

Environmental

Determination of cyanide in alkaline solutions using ion chromatography

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Keywords

Electrochemical detection, Dionex IonPac AS7 column, pulsed amperometric detection (PAD), sulfide, silver working electrode, drinking water, wastewater

Goal

To update a method for the determination of cyanide in alkaline solutions using an ion chromatography (IC) system with a Thermo Scientific™ Dionex™ IonPac™ AS7 column and pulsed amperometric detection (PAD)

Introduction

Free cyanide includes hydrogen cyanide (HCN) and cyanide (CN⁻) and is extremely toxic¹ and regulated.

According to the United States Environmental Protection Agency (U.S. EPA), the maximum contaminant level (MCL) of free cyanide in drinking water is 0.2 mg/L.² Total cyanide, which includes free cyanide, weak metal cyanide complexes, and strong metal cyanide complexes, is also regulated because metal cyanide complexes can generate free cyanide. Different methods have been used to determine free cyanide and total cyanide.^{2,3} The EPA methods for the determination of total cyanide^{4,5} generally include two steps:

1. Releasing cyanide from cyanide complexes as hydrocyanic acid (HCN) gas by a reflux-distillation in a strong acid and absorbing the liberated HCN gas in a scrubber (a strongly alkaline solution)
2. Determining cyanide

Ion chromatography (IC) coupled with electrochemical detection (ED) is used for the determination of cyanide,⁶⁻⁸ including direct determination of cyanide in strongly alkaline solutions using IC with DC amperometric detection.⁶ Compared to colorimetric or titration methods, the IC method has fewer interferences. Unfortunately, the DC amperometry method exhibits electrode fouling problems over time. Here we show an updated IC method with pulsed amperometric detection (PAD) for the determination of cyanide in different absorbing solutions, including the 1.25 M NaOH solution specified in U.S. EPA Method 335.2 and the 0.25 M NaOH solution specified in U.S. EPA Method 335.4. Determination of cyanide in drinking water and wastewater using this method is also demonstrated. The improved method uses a 2 mm version of the Dionex IonPac AS7 column and a 50 μ L injection.⁹

Experimental

Equipment and consumables

- Thermo Scientific™ Dionex™ ICS-6000 Dual Channel HPIC™ System with RFIC-EG module, and Electrochemical Detection*
- Thermo Scientific™ Dionex™ AS-AP Autosampler with 250 μ L syringe and tray temperature control
- Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) Software, version 7.2.10
- Thermo Scientific™ Dionex™ ICS-6000/4000 ED Electrochemical Detector (P/N 072042) with Electrochemical Detector Cell (P/N 072044)

Consumables

- Thermo Scientific™ Dionex™ IonPac™ AS7 Analytical column, 2 x 250 mm, (P/N 063097)
- Thermo Scientific™ Dionex™ IonPac™ AG7 Guard column, 2 x 50 mm (P/N 063099)
- Thermo Scientific™ Dionex™ AS-AP Autosampler Vials 10 mL (P/N 074228)
- Thermo Scientific™ Dionex™ ICS-5000+/ICS-6000 ED Electrochemical Conventional Electrode-Silver Electrode (P/N 079856)
- Thermo Scientific™ Dionex™ ICS-6000/4000 ED Electrochemical Detector Reference Electrode-Ag/AgCl pH Reference Electrode (P/N 061879)

* Equivalent results can be achieved using a single channel Dionex ICS-6000 system, Thermo Scientific™ Dionex™ ICS-5000+ system, or Thermo Scientific™ Dionex™ Integrion™ HPIC™ System with electrochemical detection.

Reagent and standards

- Degassed deionized (DI) water, 18 M Ω -cm resistance or better
- Sodium cyanide (NaCN) (>97%), A.C.S. reagent grade or better, for preparing cyanide standards
- Sodium sulfide (Na₂S), nonahydrate $\geq$ 99.99% trace metals basis
- Sodium hydroxide (NaOH) solution (50% w/w Certified), Fisher Chemical (Fisher Scientific [P/N SS254-500](#))
- Sodium acetate, anhydrous, Electrochemical grade, Thermo Scientific Dionex ([P/N 059326](#))

Samples

Simulated samples: mixed cyanide and sulfide standards in DI water, 0.25 N NaOH, and 1.25 N NaOH

A drinking water sample was collected locally. A wastewater (effluent) sample, was obtained from a local water district lab.

Preparation of eluent, standards, and samples

Eluent solution

Prepare 1 L of 0.5 M sodium acetate in 100 mM sodium hydroxide and 0.5% (v/v) ethylenediamine by dissolving 41.0 g of anhydrous sodium acetate in approximately 800 mL of DI water in a plastic bottle. Filter the acetate solution through a 0.2 μ m nylon filter unit. Rinse the bottle using approximately 30 mL of DI water three times and filter. Degas the acetate solution by vacuum and sonication for more than 30 min. Transfer the solution to a 1 L plastic volumetric flask, add 5.2 mL (8 g) of 50% (w/w) NaOH, 5 mL of ethylenediamine, and bring to volume with degassed DI water. Refer to Thermo Scientific Technical Note 71 for detailed information on preparing hydroxide eluents for electrochemical applications.¹⁰

This solution should be immediately transferred to the plastic eluent bottle on the IC system and blanketed with helium or nitrogen at 34 to 55 kPa (5 to 8 psi). Gently swirl the bottle to complete mixing before starting the flow.

