

USEPA Method OIA-1677: A Novel Approach to Cyanide Analysis



On July 7, 1998, the U.S. Environmental Protection Agency (USEPA) proposed amending the Guidelines Establishing Test Procedures for the Analysis of Pollutants under Section 304(h) of the Clean Water Act by adding Method OIA-1677: Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry. This method employs flow injection analysis (FIA) and was developed and validated by ALPKEM™ (purchased by OI Analytical in 1996) in cooperation with the University of Nevada - Reno Mackay School of Mines. According to the Federal Register notice, the USEPA considers Method OIA-1677

to be "a significant addition to the suite of analytical testing procedures for available cyanide because it (1) has greater specificity for cyanide in matrices where interferences have been encountered using currently approved methods, (2) has improved precision and accuracy compared to currently approved CATC cyanide methods, (3) measures available cyanide at lower concentrations, (4) offers improved analyst safety, (5) shortens sample analysis time, and (6) reduces laboratory waste."

Introduction

Cyanide is a toxic pollutant pursuant to section 307(a)(1) of the Clean Water Act (CWA)¹ and is a priority pollutant as derived from the toxic pollutant list.² Public and private entities subject to regulatory limits for cyanide amenable to chlorination (CATC) under the CWA, as well as CWA regulatory authorities, have indicated interference problems when the currently approved methods were used to test certain sample matrices. During the Seventeenth Annual EPA Conference on Analysis of Pollutants in the Environment (May 3-5, 1994), the interference situation was publicized and a request was made for comments and suggestions for a cyanide method that reduced or eliminated these interferences.³ At the time, ALPKEM was working with the University of Nevada - Reno Mackay School of Mines on a dramatically simplified and improved method for cyanide analysis. ALPKEM began a dialog with the USEPA that rapidly developed into a process whereby the new cyanide method became a test vehicle for the USEPA's Streamlining Proposal.⁴

Summary of Method OIA-1677

Method OIA-1677 is divided into two parts: (1) sample pretreatment and (2) cyanide quantification via amperometric detection. In the sample pretreatment step, ligand-exchange reagents are added to a sample. The ligand-exchange reagents displace cyanide ions (CN⁻) from weak and intermediate strength metallo-cyanide complexes. Figure 1 is a representation of the available cyanide flow scheme.

In the FIA system, a 200- μ L aliquot of the pretreated sample is injected into the flow injection manifold. The addition of hydrochloric acid (HCl) converts cyanide ion to hydrogen cyanide (HCN). The HCN diffuses through a gas diffusion (GD) membrane into an alkaline-receiving solution where it is converted back to cyanide ion (CN⁻). The amount of cyanide ion in the alkaline-receiving solution is measured amperometrically (AMP) with a silver-working electrode, silver/silver chloride reference electrode, and platinum counter electrode at an applied potential of zero volts. The current generated in the cell is proportional to the concentration of cyanide in the original sample, as determined by calibration. The analysis time is 90 seconds per sample.

