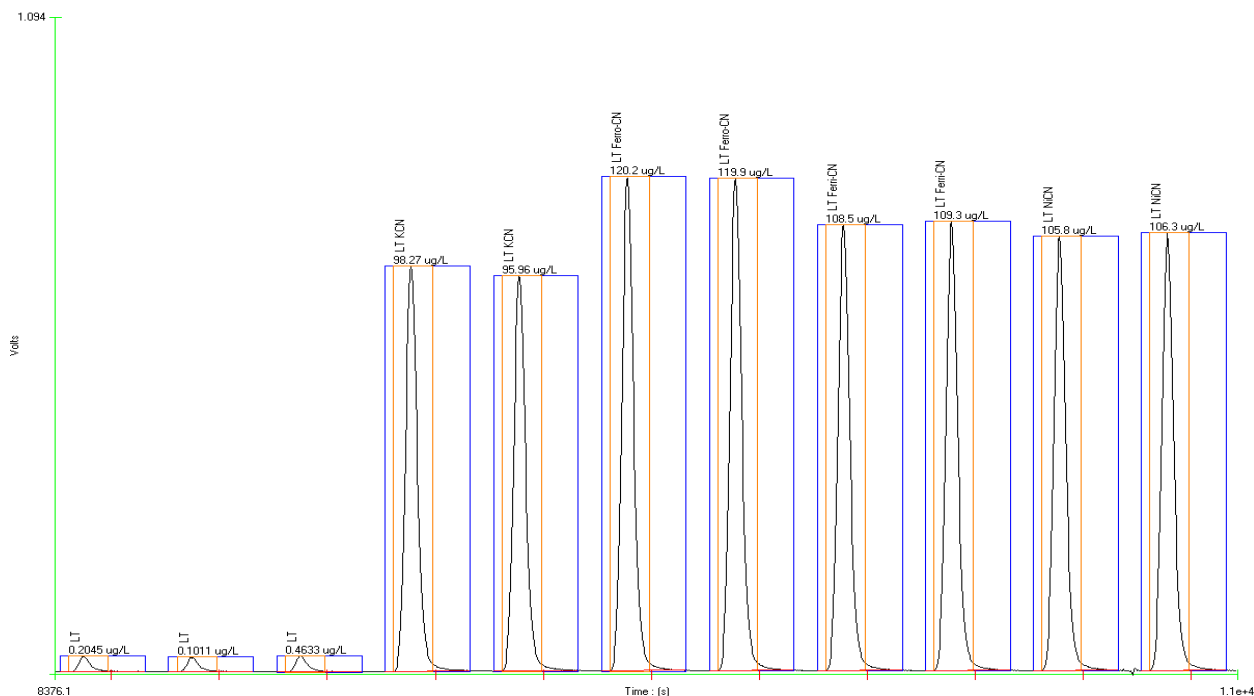
**% RSD = 1.53 %**

Standard Deviation (s) = 1.56 µg CN<sup>-</sup>/L, Mean (x) = 101.75 µg CN<sup>-</sup>/L, Known value = 100 µg CN<sup>-</sup>/L

File Name: 5-22 Prec mdl.omn

Acq. Date: 22 May 2008

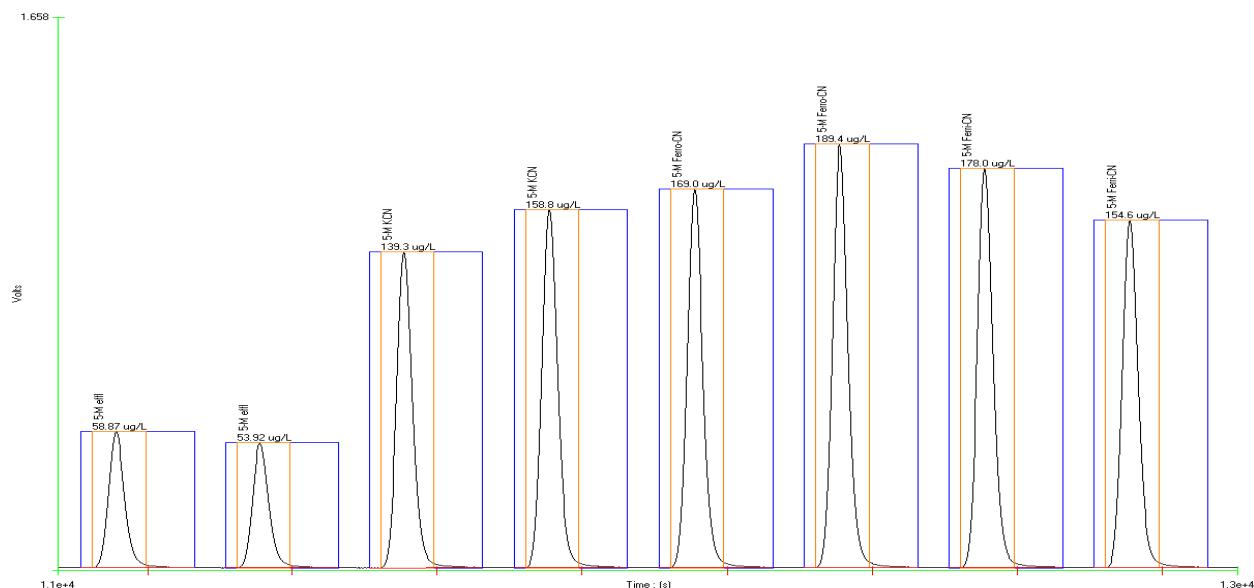
## Cyanide Spike Recoveries in Loveland, CO Tap Water