Stock standard solutions (1,000 mg/L)

Prepare sulfide and cyanide stock standard solutions by dissolving their salts in 100 mL of 0.2% sodium hydroxide. The stock standards should be stored at 4 °C.

Table 1. Amount of salt used to prepare 100 mL of 1,000 mg/L stock standard solution

	Salt	Amount (g)
Cyanide (CN ⁻)	Sodium cyanide	0.19
Sulfide (S ²⁻)	Sodium sulfide, nonahydrate	0.75

Calibration standards

Dilute 160 g of NaOH solution (50% w/w) with degassed DI water in a 1 L plastic volumetric flask to make a liter of 2 N NaOH solution. Dilute further with degassed DI water to volume to make 0.25 N, 0.50 N, and 1.25 N NaOH solutions.

Prepare three sets of diluted calibration standard solutions from the 1,000 mg/L cyanide stock standard using degassed DI water, 0.25 N NaOH, and 1.25 N NaOH.

Table 2. Calibration standards (mg/L)

Level	1	2	3	4	5
Cyanide (mg/L)	0.005	0.01	0.02	0.04	0.1
Level	6	7	8	9	10
Cyanide (mg/L)	0.2	0.5	1	1.5	2

Samples

Prepare simulated samples by mixing 1,000 mg/L of cyanide and 1,000 mg/L of sulfide stock standard solutions and diluting with DI water, 0.25 N NaOH, and 1.25 N NaOH solutions (Table 3).

Prepare drinking water and wastewater samples by one-to-one dilution with 0.5 N NaOH. Prepare the spiked water samples by spiking cyanide standard in the diluted water samples.

Table 3. Composition of simulated samples

Ratio*	Sulfide (mg/L)	Cyanide (mg/L)	Matrix
10 to 1	1	0.01	Water, basic
	1	0.01	0.25 N NaOH
	1	0.01	1.25 N NaOH
	10	1	Water, basic
	10	1	0.25 N NaOH
	10	1	1.25 N NaOH
1 to 5	0.02	0.1	Water, basic
	0.02	0.1	0.25 N NaOH
	0.02	0.1	1.25 N NaOH
	0.2	1	Water, basic
	0.2	1	0.25 N NaOH
	0.2	1	1.25 N NaOH