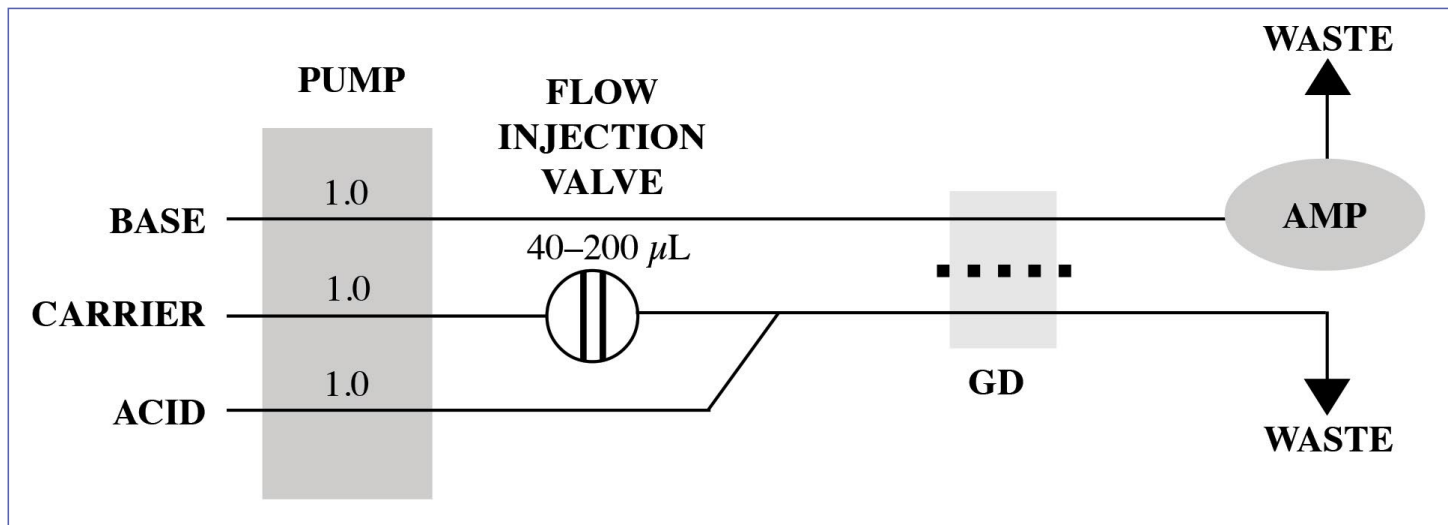


Figure 1. Available Cyanide Flow Injection Analysis Flow Scheme

Single-Laboratory Study

Initially, ALPKEM conducted a single-laboratory validation study in order to refine the method and demonstrate the method's specificity and selectivity. The results of that study are available from the USEPA in the Report of the Draft Method OIA-1677 Single-Laboratory Validation Study.⁵ The single-laboratory study consisted of three groups of tests to establish (1) the ability of Method OIA-1677 to measure the cyanide from various metalocyanide complexes, (2) the ability of Method OIA-1677 to identify cyanide in the presence of interferences, and (3) the recovery and precision of Method OIA-1677 compared to USEPA Method 335.1 and SM 4500 CN- I.

To determine the method's ability to measure cyanide from various metallo-cyanide complexes, two different concentrations (0.2 and 2.0 mg/L) of 15 different metallo-cyanide complexes were analyzed individually in triplicate, for a total of 90 analyses (see Table 1). Method OIA-1677 yielded recoveries ranging from 97% to 104% for seven of the 15 complexes (zinc, cadmium, copper, silver, nickel, and mercury). As with the currently approved methods for available cyanide, Method OIA-1677 did not determine cyanide from the thermodynamically and kinetically stable complexes of cobalt, gold, and iron.

To compare the performance of Method OIA-1677 to the performance of currently approved methods, two different concentrations of the same 15 metallo-cyanide complexes given above were analyzed individually in triplicate by the USEPA-approved CATC Method 335.1 SM 4500 CN- I and Method OIA-1677. This resulted in a another set of 180 data points (see Table 2). These results show improved recoveries and improved precision for Method OIA-1677 compared to both the SM 4500 CN- I and the CATC methods for selected analytes. For the mercury cyanide complexes, recovery improved from 59% for SM 4500 CN- I to 99% for Method OIA-1677, and high levels of interferences in the nickel and silver determinations showed similar improvements over the CATC method. Data for zinc, cadmium, and copper were comparable among the three cyanide procedures. Again, there were no recovery differences for the thermodynamically and kinetically stable cobalt, gold, or iron cyanide complexes.

Table 1. Species Dependent Cyanide Recoveries (%) Obtained with the Flow Injection Available Cyanide Method OIA-1677^a

Species	0.200 mg/L CN ⁻		2.00 mg/L CN ⁻	
	Recovery (%)	Relative SD (%)	Recovery (%)	Relative SD (%)
Zn(CN) ₄] ²⁻	97.4	(0.7)	98.5	(0.7)
[Cd(CN) ₄] ²⁻	100.0	(0.8)	100.0	(0.2)
[Cu(CN) ₄] ³⁻	100.9	(1.3)	99.0	(0.6)
[Ag(CN) ₄] ⁻	101.8	(0.9)	100.0	(0.5)
[Ni(CN) ₄] ²⁻	104.3	(0.2)	103.0	(0.5)
[Hg(CN) ₄] ²⁻	100.0	(0.6)	99.0	(0.3)
Hg(CN) ₂	103.4	(0.4)	98.0	(0.3)
[Fe(CN) ₆] ⁴⁻	0.0		0.0	
[Fe(CN) ₆] ³⁻	0.0		0.0	
[Pd(CN) ₄] ²⁻	0.0		0.15	(4.9)
[Pt(CN) ₄] ²⁻	0.0		0.0	
[Pt(CN) ₆] ²⁻	0.0		0.0	
[Ru(CN) ₆] ⁴⁻	0.0		0.0	
[Au(CN) ₂] ⁻	1.3	(0.0)	0.0	
[Co(CN) ₆] ³⁻	2.9 ^b	(0.0)	2.0 ^b	(0.0)