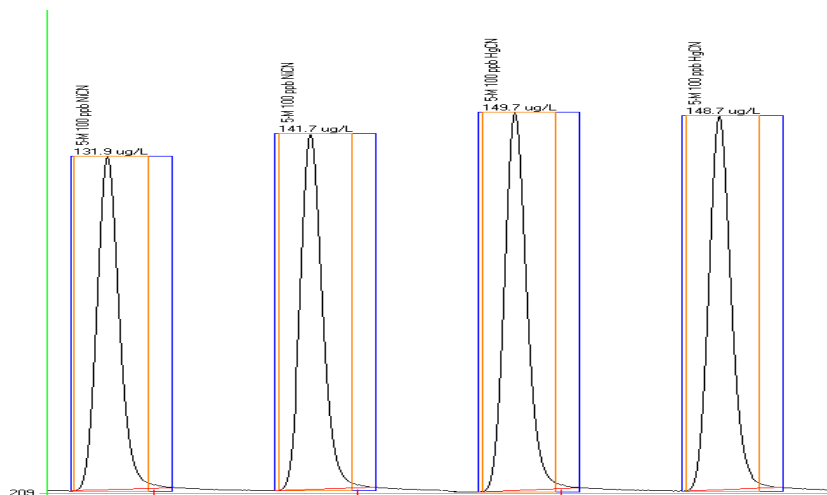


Sample ID	Average spike recoveries ( $\mu\text{g CN/L}$ )	Spike Level ( $\mu\text{g CN/L}$ )	% Recovery
Loveland Tap Water	0.256	---	---
Loveland Tap Water KCN	97.12	100	96.86
Loveland Tap Water Ferro-CN	120.1	100	119.8
Loveland Tap Water Ferri-CN	108.9	100	108.6
Loveland Tap Water $\text{K}_2\text{Ni}(\text{CN})_4$	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

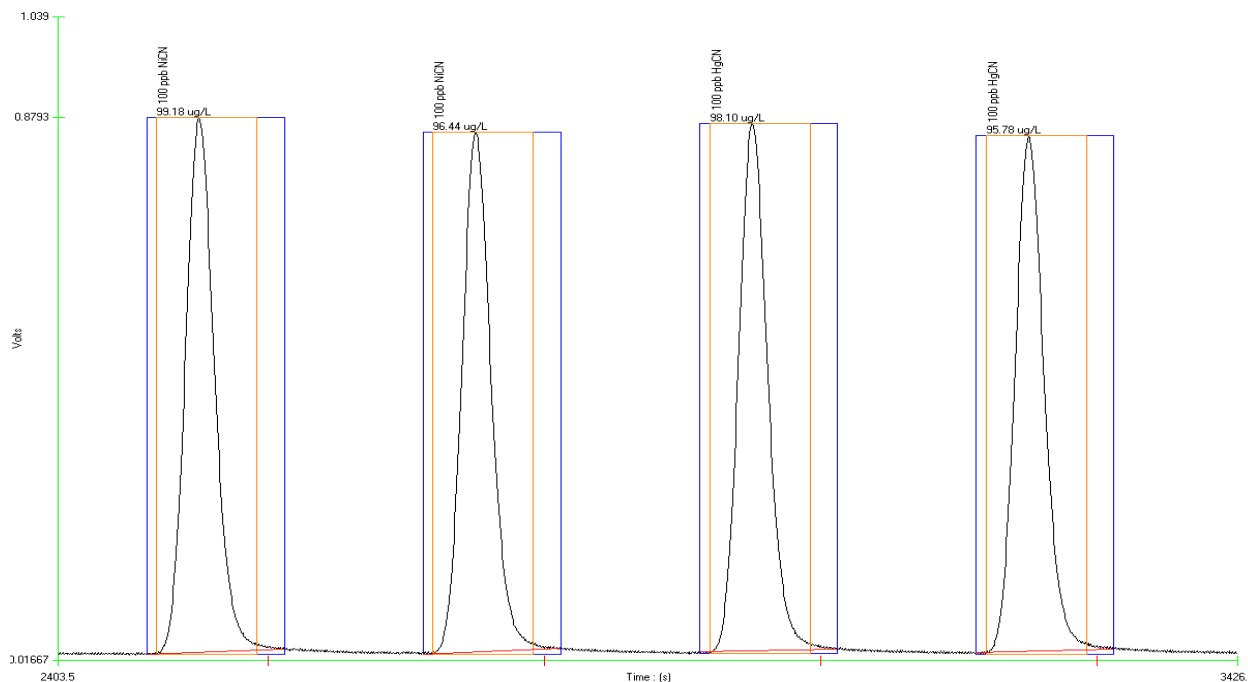




Sample ID	Average (µg CN/L)	Spike Level (µg CN/L)	% Recovery
5-M effluent, unspiked	56.40	---	---
5-M KCN	149.1	100	92.66
5-M Ferro-CN	179.2	100	122.8
5-M Ferri-CN	166.3	100	109.9
5-M K <sub>2</sub> Ni(CN) <sub>4</sub>	136.8	100	80.41
5-M Hg(CN) <sub>2</sub>	149.2	100	92.81

**Conclusion:** Potassium, Ferro, Ferri, Nickel and Mercury cyanide are recovered at levels greater than 80% in a wastewater matrix.