*Sulfide to cyanide ratio

Instrumentation

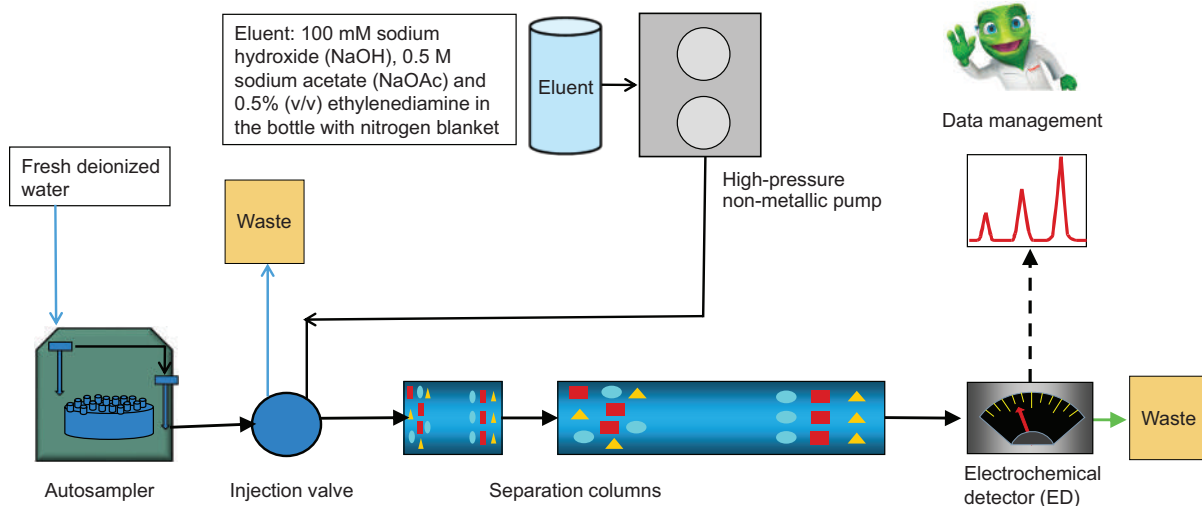


Figure 1. Schematic diagram of an IC system for the determination of cyanide

Table 4. Chromatography conditions

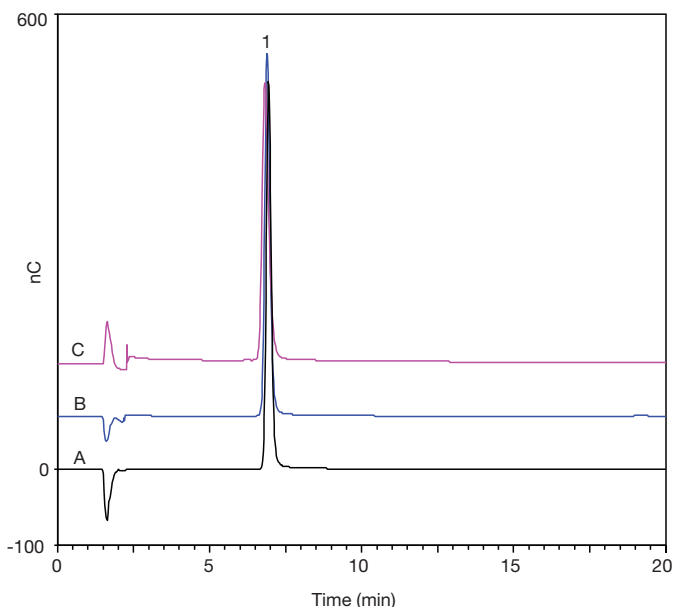
Columns	Dionex IonPac AS7 Analytical column, 2 x 250 mm		
	Dionex IonPac AG7 Guard column, 2 x 50 mm		
Eluent	100 mM sodium hydroxide (NaOH), 0.5 M sodium acetate (NaOAc), and 0.5% (v/v) ethylenediamine		
Flow rate	0.25 mL/min		
Injection volume	50 µL (full loop)		
Column temp.	30 °C		
Sample tray temp.	4 °C		
Detection	Electrochemical detection: PAD mode		
Working electrode	Conventional silver		
Reference electrode	Ag/AgCl pH, Ag mode		
Run time	20 min		
Waveform	Silver (sulfide, cyanide, iodide, thiosulfate)		
	Time (s)	Potential (V) vs. Ag/AgCl	Integration
	0.00	-0.10	
	0.20	-0.10	Start
	0.90	-0.10	End
	0.91	-1.00	
	0.93	-0.30	
	1.00	-0.30	

Results and discussion

Separation

The Dionex IonPac AS7 column is designed for the determination of a wide range of anions, including cyanide. It can be used to analyze high ionic strength samples and can be used with a wide range of eluents (pH 0–14 eluents and eluents containing organic solvents from 0 to 5% in concentration). It is well suited for the determination of cyanide in a strongly alkaline scrubber solution without dilution or pretreatment. Figure 2 shows the chromatograms of a 1 mg/L cyanide standard in

different matrices using 0.5 M sodium acetate in 100 mM sodium hydroxide and 0.5% (v/v) ethylenediamine eluent and electrochemical detection. Cyanide elutes in less than 10 min and is well resolved from the void volume and the baseline dip/peak, which is due to dissolved oxygen, at approximately 17 min. However, cyanide retention time and peak area were impacted by the sample matrices (Table 5 and Figure 2). To determine the cyanide concentration in samples that are in alkaline absorption solutions, the calibration standards need to be prepared in the same absorption solution.



Columns:	Dionex IonPac AG7, 2 x 50 mm and Dionex IonPac AS7, 2 x 250 mm		
Eluent:	100 mM sodium hydroxide (NaOH), 0.5 M sodium acetate (NaOAc), and 0.5% (v/v) ethylenediamine		
Flow rate:	0.25 mL/min		
Inj. volume:	50 µL (Full loop)		
Column temp.:	30 °C		
Sampler temp.:	4 °C		
Detection:	Electrochemical detection with Ag/AgCl reference electrode and silver working electrode		
Samples:	A (black): 1 mg/L cyanide in water B (blue): 1 mg/L cyanide in 0.25 N NaOH C (pink): 1 mg/L cyanide in 1.25 N NaOH		
Peaks:	Sample	Min	
	1-Cyanide	A	6.93
		B	6.88
		C	6.82

Figure 2. Chromatograms of a 1 mg/L cyanide in different matrices

Table 5. Comparison of the response of 1 mg/L of cyanide in different matrices

Matrix	Retention time (min)	Peak area (nC*min)
Water	6.93	94.8
0.25 N NaOH	6.88	94.5
1.25 NaOH	6.82	92.2

Table 6. Linearity and method detection limit for cyanide in different matrices

Analyte	Matrix	Injection volume (µL)	Range (mg/L)	Coefficient of determination* (r^2)	Calculated MDL (mg/L)
Cyanide	Water	50	0.005–2	0.999	0.0001**
Cyanide	0.25 N NaOH	50	0.005–2	0.9999	0.01
Cyanide	1.25 N	50	0.005–2	0.9999	0.03

* Quadratic fit

**MDL = $(t) \times (S)$

Where t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom (t = 3.14 for seven replicates)

S = standard deviation of the replicate analyses

Linearity and method detection limit

In this study, cyanide in the three matrices was calibrated with ten concentration levels ranging from 0.005 to 2 mg/L. In each matrix, a quadratic relationship of peak area to concentration was observed with coefficients of determination (r^2) ranging from 0.9996 to 0.9999 (Table 6 and Figures 3, 4, and 5).

The method detection limit (MDL) usually was determined by performing seven replicate injections of the cyanide standard at a concentration of three to ten times the estimated detection limit. However, the strongly alkaline solution has an impact on ED detection, causing a small negative peak in the background (Figure 6) and increasing the MDL in that matrix. The 0.25 N and 1.25 N NaOH MDLs were estimated by injection of the low concentration cyanide standard (Figure 7). The method is sensitive for regulatory monitoring of cyanide with MDL = 0.0001 mg/L in water, MDL = 0.01 mg/L in 0.25 N NaOH, and MDL = 0.03 mg/L in 1.25 N NaOH.