Table 2. Species Dependent Cyanide Recoveries (%) Obtained with the ASTM WAD and the USEPA CATC Distillation Methods^a

Species	0.200 mg/L CN ⁻		2.00 mg/L CN ⁻	
	WAD	CATC	WAD	CATC
Zn(CN) ₄] ²⁻	102.3	(1.9)	99.5	(0.5)
[Cd(CN) ₄] ²⁻	101.5	(1.5)	103.8	(1.3)
[Cu(CN) ₄] ³⁻	97.3	(1.1)	97.7	(1.9)
[Ag(CN) ₄] ⁻	97.8	(1.6)	97.8	(1.2)
[Ni(CN) ₄] ²⁻	105.8	(1.2)	104.2	(3.5)
[Hg(CN) ₄] ²⁻	71.3	(2.5)	95.8	(2.5)
Hg(CN) ₂	38.8	(9.0)	98.0	(5.9)
[Fe(CN) ₆] ⁴⁻	0.0		0.0	
[Fe(CN) ₆] ³⁻	0.0		0.0	
[Pd(CN) ₄] ²⁻	32.6	(6.5)	0.0	
[Pt(CN) ₄] ²⁻	0.0		0.0	
[Pt(CN) ₆] ²⁻	0.0		0.0	
[Ru(CN) ₆] ⁴⁻	0.0		0.0	
[Au(CN) ₂] ⁻	10.0		0.0	
[Co(CN) ₆] ³⁻	0.0		1.9 ^b	(2.0)
			1.9 ^b	(4.3)

^a % relative standard deviation (n=3) is given in parentheses.

^b Commercial product contains some free cyanide.

Interfering Species	Interferent Concentration 20 mg/L	Interferent Concentration 200 mg/L
CH ₃ CHO	0.0	0.0
C ₆ H ₁₂ O ₆	0.0	0.0
C ₃ H ₈ O ₃	0.0	0.0
OCl ⁻	0.0	0.0 ^b
OCN ⁻	0.0	0.0
SO ₃ ²⁻	0.0	2.0
SCN ⁻	0.0	1.0
SO ₄ ²⁻	0.0	0.0
S ₂ O ₃ ²⁻	0.0	2.0
Cl ⁻	0.0	0.0
Br ⁻	0.0	0.0
I ⁻	0.0	0.0
CO ₃ ²⁻	0.0	0.0
NH ₄ ⁻	0.0	0.0
NO ₂ ⁻	0.0	0.0
NO ₃ ⁺	0.0	0.0

Table 3. Response of the Available Cyanide Method to the Presence of Possible Interferents

To test the ability of Method OIA-1677 to identify cyanide in the presence of interfering species, 16 different possible interferents were tested at two different concentrations (20 and 200 mg/L) (see Table 3). Additionally, two different concentrations of 16 interferents were analyzed in triplicate for a single cyanide test solution, resulting in a second set of 96 analyses (see Table 4). Even in the presence of these interferents, cyanide recoveries ranged from 99% to 103% using this method. Tables 5 and 6 present data of interferents and various masking agents. Tables 7 and 8 present the data on the sulfide interferents. Figures 2 and 3 are calibration curves covering the dynamic range of the method.

^a In µg/L (ppb) apparent cyanide.

^b After the addition of ascorbic acid; otherwise 3 µg/L apparent cyanide.

Table 4. Determination of CN⁻ by the Available Cyanide Method in the Presence of Possible Interferents^a (% relative standard deviation (n=3) is given in parentheses)

Species	Ratio ^b	CN ⁻ Found (mg/L)	Ratio ^b	CN ⁻ Found (mg/L)
CH ₃ CHO	59	0.157 (0.5)	590	0.019 (1.5)
C ₆ H ₁₂ O ₆	14	0.199 (1.2)	140	0.200 (2.5)
C ₃ H ₈ O ₃	28	0.200 (2.2)	280	0.201 (1.0)
OCl ⁻	50	0.0	500	0.0
OCN ⁻	62	0.199 (0.1)	620	0.198 (0.2)
SO ₃ ²⁻	32	0.073 (0.8)	320	0.003 (7.9)
SCN ⁻	45	0.197 (0.2)	450	0.200 (0.4)
SO ₄ ²⁻	27	0.201 (1.4)	270	0.200 (0.6)
S ₂ O ₃ ²⁻	23	0.199 (1.0)	230	0.200 (0.2)
Cl ⁻	73	0.198 (1.2)	730	0.200 (2.1)
Br ⁻	33	0.199 (1.0)	330	0.199 (0.2)
I ⁻	20	0.195 (1.2)	200	0.201 (0.2)
CO ₃ ²⁻	43	0.199 (1.2)	430	0.198 (0.6)
NH ₄ ⁺	144	0.200 (1.7)	1440	0.198 (0.9)
NO ₂ ⁻	56	0.200 (1.0)	560	0.201 (0.6)
NO ₃ ⁻	42	0.199 (0.4)	420	0.197 (2.4)

^a All samples containing 0.200 mg/L CN⁻ and 20 or 200 mg/L of the potentially interfering species were kept in the refrigerator at 4°C for 12 hours before analysis.