### 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% in a DI water matrix.

**QuikChem<sup>®</sup> Method 10-210-00-1-X**

**Phenolic Compounds in Waters  
(MICRO DIST<sup>®</sup> method)**

**5.0 to 200 µg phenol/L Low Range  
0.05 to 2.0 mg phenol/L High Range**

**– Principle –**

Volatile phenolic compounds are separated from the sample matrix by distillation. The distillate is collected and analyzed. Phenol, ortho- and meta- substituted phenols, and para- substituted phenols where the para- group is a carboxyl, a halogen, a methoxyl, or a sulfuric acid group are all determined by reaction with 4-aminoantipyrine. Not determined are para-cresol, and other para- substituted phenols where the para- group is an alkyl, an aryl, a nitro, a benzoyl, a nitroso, or an aldehyde group.

**– Interferences –**

1. Interference from sulfur compounds are eliminated by acidifying the sample to a pH of 4.0 and aerating briefly by stirring.
2. Oxidizing agents such as chlorine, detected by the liberation of iodine upon acidification in the presence of potassium iodide, are removed immediately after sampling by the addition of an excess of ferrous ammonium sulfate (Reagent 4). If chlorine is not removed, the phenolic compounds may be partially oxidized and the results may be low.
3. Glass containers for standards and samples are required.
4. Oils and tars can be removed prior to distillation by extraction with chloroform (CHCl<sub>3</sub>). Adjust the sample pH to 12-12.5 by adding NaOH pellets, and then extract the oil or tar with a 50 mL portion of chloroform. Remove the chloroform layer and evaporate the excess chloroform in the aqueous layer by gently warming the aqueous layer.
5. Method interference may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that bias analyte response.

**– Special Apparatus –**

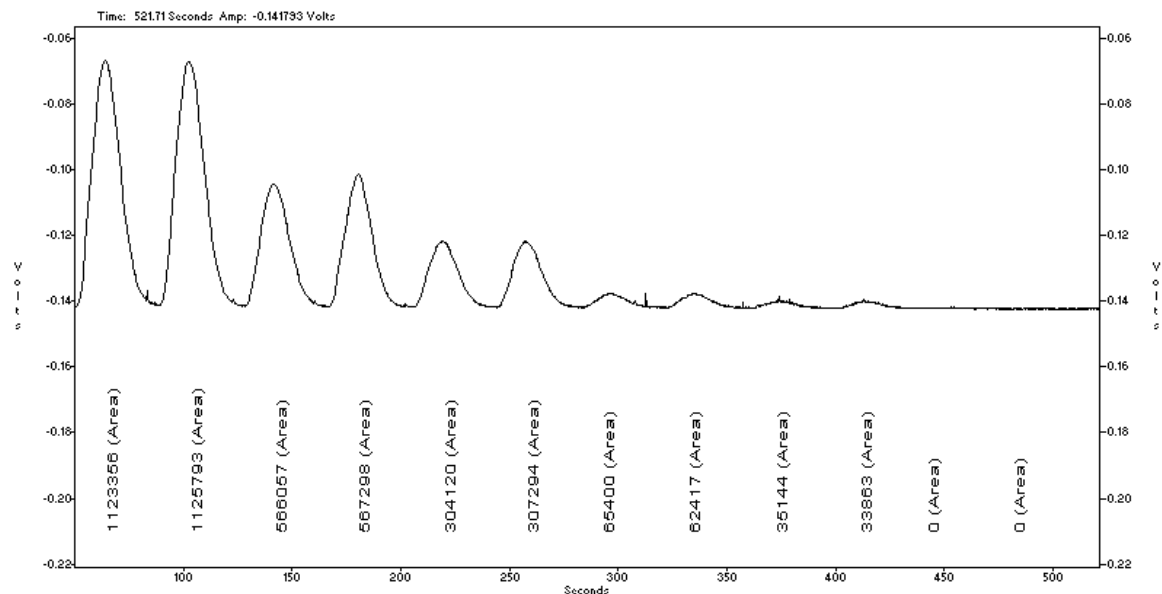
Please see Parts and Price list for Ordering Information

1. MICRO DIST<sup>®</sup> DISTILLATION SYSTEM
2. MICRO DIST<sup>®</sup> tubes (Lachat Part No. A17002)
3. Glass calibration vials must be used, (Lachat Part No. 21304) for XYZ samplers.