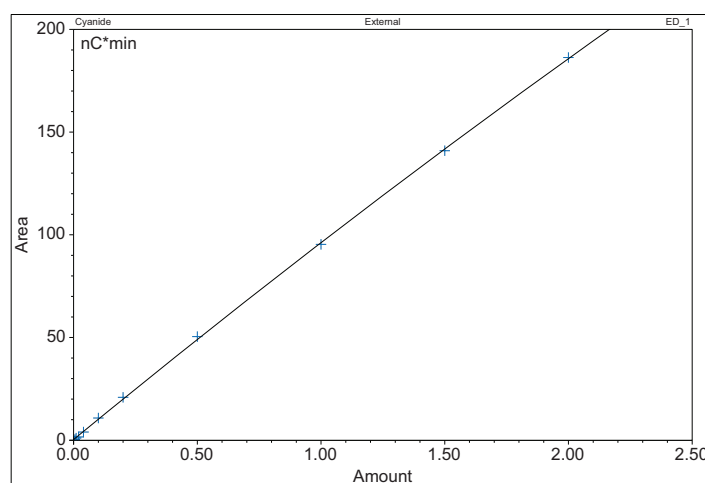


Figure 4. Calibration plot for the determination of cyanide in 0.25 N NaOH

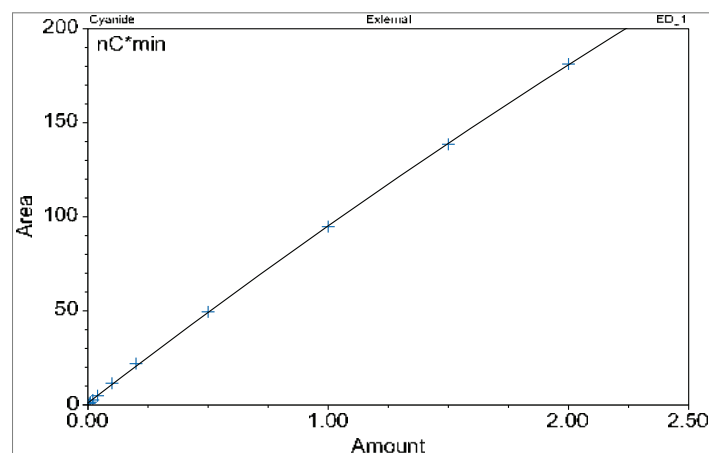


Figure 3. Calibration plot for the determination of cyanide in water

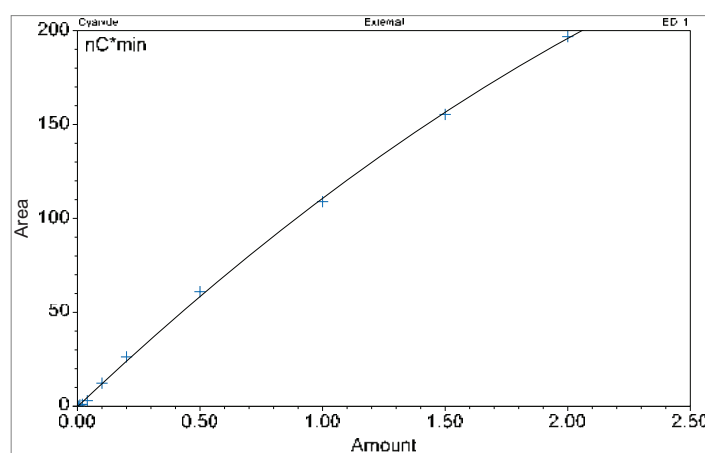
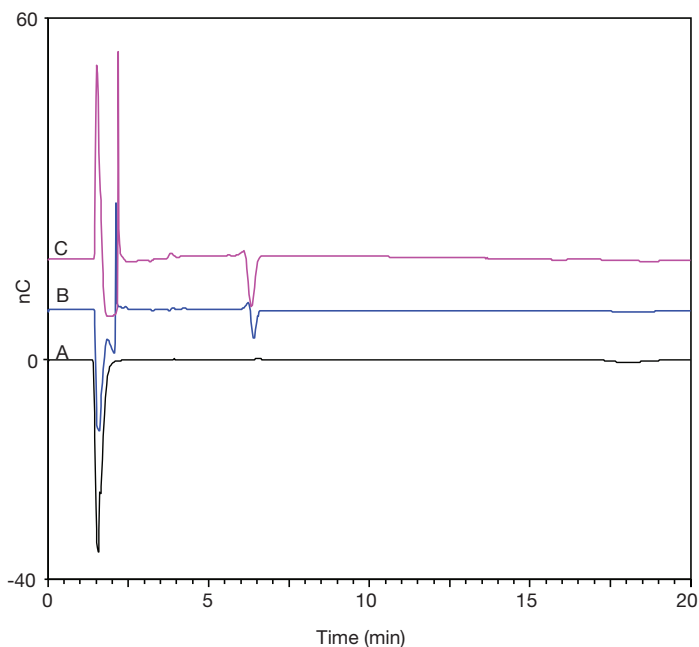