^b Molar concentration ratio: species/CN⁻

Species	Ratio	CN ⁻ Found (mg/L)	
		Untreated	Treated ^b
CH ₃ CHO	0.59 590	0.157 (0.5) 0.019 (1.5)	0.190 (1.1) 0.142 (1.3)
OCl ⁻	5 50	0.000 0.000	0.120 (0.2) 0.000
SO ₃ ²⁻	3.2 32	0.168 (0.7) 0.073 (0.8)	0.193 (0.6) 0.171 (0.7)

Table 5. Response of the Available Cyanide Method to the Presence of Possible Interferents With and Without Interferent Treatment^a (% relative standard deviation (n=3) is given in parentheses)

^a All samples contained 0.200 mg/L CN⁻ and were refrigerated at 4°C for 12 hours prior to analysis.

^b With the interference removal method (for CH₃CHO: 2 mL of 3.5% ethylene diamine solution per 100 mL of sample; for OCl⁻ and SO₃²⁻: 50 mg of ascorbic acid per 100 mL of sample).

Note: Ethylene diamine or ascorbic acid was added within one minute after mixing CN⁻ with the interferent.

Table 6. Response of the Flow Injection/Ligand Exchange Available Method to the Presence of Potential Reductants^a (% relative standard deviation (n=3) is given in parentheses)^a

Sample + Reductant	Molar Ratio (Red/CN ⁻)	Cyanide Concentration (ppm)			
		0 h	24 h	48 h	72 h
0.2 ppm CN ⁻ + 0.010 g Sodium Arsenite	100	0.197 (0.52)	0.197 (1.56)	0.197 (0.26)	0.198 (0.62)
0.2 ppm CN ⁻ + 0.025 g Sodium Arsenite	250	0.198 (0.44)	0.196 (1.34)	0.198 (0.51)	0.196 (0.99)
0.2 ppm CN ⁻ + 0.050 g Sodium Arsenite	500	0.198 (2.70)	0.191 (0.52)	0.198 (1.21)	0.198 (0.25)
0.2 ppm CN ⁻ + 0.010 g Ascorbic Acid	74	0.200 (1.79)	0.181 (0.69)	0.176 (0.49)	0.173 (0.48)
0.2 ppm CN ⁻ + 0.025 g Ascorbic Acid	185	0.197 (0.62)	0.182 (0.16)	0.168 (0.10)	0.139 (0.58)
0.2 ppm CN ⁻ + 0.050 g Ascorbic Acid	370	0.196 (0.40)	0.172 (0.33)	0.141 (0.62)	0.063 (0.67)
0.2 ppm CN ⁻ + 0.010 g Oxalic Acid	103	0.199 (0.56)	0.204 (0.42)	0.199 (0.31)	0.201 (0.88)
0.2 ppm CN ⁻ + 0.025 g Oxalic Acid	258	0.196 (0.38)	0.196 (0.38)	0.198 (0.76)	0.199 (1.61)
0.2 ppm CN ⁻ + 0.050 g Oxalic Acid	516	0.200 (0.160)	0.200 (0.18)	0.201 (0.18)	0.201 (0.48)
0.2 ppm CN ⁻ + 0.010 g Sodium Thiosulfate	82	0.202 (0.26)	0.198 (0.86)	0.198 (0.94)	0.198 (0.36)
0.2 ppm CN ⁻ + 0.025 g Sodium Thiosulfate	205	0.203 (0.34)	0.203 (0.57)	0.202 (0.75)	0.200 (0.51)
0.2 ppm CN ⁻ + 0.050 g Sodium Thiosulfate	410	0.202 (0.64)	0.200 (0.53)	0.197 (0.39)	0.200 (0.66)

^a All samples were refrigerated at 4°C.