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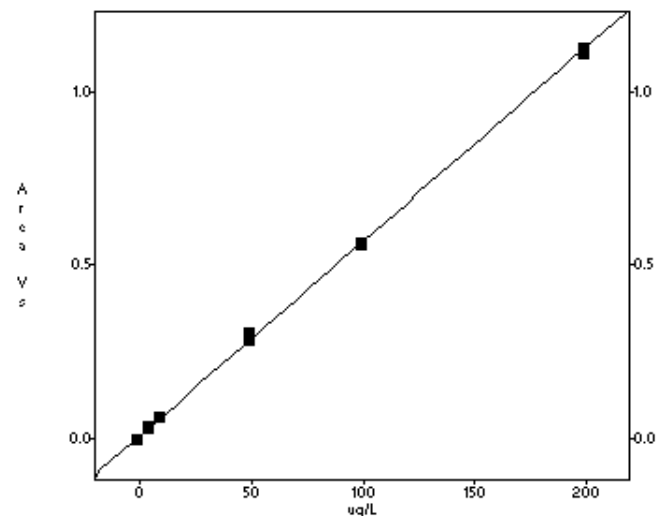
## Low Range Calibration Data for Phenol



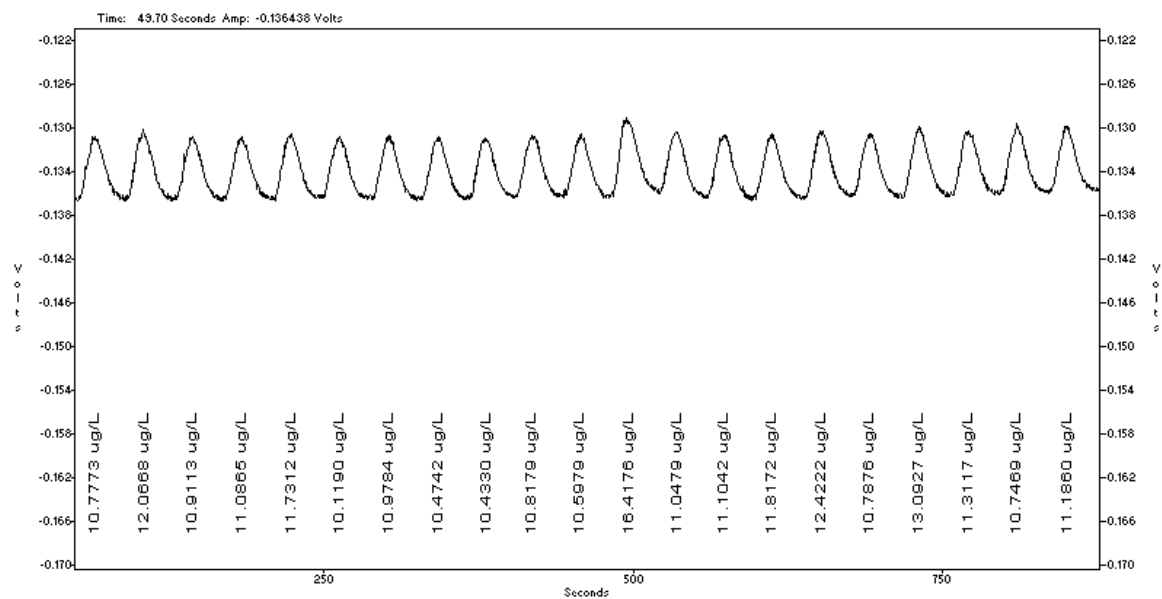
Data Filename: 960226c3.fdt  
Acquisition Time: 26 Feb 1996,13:54:20

## Calibration Graph and Statistics

Level	Area	µg phenol/L (known)	µg phenol/L (determined)	Replic %RSD	% residual
1	1125793	200	199.2	0.5	0.4
2	567298	100	99.7	0.5	0.3
3	307294	50	53.3	3.4	-6.6
4	62417	10	9.65	2.0	3.5
5	33863	5	4.56	4.2	8.9
6	0	0	1.49	0.0	--



Scaling: None  
Weighting: None  
1st Order Poly  
Conc = 1.783e-004 Area - 1.486e+000  
R<sup>2</sup> = 0.9995