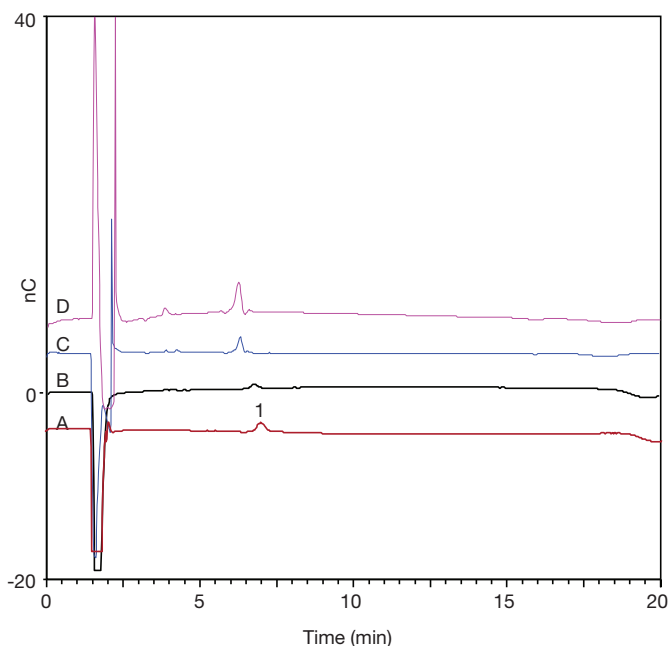
Figure 5. Calibration plot for the determination of cyanide in 1.25 N NaOH



Columns: Dionex IonPac AG7, 2 x 50 mm and Dionex IonPac AS7, 2 x 250 mm
 Eluent: 100 mM sodium hydroxide (NaOH), 0.5 M sodium acetate (NaOAc), and 0.5% (v/v) ethylenediamine
 Flow rate: 0.25 mL/min
 Inj. volume: 50 μ L (Full loop)
 Column temp.: 30 $^{\circ}$ C
 Sampler temp.: 4 $^{\circ}$ C
 Detection: Electrochemical detection with Ag/AgCl reference electrode and silver working electrode

Samples: A (black): DI water
 B (blue): 0.25 N NaOH
 C (pink): 1.25 N NaOH

Figure 6. Chromatograms of different matrices



Columns: Dionex IonPac AG7, 2 x 50 mm and Dionex IonPac AS7, 2 x 250 mm
 Eluent: 100 mM sodium hydroxide (NaOH), 0.5 M sodium acetate (NaOAc), and 0.5% (v/v) ethylenediamine
 Flow rate: 0.25 mL/min
 Inj. volume: 50 μ L (Full loop)
 Column temp.: 30 $^{\circ}$ C
 Sampler temp.: 4 $^{\circ}$ C
 Detection: Electrochemical detection with Ag/AgCl reference electrode and silver working electrode

Samples: A (brown): 0.001 mg/L cyanide in basic water
 B (black): 0.0002 mg/L cyanide in basic water
 C (blue): 0.01 mg/L cyanide in 0.25 N NaOH
 D (pink): 0.03 mg/L cyanide in 1.25 N NaOH

Peak: 1 – Cyanide

Figure 7. Chromatograms of cyanide standards used for the MDL determination

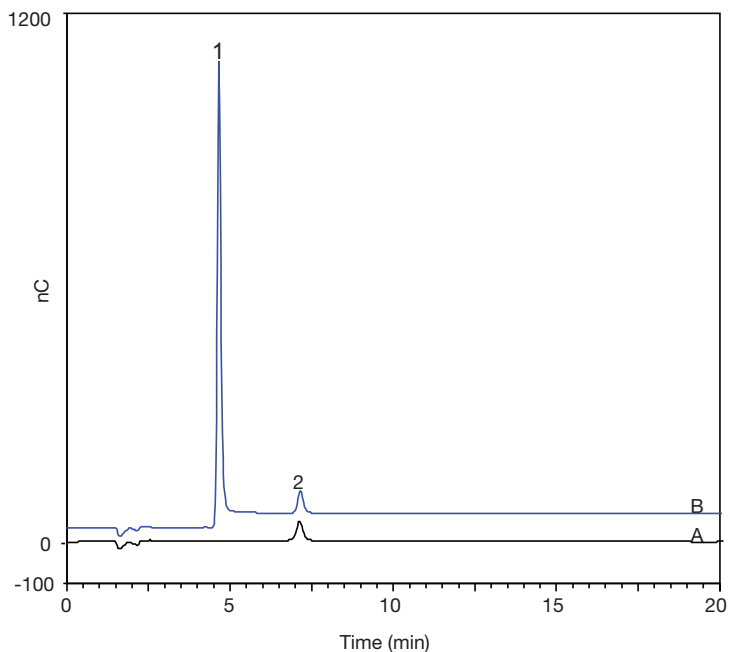
Determination of cyanide in samples

Determination of cyanide in the scrubber solution samples

Combining IC separation with the specificity of electrochemical detection, this IC method is subject to fewer interferences than titrimetric or spectrophotometric methods for the total cyanide determination. The major interference is sulfide, which is also released from some samples and captured by the scrubber solution. The scrubber solutions include the 1.25 M NaOH solution specified in U.S. EPA Method 335.2 and the 0.25 M NaOH solution specified in U.S. EPA Method 335.4.

Figures 8A and 8B show the chromatograms of two simulated samples in 0.25 N NaOH. Figures 9A and 9B show the chromatograms of two simulated samples in 1.25 N NaOH. In both matrices, cyanide and sulfide are well separated. This IC method can be used for the direct determination of cyanide in the absorbing solutions used in U.S. EPA Methods 335.2 and 335.4.

The method accuracy is evaluated by spike recovery of 0.1 and 1 mg/L of cyanide in different matrices with 1/5 or 10 x sulfide (relative to cyanide) in the samples (Table 3). The results (Table 7) show that the method is accurate with 93 to 108% recovery.