Table 7. Determination of Cyanide by the Available Cyanide Method in the Presence of Sulfide (S²⁻)^a
 (% relative standard deviation (n=3) is given in parentheses)

Ratio S ²⁻ /CN ⁻	CN ⁻ Found (mg/L)		Ratio S ²⁻ /CN ⁻	CN ⁻ Found (mg/L)	
	Filtered After PbCO ₃ Addition	Unfiltered for 12 hr		Filtered After PbCO ₃ Addition	Unfiltered for 12 hr
81	0.185 (1.5)	0.0	810	0.173 (0.60)	0.0

^a Samples containing 0.200 mg/L CN⁻ and 20 or 200 mg/L S²⁻ were treated with PbCO₃ until a test for S²⁻ was negative. For filtration of PbS, Acrodisc 0.2-µM syringe filters were used. Samples (filtered and unfiltered) were kept at 4°C for 12 hours before analysis.

Table 8. Recovery of Cyanide by the Flow Injection/Ligand Exchange Available Cyanide Method in the Presence of Sulfide (S²⁻) as a Function of Time^a

Time (hrs)	Ratio S ²⁻ /CN ⁻	CN ⁻ Found (mg/L)		CN ⁻ Found (mg/L)	
		Analyzed Immediately After Filter	Analyzed After 12 hrs	Analyzed Immediately After Filter	Analyzed After 12 hrs ^b
2	81	0.181	0.185	0.178	0.173
4	81	0.163	0.168	0.135	0.131
6	81	0.159	0.157	0.082	0.085

^a Samples containing 0.200 mg/L CN⁻ and 20 or 200 mg/L S²⁻ were treated with PbCO₃, and the precipitated PbS was filtered after 2, 4, and 6 minutes of contact time.

^b No cyanide was detected in the unfiltered samples analyzed after 12 hours indicating complete reaction of CN⁻ + S²⁻ to SCN⁻.