**Phenol MDL study using 10.0 µg phenol/L standard**

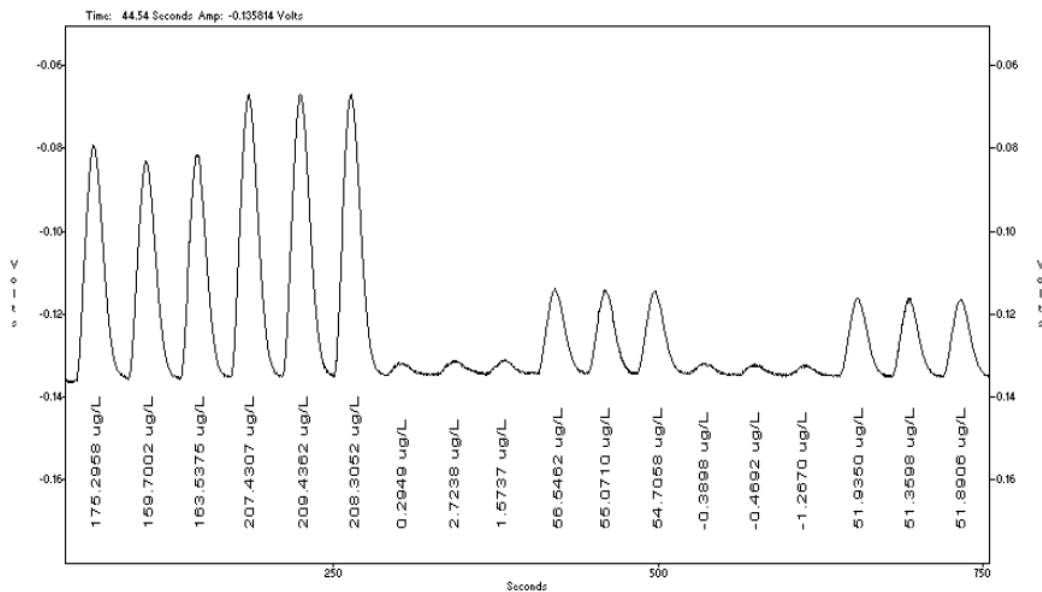
**MDL = 3.4 µg phenol/L**

Mean (x) = 11.43 µg phenol/L, standard deviation (s) = 1.34 µg phenol/L, known concentration = 10.0 µg phenol/L

Data Filename: 960227s2.fdt

Acquisition Time: 27 Feb 1996, 16:05:54

**Spike Recoveries**



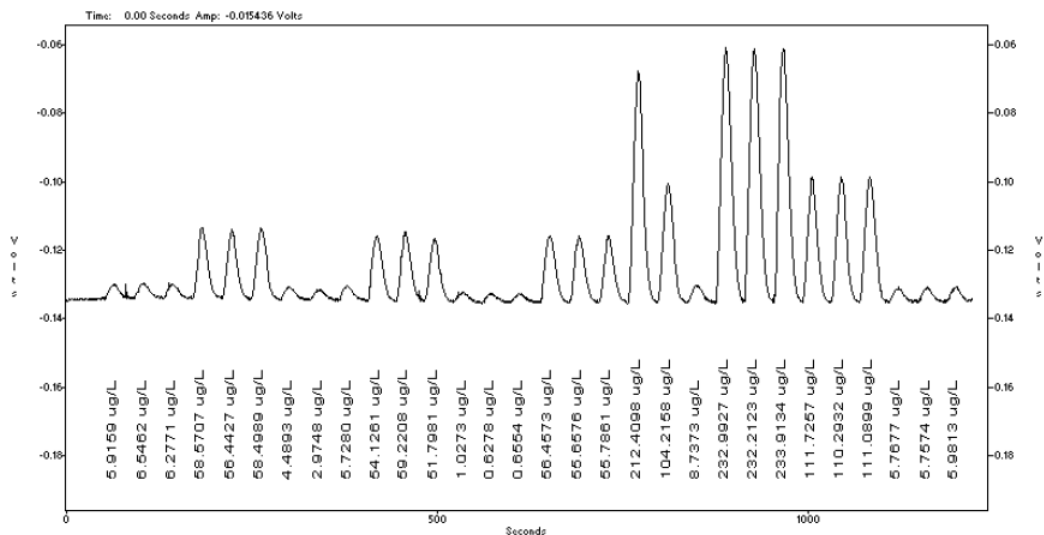
**Recoveries of phenol in ERA water and soil samples and Lake Michigan samples**