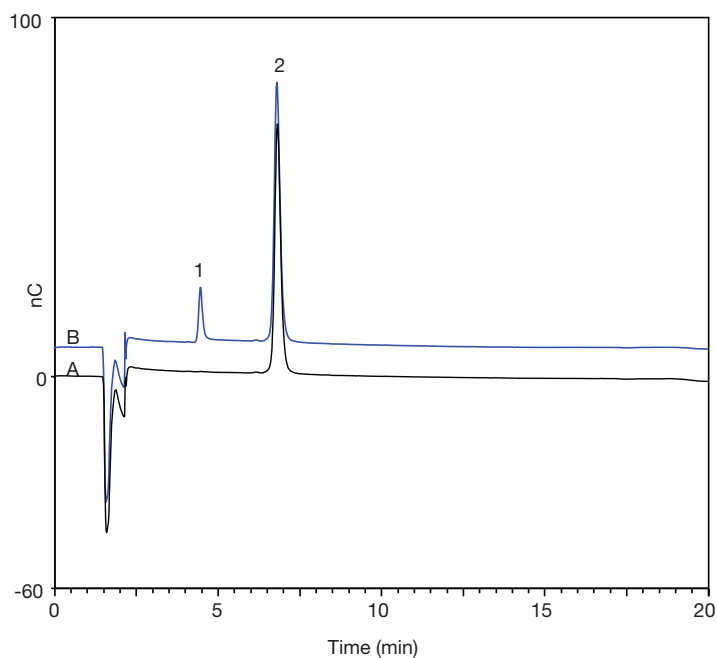


Columns: Dionex IonPac AG7, 2 x 50 mm and Dionex IonPac AS7, 2 x 250 mm
 Eluent: 100 mM sodium hydroxide (NaOH), 0.5 M sodium acetate (NaOAc), and 0.5% (v/v) ethylenediamine
 Flow rate: 0.25 mL/min
 Inj. volume: 50 μ L (Full loop)
 Column temp.: 30 $^{\circ}$ C
 Sampler temp.: 4 $^{\circ}$ C
 Detection: Electrochemical detection with Ag/AgCl reference electrode and silver working electrode

Samples: Cyanide in 0.25 N NaOH
 A (black): 0.1 mg/L cyanide
 B (blue): 0.1 mg/L cyanide and 1.0 mg/L sulfide

Peaks: 1 – Sulfide
 2 – Cyanide

Figure 8A. The chromatograms of 0.1 mg/L cyanide in 0.25 N NaOH with/without sulfide

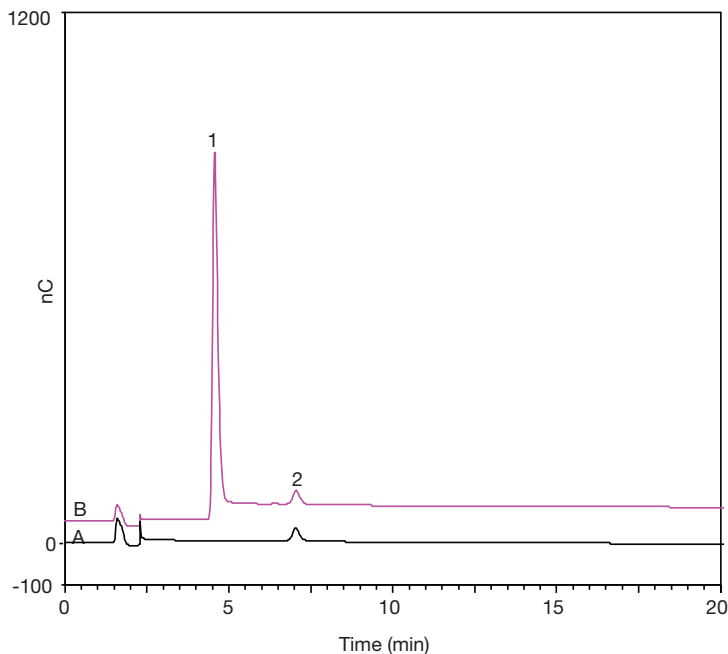


Columns: Dionex IonPac AG7, 2 x 50 mm and Dionex IonPac AS7, 2 x 250 mm
 Eluent: 100 mM sodium hydroxide (NaOH), 0.5 M sodium acetate (NaOAc), and 0.5% (v/v) ethylenediamine
 Flow rate: 0.25 mL/min
 Inj. volume: 50 μ L (Full loop)
 Column temp.: 30 $^{\circ}$ C
 Sampler temp.: 4 $^{\circ}$ C
 Detection: Electrochemical detection with Ag/AgCl reference electrode and silver working electrode

Samples: Cyanide in 0.25 N NaOH
 A (black): 0.1 mg/L cyanide
 B (blue): 0.1 mg/L cyanide and 0.02 mg/L sulfide

Peaks: 1 – Sulfide
 2 – Cyanide

Figure 8B. The chromatograms of 0.1 mg/L cyanide in 0.25 N NaOH with/without sulfide