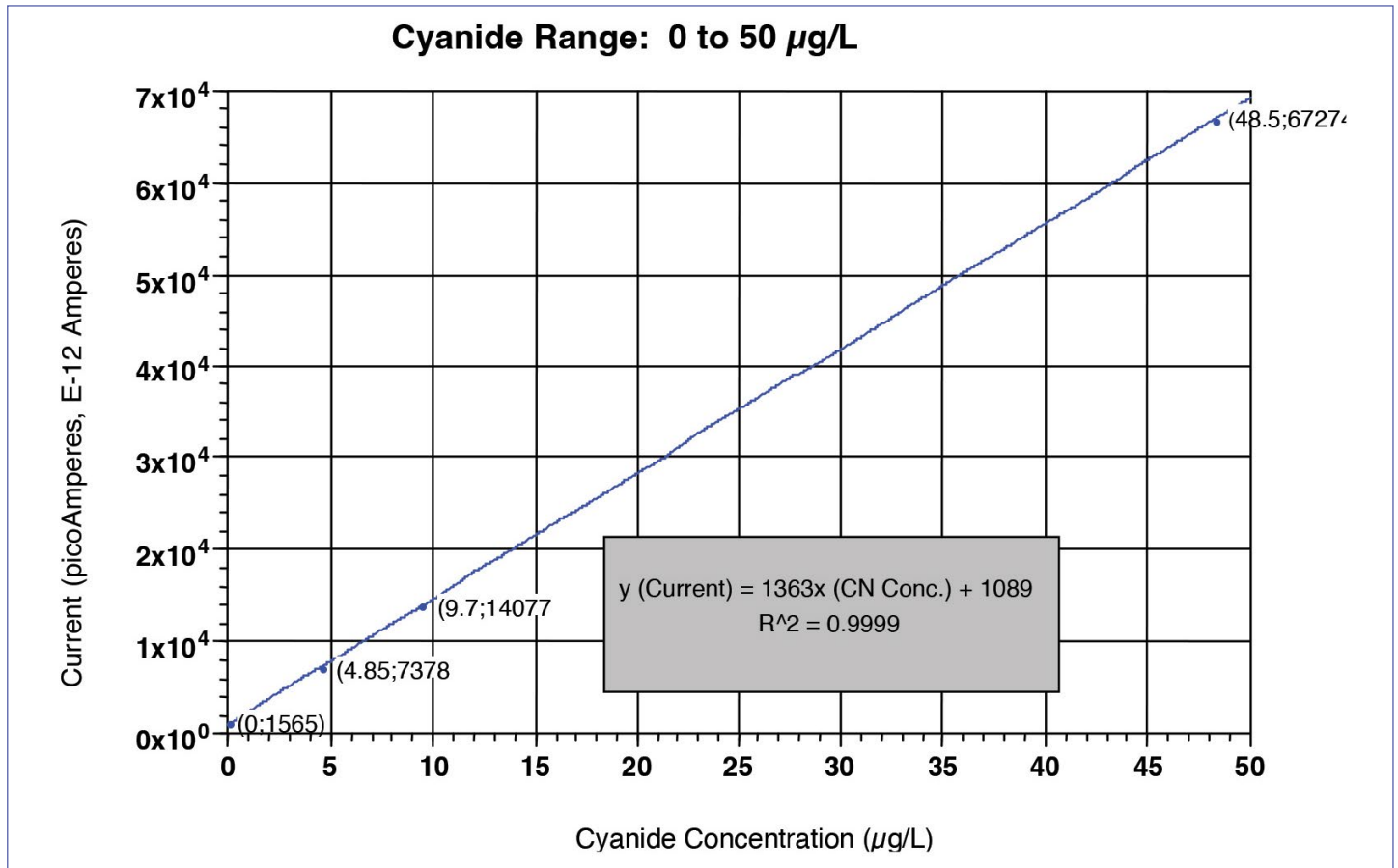


Figure 2. Available Cyanide Calibration Curve for 0 to 50 µg/L.

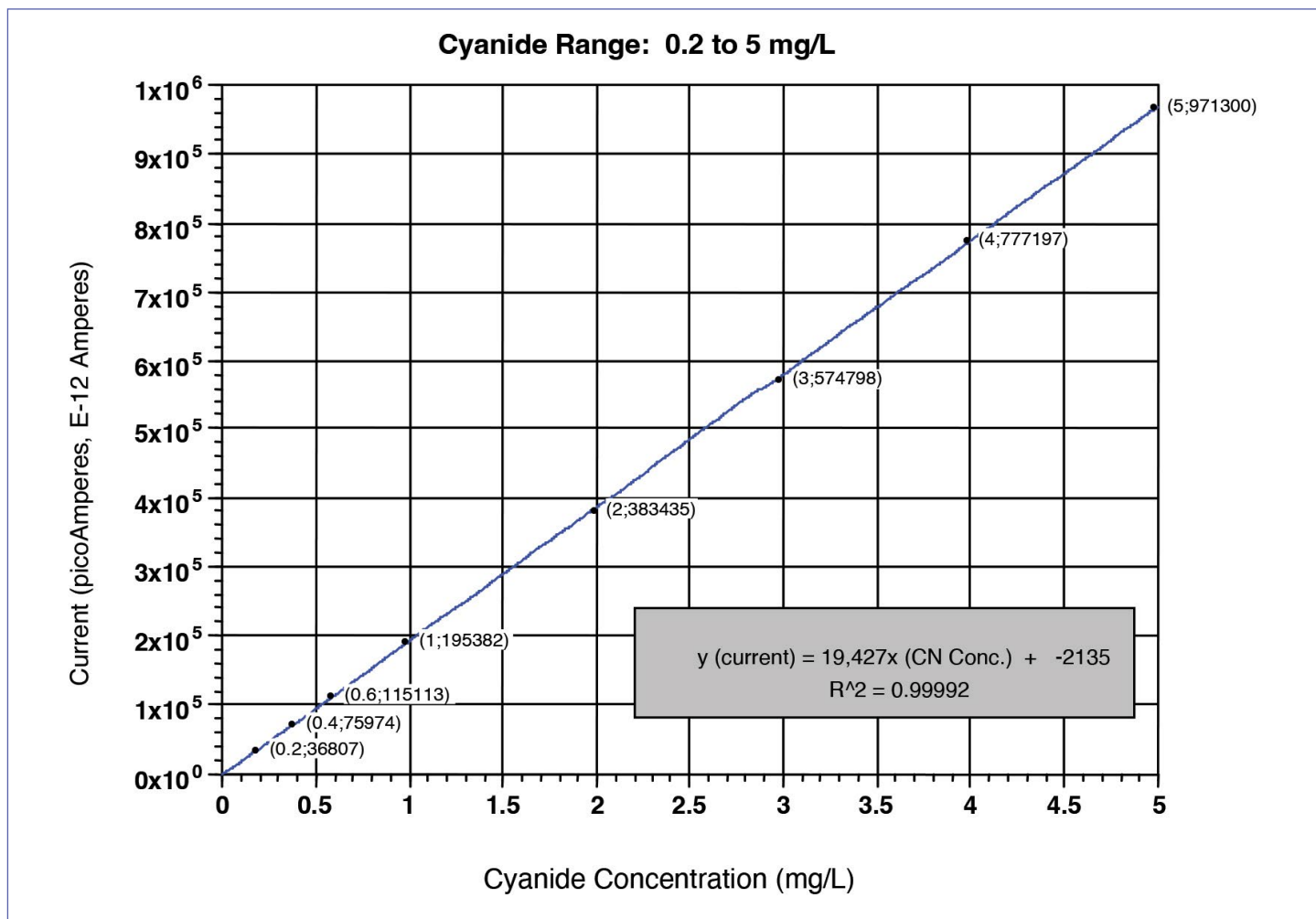


Figure 3. Available Cyanide Calibration Curve for 0.2 to 5 mg/L.