Data Filename: 960228s1.fdt

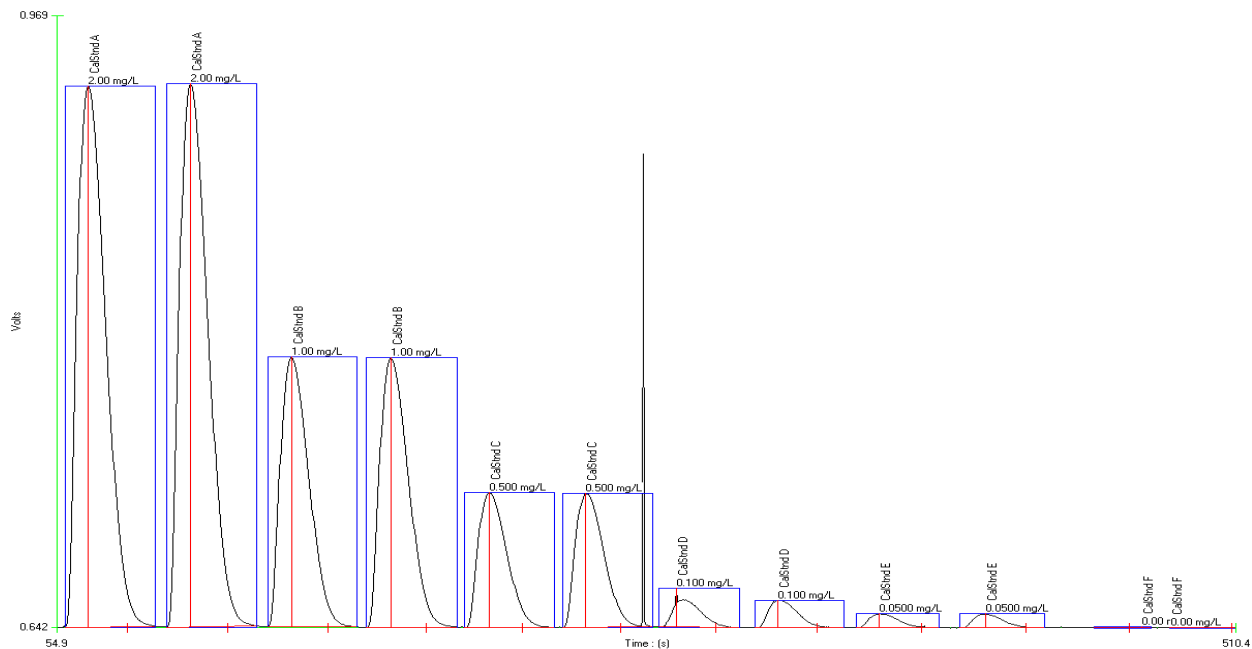
Acquisition Time: 28 February 1996

Recoveries of phenol in ERA and Lake Michigan sample spikes

Cup Numbers	Sample Type	Average ( $\mu\text{g phenol/L}$ )	Spike Level ( $\mu\text{g phenol/L}$ )	Recovery
1-3	ERA sample	166.18	---	---
4-6	ERA sample - spiked	208.41	50	84.5%
7-9	soil sample	1.53	---	---
10-12	soil sample - spiked	55.44	50	107.8%
13-15	Lake Michigan	0	---	---
16-18	Lake Michigan -spiked	51.73	50	103.5%