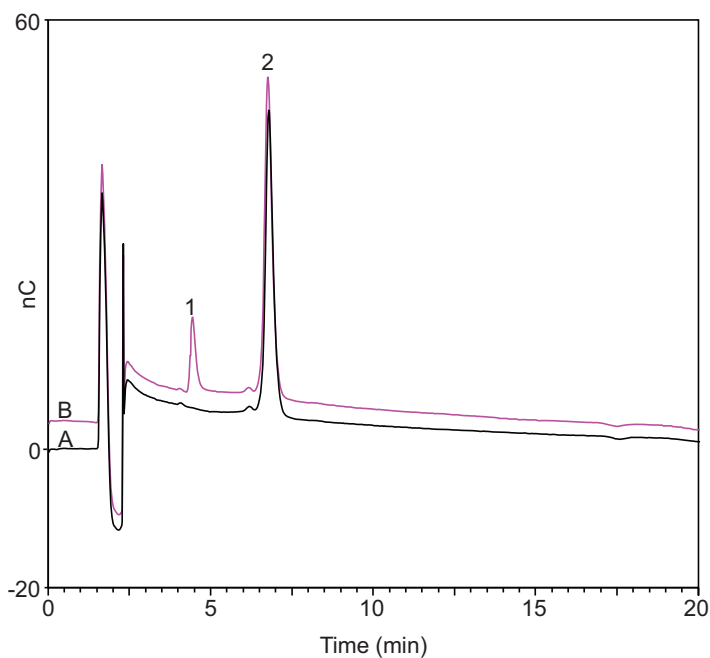


Columns: Dionex IonPac AG7, 2 x 50 mm and Dionex IonPac AS7, 2 x 250 mm
 Eluent: 100 mM sodium hydroxide (NaOH), 0.5 M sodium acetate (NaOAc), and 0.5% (v/v) ethylenediamine
 Flow rate: 0.25 mL/min
 Inj. volume: 50 μ L (Full loop)
 Column temp.: 30 $^{\circ}$ C
 Sampler temp.: 4 $^{\circ}$ C
 Detection: Electrochemical detection with Ag/AgCl reference electrode and silver working electrode

Samples: Cyanide in 1.25 N NaOH
 A (black): 0.1 mg/L cyanide
 B (pink): 0.1 mg/L cyanide and 1.0 mg/L sulfide

Peaks: 1 – Sulfide
 2 – Cyanide

Figure 9A. The chromatograms of 0.1 mg/L cyanide in 1.25 N NaOH with/without sulfide



Columns: Dionex IonPac AG7, 2 x 50 mm and Dionex IonPac AS7, 2 x 250 mm
 Eluent: 100 mM sodium hydroxide (NaOH), 0.5 M sodium acetate (NaOAc), and 0.5% (v/v) ethylenediamine
 Flow rate: 0.25 mL/min
 Inj. volume: 50 μ L (Full loop)
 Column temp.: 30 $^{\circ}$ C
 Sampler temp.: 4 $^{\circ}$ C
 Detection: Electrochemical detection with Ag/AgCl reference electrode and silver working electrode

Samples: Cyanide in 1.25 N NaOH
 A (black): 0.1 mg/L cyanide
 B (pink): 0.1 mg/L cyanide and 0.02 mg/L sulfide

Peaks: 1 – Sulfide
 2 – Cyanide

Figure 9B. The chromatograms of 0.1 mg/L cyanide in 1.25 N NaOH with/without sulfide

Table 7. Spiked recovery of cyanide in sulfide in different matrices

Ratio*	Sulfide (mg/L)	Cyanide (mg/L)	Matrix	Recovery (%)
10 to 1	1	0.01	Water, basic	104
	1	0.01	0.25 N NaOH	101
	1	0.01	1.25 N NaOH	93
	10	1	Water, basic	101
	10	1	0.25 N NaOH	107
	10	1	1.25 N NaOH	96
1 to 5	0.02	0.1	Water, basic	97
	0.02	0.1	0.25 N NaOH	103
	0.02	0.1	1.25 N NaOH	104
	0.2	1	Water, basic	107
	0.2	1	0.25 N NaOH	106
	0.2	1	1.25 N NaOH	108

* Sulfide to cyanide

Determination of cyanide in water samples

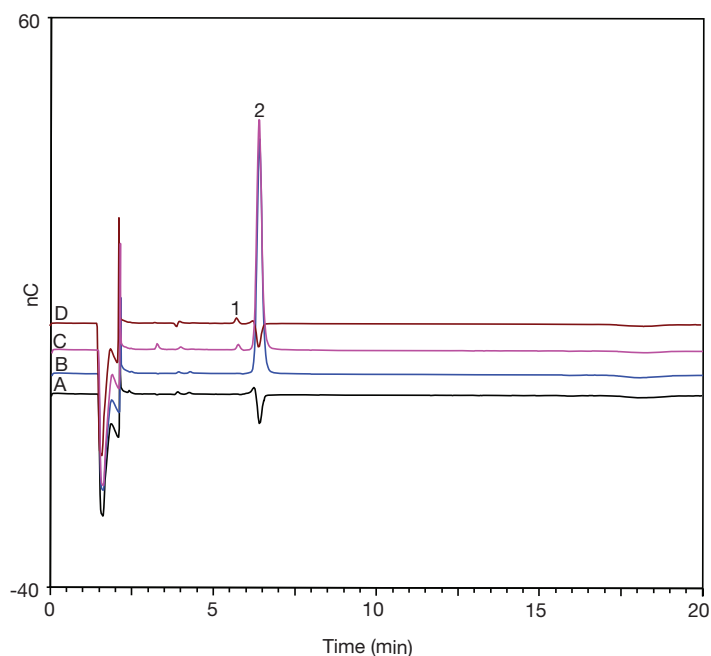
Drinking water and wastewater were tested using this method.

