

Direct determination of cyanide in drinking water by ion chromatography with pulsed amperometric detection

Authors: Terri Christison and Jeffrey Rohrer
Thermo Fisher Scientific, Sunnyvale, CA

Introduction

The toxicity of cyanide is well known. Cyanide occurs naturally in many foods (cassava, sorghum, African lima beans, bamboo shoots, bitter almonds, and apricot, cherry, and peach pits) and is naturally generated by microorganisms.¹ Cyanide is used in many industries (e.g., plating and mining) and it can be released into the air from burning coal and plastics. In the U.S., drinking water contamination with cyanide is typically from an industrial source or leached from waste sites.

The U.S. government classifies cyanide as a regulated inorganic contaminant in drinking water (U.S. National Primary Drinking Water Regulations, 40CFR 141.62).² These regulations are enforced by the U.S. EPA and state EPA agencies. Bottled water is classified separately as a food, and is regulated by the FDA Center for Food Safety and Applied Nutrition (CFSAN) division. For community water systems, non-transient non-community water systems (defined as a temporary water system for ≥ 25 people used for less than six months), and drinking water, the maximum contaminant level (MCL) is 200 $\mu\text{g/L}$ cyanide as free cyanide. Typical free cyanide levels are much lower. A 1978 U.S. EPA survey showed that only 7% of drinking water had cyanide concentrations $>10 \mu\text{g/L}$.³



Cyanide is determined as total cyanide (EPA 335.2),⁴ disassociated cyanide, and free (amenable) cyanide. Total cyanide is determined by distillation with acid and an oxidizing agent to generate hydrogen cyanide gas that is captured in a pH 13 sodium hydroxide solution, and then determined by a colorimetric or titration method.

The EPA approved free cyanide methods use spectrophotometry (335.1),⁵ colorimetry (335.3),⁶ and ion-selective electrode detection (Standard Methods SM-4500-CN-F).⁷ The colorimetric and spectrophotometric methods require distillation and have many interferences, including difficulty with high-pH solutions, oxidizers, and sulfur-bearing compounds. The ion-selective electrode method does not require distillation, but it is very matrix sensitive. Ion chromatography (IC) methods for cyanide use DC amperometric detection⁸ or pulsed amperometric detection

(PAD).⁹⁻¹⁰ The DC amperometry method exhibits electrode fouling problems over time. The reported PAD detection based methods did not determine cyanide in drinking water samples.

This Application Note demonstrates fast, accurate determinations of free cyanide in drinking water samples using IC-PAD with a waveform optimized for cyanide, and use with a disposable silver working electrodes. This method is compatible with the basic solutions used to preserve drinking water samples for cyanide analysis and is unaffected by other compounds typically found in drinking water.

Experimental equipment

Thermo Scientific™ Dionex™ ICS-3000 system consisting of:

- Single Gradient Pump (SP) or Dual Gradient Pump (DP) module with degas option and gradient mixer (Thermo Scientific Dionex GM-4, P/N 049135)
- Detector and Chromatography Compartment (DC) with a single temperature zone and one injection valve
- Electrochemical Detector ED (P/N 079830) with an electrochemical cell containing a combination pH-Ag/AgCl reference electrode (cell and reference electrode, P/N AAA-061756, reference electrode P/N 071879) and a Certified Disposable Silver (Ag) working electrode (Package of 6 electrodes, P/N 063003)
- Thermo Scientific Dionex AS Autosampler with Sample Tray Temperature Controlling option and 1.5 mL sample tray
- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System with Chromeleon 6.7
- Filter unit, 0.2- μ m nylon (Nalgene Media-Plus with 90-mm filter, Nalge Nunc International, P/N 164-0020) or equivalent nylon filter
- Vacuum pump
- 1.5-mL polypropylene sample vials, with caps and slit septa (Thermo Scientific Dionex vial kit, P/N 079812)
- Disposable polystyrene 10-mL and 25-mL graduated pipettes
- Micropipettor and tips for preparing samples, standards, and pipetting samples into vials
- Thermo Scientific™ Dionex™ OnGuard™ II H cartridges (2.5 cc, package of 48, P/N 057086)

- Black PEEK (0.254-mm or 0.010-in. i.d.) tubing, used for eluent connections to cell, Pump 1, and columns (5 ft, P/N 052306)
- Red PEEK (0.127-mm or 0.005-in. i.d.) tubing, installed in DC heat exchanger (5 ft, P/N 052310)
- Green PEEK (0.76-mm or 0.030-in. i.d.) tubing, installed in AS Autosampler (5 ft, P/N 052305)