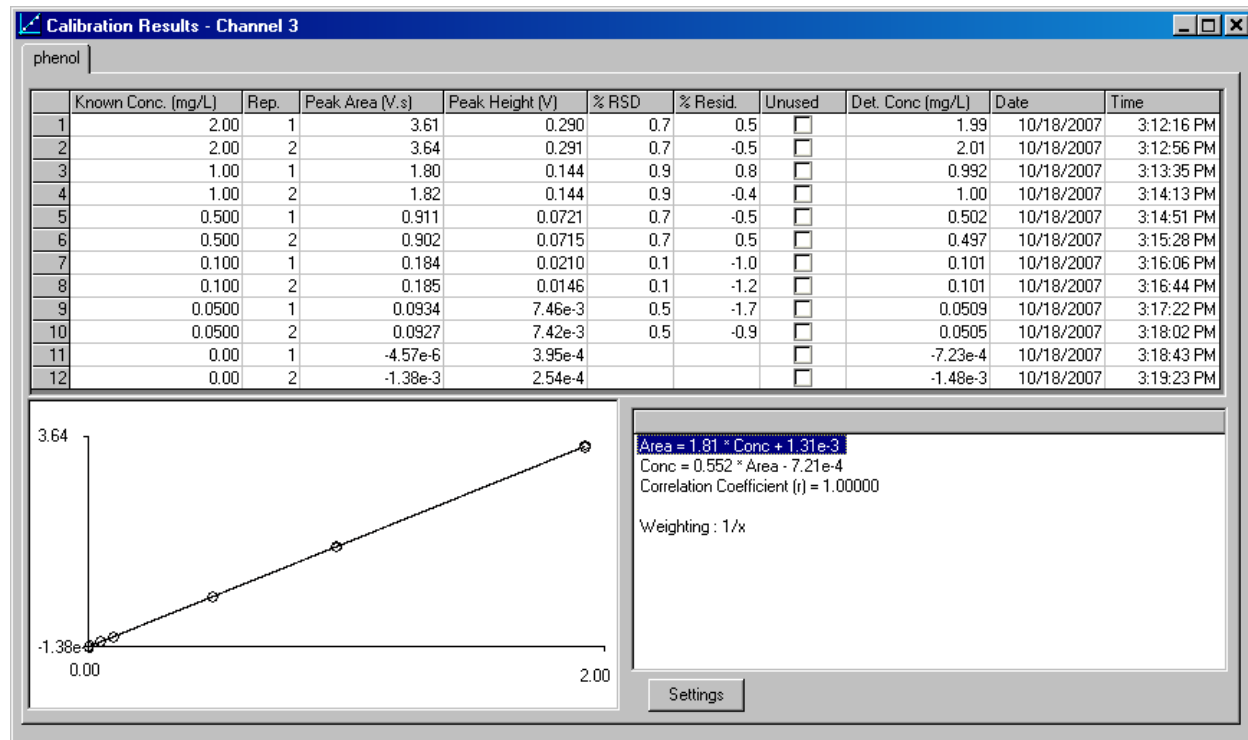


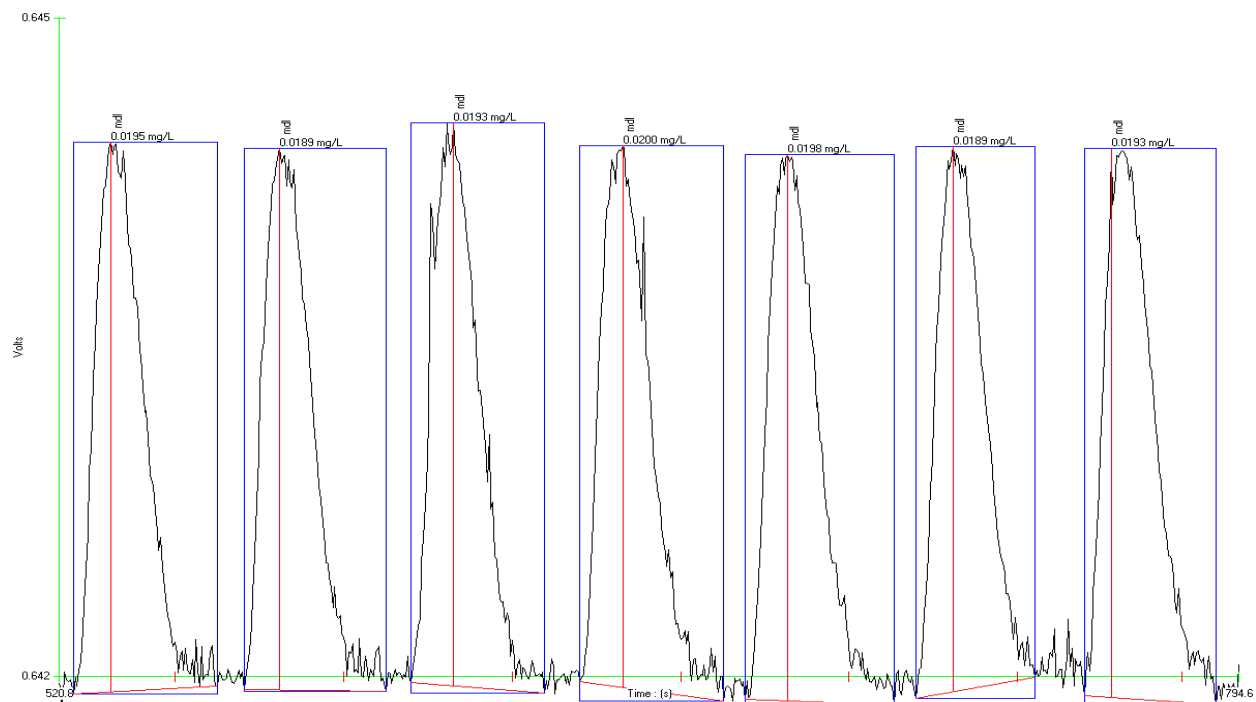
Recoveries of phenol in New Berlin well water, refinery sludge, and Milwaukee STP effluent  
Data Filename: 960229s1.fdt  
Acquisition time: 29 February 1996



File name: OM\_10-18-2007\_03\_11\_31PM.OMN  
Acq. Date: 18 October 2007

### Calibration Graph and Statistics





**Method Detection Limit for phenol using 0.02 mg phenol/L standard**

**MDL= 1.3 µg phenol/L**

Standard Deviation (s) = 0.418 µg phenol/L, Mean (x) = 0.019 mg phenol/L, Known value = 0.02 mg phenol/L

File name: OM\_10-18-2007\_03\_11\_31PM.OMN

Acq. Date: 18 October 2007

**QuikChem<sup>®</sup> Method 10-116-29-1-A**

**Sulfide by MICRO DIST<sup>®</sup>**

**0.02 to 2.0 mg S/L**

**– Principle –**

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

**– Interferences –**

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

**– Special Apparatus –**

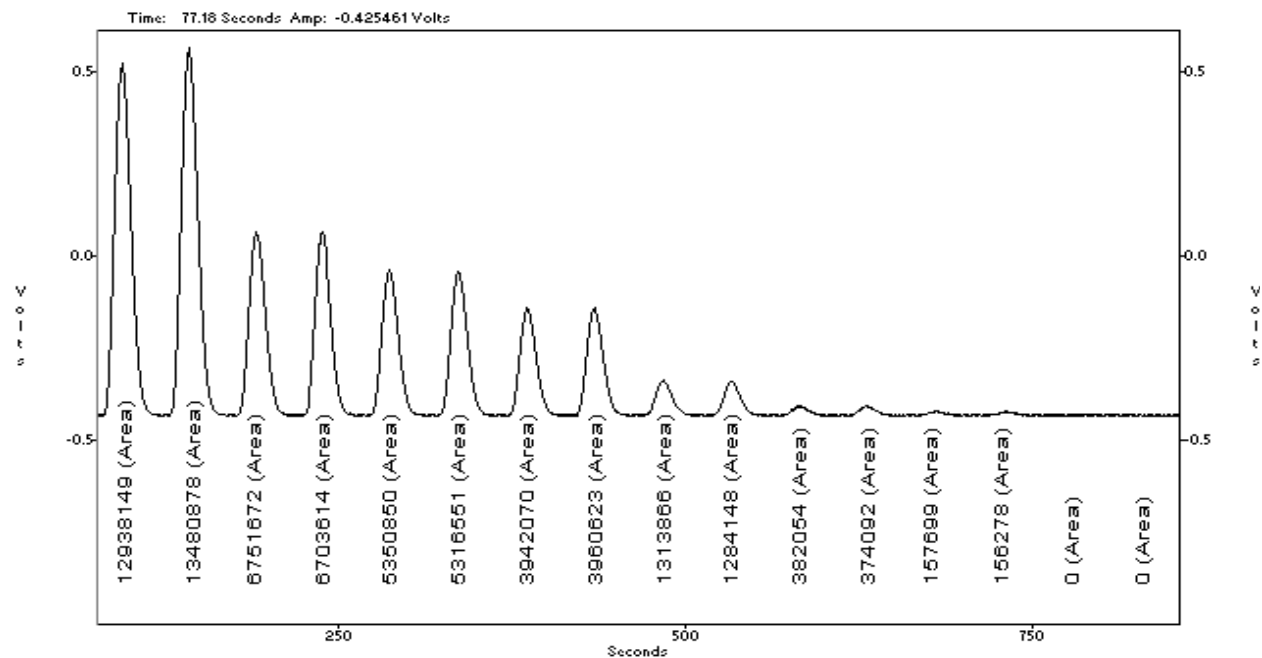
Please see Parts and Price list for Ordering Information