The water samples were diluted with 0.5 N NaOH to ensure the samples have a high pH. Determination of cyanide in drinking water and wastewater were demonstrated by cyanide spiked into the water samples (Table 8 and Figures 10 and 11).

Figure 10 shows the chromatograms of drinking water and cyanide-spiked drinking water. Figure 11 shows the chromatograms of wastewater and cyanide-spiked wastewater.

Although multiple unidentified peaks were found in water and wastewater samples, the cyanide peak is well separated from these unknown peaks. This IC method can be used for the direct determination of cyanide in drinking water and wastewater. No cyanide was found in the two water samples.

The method accuracy was evaluated by spike recovery of 0.1 mg/L of cyanide in the water samples. The results (Table 8) show that the method is accurate with 99% recovery in drinking water and 96% recovery in wastewater.

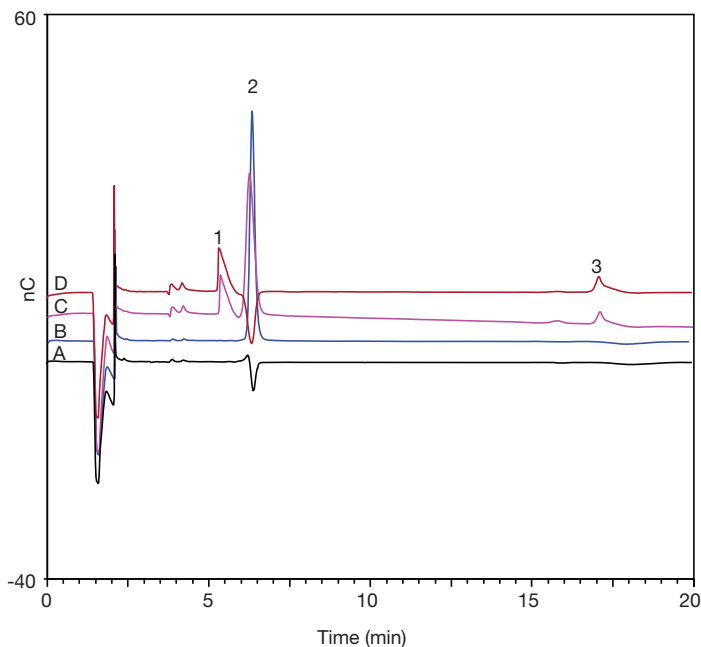


Columns: Dionex IonPac AG7, 2 x 50 mm and Dionex IonPac AS7, 2 x 250 mm
 Eluent: 100 mM sodium hydroxide (NaOH), 0.5 M sodium acetate (NaOAc), and 0.5% (v/v) ethylenediamine
 Flow rate: 0.25 mL/min
 Inj. volume: 50 µL (Full loop)
 Column temp.: 30 °C
 Sampler temp.: 4 °C
 Detection: Electrochemical detection with Ag/AgCl reference electrode and silver working electrode

Samples: A (black): 0.25 N NaOH
 B (blue): 0.1 mg/L cyanide in 0.25 N NaOH
 C (pink): 0.1 mg/L cyanide in sample D
 D (brown): 1:1 mixed drinking water/ 0.5 N NaOH

Peaks: 1 – Unidentified
 2 – Cyanide

Figure 10. Chromatograms of drinking water and cyanide-spiked drinking water



Columns: Dionex IonPac AG7, 2 x 50 mm and Dionex IonPac AS7, 2 x 250 mm

Eluent: 100 mM sodium hydroxide (NaOH), 0.5 M sodium acetate (NaOAc), and 0.5% (v/v) ethylenediamine

Flow rate: 0.25 mL/min

Inj. volume: 50 μ L (Full loop)

Column temp.: 30 $^{\circ}$ C

Sampler temp.: 4 $^{\circ}$ C

Detection: Electrochemical detection with Ag/AgCl reference electrode and silver working electrode

Samples: A (black): 0.25 N NaOH
B (blue): 0.1 mg/L cyanide in 0.25 N NaOH
C (pink): 0.1 mg/L cyanide in sample D
D (brown): 1:1 mixed wastewater/ 0.5 N NaOH

Peaks: 1 – Unidentified
2 – Cyanide
3 – Unidentified

Figure 11. Chromatograms of wastewater and cyanide-spiked wastewater

Table 8. Determination of cyanide in drinking water and wastewater

Sample	Cyanide (mg/L)	Spiked cyanide (mg/L)	Recovery (%)
Drinking water	0	0.1	99
Wastewater	0	0.1	96

Conclusion

This application note demonstrates the validation of an updated 20 min IC method for the determination of total cyanide in alkaline solutions. The improved method changes from DC amperometric detection to PAD and uses a 2 mm version of the Thermo Scientific Dionex IonPac AS7 column set with a 50 μ L injection instead of 4 mm column set with a 200 μ L injection. With the change in column format, the flow rate drops from 1 mL/min to 0.25 mL/min, which saves eluent and reduces waste. Using this method, cyanide is well separated from sulfide, a possible interference in some samples. The method has a quadratic calibration ($r^2 \geq 0.9996$ for the established range of 0.005–2 mg/L) in the cyanide capture solutions used in U.S. EPA Methods 335.2 and 335.4. It is sensitive (MDL = 0.0001 mg/L in water, MDL = 0.01 mg/L in 0.25 N NaOH, and MDL = 0.03 mg/L in 1.25 N NaOH) and accurate (93% to 108% recovery). This method can also be used to determine cyanide in drinking water and wastewater samples.

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