InterLaboratory Study

The interlaboratory method validation study was performed by nine laboratories, working cooperatively as the WAD Cyanide Round Robin Group. Each laboratory analyzed an identical set of nine field samples using Method OIA-1677. These field samples were collected from nine different effluents, ranging from a publicly owned treatment works (POTW) to an industry deemed likely to contain cyanide in its effluent. Each sample was analyzed in triplicate using the FIA procedure for a total of 243 analyses (nine samples performed in triplicate by nine laboratories). Field sample results are presented in Table 9. The complete study results are detailed in the report titled The Interlaboratory Validation of Method OIA-1677 and are available from the USEPA5. The purpose of the interlaboratory study was (1) to confirm the performance of Method OIA-1677 in multiple laboratories, (2) to assess Method OIA-1677 interlaboratory data variability, and (3) to develop Method OIA-1677 quality control (QC) acceptance criteria.

Along with the analysis of the field samples, each laboratory performed all required QC analyses, including initial calibration; calibration verification; determination of initial precision and recovery; blank analysis; determination of ongoing precision and recovery (OPR); determination of matrix spike recovery and matrix spike duplicate recovery (MS/MSD) in each sample type; and assessment of recovery of cyanide as $Hg(CN)_2$ spiked into samples (ligand-exchange reagent performance check or LERPC). In addition, each laboratory performed a minimum detection level (MDL) study.

The relative standard deviation (RSD) of results across all laboratories and all samples was 12%. The mean sample recoveries across all effluent types tested was 96%, and the MS and MSD mean recoveries were 99% across all effluent types tested. According to the July 7, 1998 Federal Register proposal, "these results exceed the generally accepted norms for analytical chemistry results."

Table 9. Available Cyanide Recoveries From Various Aqueous Matrices as Determined During the Interlaboratory Study

Sample Description	Sample CN- Concentration	Added CN- Concentration ^a	Average % Recovery	RSD
Reagent Water with 0.01M NaOH	0.0 µg/L	100 µg/L as KCN	108	4
POTW Secondary Effluent	3.0 µg/L	100 µg/L as KCN, 2 mg/L as [Pt(CN) ₆] ⁺	102	7
Petroleum Refinery Secondary Effluent	9.9 µg/L	2 mg/L as KCN, 5 mg/L as [Fe(CN) ₆] ⁺	87	21
Coke® Plant Secondary Effluent	14.0 µg/L	50 µg/L as KCN	95	4
Rolling Mill Direct Filter Effluent	4.0 µg/L	None	80	41
Metals Finishing Indirect Primary Effluent	1.0 µg/L	200 µg/L as KCN, 2 mg/L as KSCN	92	16
Reagent Water with 0.01M NaOH	0.0 µg/L	200 µg/L as KCN	101	8
Reagent Water with 0.01M NaOH	0.0 µg/L	10 mg/L as KCN, 10 mg/L as [Pt(CN) ₆] ⁺	103	2
Mining Tailing Pond Effluent	842.0 µg/L	4 mg/L as KCN	98	3

^a Cyano-complexes of Pt and Fe were added to the POTW and petroleum refinery effluents, respectively; thiocyanate was added to the metals finishing effluent to demonstrate that the cyanide analysis system does not determine these forms of cyanide.

Data from the interlaboratory study were used to develop QC acceptance criteria for Method OIA-1677. The interlaboratory report fully describes the laboratory procedures and QC calculations. The QC in Method OIA- 1677 is more extensive than that in currently approved methods for CATC, containing all of the standardized QC tests proposed in the USEPA's streamlining initiative (62 FR 14976) and used in the 40 CFR 136 Appendix A methods.

Criteria were developed for initial precision and recovery (IPR), ongoing precision and recovery (OPR), and recovery of cyanide as Hg(CN)₂ spiked into reagent water samples (LERPC). QC acceptance criteria for the IPR, OPR, MS, MSD, and relative percent difference (RPD) for the MS and MSD were calculated using procedures described in the USEPA's Streamlining Guide (see Table 10). In addition to those procedures, QC acceptance criteria also were developed for Hg(CN)₂ at the upper level of the analytical range. Criteria for this LERPC test were developed according to the same procedure as the IPR test.