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

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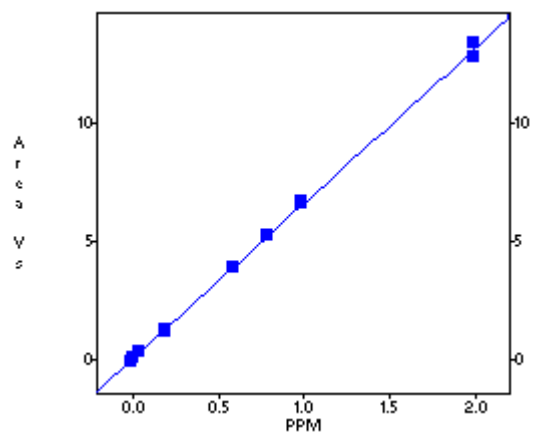
### Calibration Data for Sulfide



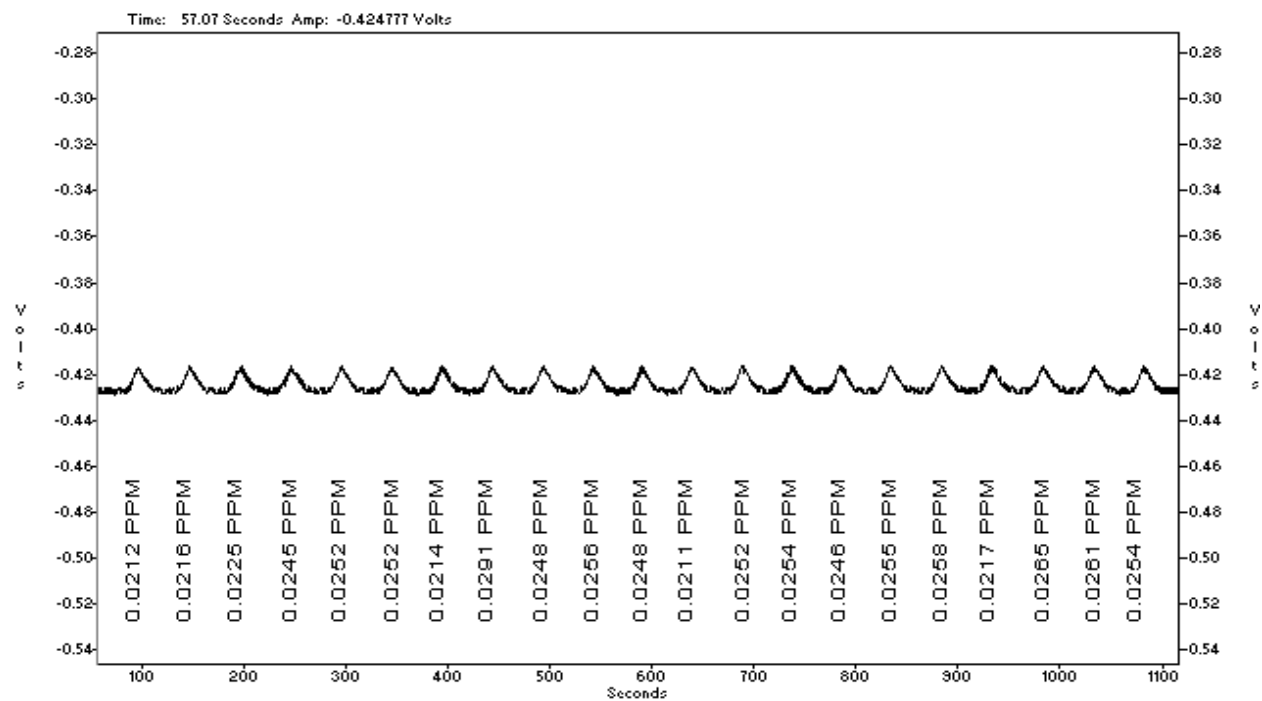
File Name: 960208c1.fdt  
Acq. Time: February 8, 1996, 14:10:00

### Calibration Graph and Statistics

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



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



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

**MDL= 0.005 mg/L**

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

Acq. Date: February 8, 1996



For Technical Assistance, Pricing Information, and Ordering:

**In the USA:**  
(800) 247-7613

**International:**  
Contact the Lachat office or distributor serving you.

**Support Email:**  
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**Sales Email:**  
sales@lachatinstruments.com

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