Table 10. Available Cyanide QC Acceptance Criteria as Determined During the Interlaboratory Study

Parameter	Required Recovery Range (%)	Precision
Initial Precision and Recovery	92 - 122	< 5.1% RSD
Ongoing Precision and Recovery	82 - 132	N/A
Calibration Verifications	86 - 118	N/A
Matrix spike/Matrix Spike Duplicate	82 -130	<11% RSD

Nine single-laboratory MDL studies were performed as part of the effort to determine MDLs and minimum levels (MLs). The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. To determine the MDL, the laboratories were required to follow the procedure in 40 CFR Part 136, Appendix B.

In the Appendix B procedure, seven aliquots of reagent water are spiked with the analyte or analytes of interest and analyzed by the proposed method. For the MDL studies, KCN was used as the spiking material. Spike levels were in the range of one to five times the estimated detection limit. Following addition of KCN, cyanide levels in each of the seven aliquots were determined. The combined MDL of all the laboratories in the study was determined to be 0.5 µg/L CN⁻. A representative single-laboratory MDL data set is included in Table 11.

Table 11. Single-Laboratory Determination of Minimum Detection Level and Minimum Level of the Available Cyanide Method

Sample	Sample ID	pico Amps	µg/L CN	Remarks
	Cal.	103	0.0	
	Cal.	2411	3.0	
	Cal.	3713	5.0	Linear
	Cal.	5050	7.0	R = 0.9991
1	3 µg/L	2412	3.17	
2	3 µg/L	2355	3.09	
3	3 µg/L	2367	3.11	
4	3 µg/L	2332	3.06	
5	3 µg/L	2351	3.09	
6	3 µg/L	2305	3.02	
7	3 µg/L	2302	3.02	
Avg	3 µg/L	2346	3.08	
Standard Deviation		38.1	0.054	
RSD			1.76%	
Minimum Detection Level = $3.14 \times 0.054 = 0.17$ ppb				
Minimum Level (ML) = $3.18 \times 0.17 = 0.5$ ppb				

The ML is defined as the level at which the entire analytical system produces a recognizable signal and an acceptable calibration point. The ML is determined by multiplying the MDL by 3.18 and rounding the resulting value to the number nearest to $[1, 2, \text{ or } 5] \times 10^n$, where n is an integer. The ML for Method OIA-1677 was determined to be 1.0 µg/CN⁻. Results of the MDL studies, along with the relevant calculations are detailed in the interlaboratory study report⁵.

Conclusion

Method OIA-1677 demonstrates greater specificity for cyanide in matrices where interferences have been encountered using CATC methods. In addition, Method OIA-1677 measures cyanide at lower concentrations and offers improved precision and accuracy over currently approved CATC methods. This proposed method offers improved laboratory safety and reduces laboratory waste compared to currently approved CATC methods and, of particular note, eliminates distillation, thereby significantly reducing the possibility of laboratory accidents. Because the determinative step in Method OIA-1677 is amperometry, the chemical reagents employed for colorimetric determinations under currently approved methods are unnecessary. This significantly reduces the generation of hazardous waste by the laboratory. Cyanide analysis by Method OIA-1677 is also significantly more rapid than by currently approved methods. Proposal of Method OIA-1677 has been the result of a collaborative effort between OI Analytical and the USEPA. It is a method that applies the innovative technologies of ligand exchange, flow injection analysis, and amperometric detection to the determination of available cyanide, a pollutant regulated under the CWA. Approval of Method OIA-1677 will allow the use of these technologies to overcome interference problems commonly encountered in the determination of available cyanide and would thereby provide more reliable results for compliance determinations.

References

1. 40 CFR 401.15. Toxic Pollutants.
2. 40 CFR 423. Appendix.
3. Article by Goldberg, et al. Available from the USEPA Sample Control Center, 300 N. Lee Street, Alexandria, VA 22314, (703) 519-1140.
4. Guide to Method Flexibility and Approval of EPA Water Methods, EPA-821-D-96-004, December 1996. Available from the USEPA Water Resource Center, (202) 260-7786.
5. A copy of the supporting documents for Method OIA-1677 are available for review at the USEPA Water Docket, 401 M Street, SW, Washington DC 20460. For access to docket materials, call (202) 260-3027 between 9 a.m. and 3:30 p.m. for an appointment.



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