Reagents and standards

- Use only ACS reagent grade chemicals for all reagents and standards.
- Deionized water, Type 1 reagent-grade, 18.2 M Ω -cm resistivity or better, freshly degassed by vacuum filtration
- Sodium cyanide, anhydrous (Aldrich, P/N 20,522-2)
- Sodium hydroxide, 50% (w/w) (Fisher Chemicals, P/N SS254-500)
- pH 7 (yellow) and pH 10 (blue) buffer solutions (VWR International, P/N 34170-130, 34170-133)

Samples

- City of Sunnyvale (sampled on multiple days), City of San Jose, and Twain Harte Valley, CA drinking water samples
- The sources (flumes) of Twain Harte Valley (an old gold mining region) drinking water and Alamos Creek in Almaden region (an old mercury mining region) of San Jose, CA

Electrochemical detector (ED)

The Dionex ICS-3000 electrochemical detector is composed of an ED module with the electronics and an amperometric cell containing working, reference, and counter electrodes. The ED is a “plug and play” module and easily installs into the Dionex ICS-3000 Detector/Chromatography (DC) upper chamber and the cell mounts on the ED.

In this application, the working electrode is a disposable silver working electrode. Typically, the background will rise or fall to the equilibrium background within 10 min. The waveform was optimized for cyanide but it can also detect sulfide, bromide, and thiosulfate (Table 1).¹⁰

Calibration, handling, and installation tips for the reference electrode and Certified Disposable Silver working electrodes are thoroughly described in the System

Preparation and Setup section of this application note, the Thermo Scientific Dionex ICS-3000 Operator's Manual,¹¹ and the Dionex Product Manual for Gold and Silver Disposable Electrodes.¹²

Table 1. Cyanide waveform

Time (sec)	Potential vs Ag/AgCl (V)	Gain region	Integration	Ramp
0.00	-0.10	Off	Off	On
0.20	-0.10	On	On (start)	On
0.90	-0.10	On	Off (end)	On
0.91	-1.00	On	Off	On
0.93	-0.30	Off	Off	On
1.00	-0.30	Off	Off	On

Conditions	
Columns:	Thermo Scientific™ Dionex™ IonPac™ AS15 Analytical, 2 × 250 mm (P/N 053941) Dionex IonPac AG15 Guard, 2 × 50 mm (P/N 053943)
Flow Rate:	0.25 mL/min
Eluent:	63 mM Sodium hydroxide (31.5% Eluent B, 200 mM sodium hydroxide)
Column Temp:	30 °C
Tray Temp:	10 °C
Inj. Volume:	10 µL (PEEK sample loop, P/N 042949), full-loop injection
Detection:	Pulsed Amperometric Detection (PAD)
Waveform:	See Table 1
Electrodes:	Reference: pH-Ag/AgCl electrode (P/N 061879) in AgCl mode
Working:	Certified disposable Ag working electrode
Background:	3–13 nC versus Ag/AgCl
Backpressure:	~1100 psi
Noise:	< 7 pC
Run Time:	25 min
Syringe Speed:	4
Flush Volume:	250 µL

Preparation of solutions and reagents

Eluent preparation

It is essential to use high quality Type 1 water (> 18.2 MΩ-cm) containing as little dissolved carbon dioxide as possible. Degas the deionized water before eluent preparation. It is also essential to use high quality 50% (w/w) sodium hydroxide solution for eluent and diluent preparation. Sodium hydroxide pellets are coated with sodium carbonate and, therefore, are not acceptable for this application. Eluent preparation is thoroughly discussed in the Thermo Scientific™ Dionex™ AminoPac™ PA10 and AAA-Direct™ Product Manuals.^{13,14}

Eluent A (degassed deionized water)

To prepare degassed deionized Type 1 water (Eluent A), degas 2 L of Type 1 deionized water using ultrasonic agitation and applied vacuum to aid in removing the gas bubbles. Pour the degassed deionized water into a 2 L precleaned eluent bottle. Connect the eluent bottle to the Eluent A line from the pump and place the eluent bottle under ~4–5 psi of helium or other inert gas. Prime the pump with the new eluent.

Eluent B (200 mM sodium hydroxide)

Add 2000.0 g of degassed Type 1 deionized water into a 2 L precleaned eluent bottle. This is measured on a top loader balance that is accurate to ±0.01 g. Rinse a 25 mL graduated plastic pipette several times with deionized water and shake out the excess water. Using the pipette, remove 21.0 g of deionized water from the 2 L eluent bottle and discard it. Shake out or blow out with a pipette bulb the last remaining drops in the pipette. Using the same pipette, add 32.0 g (~21.0 mL) of 50% (w/w) sodium hydroxide solution into the 2 L eluent bottle. Connect the eluent bottle to the Eluent B line from the pump and place the eluent bottle under ~4–5 psi of helium or other inert gas. Swirl the eluent bottle to thoroughly mix the eluent. Prime the pump with the new eluent.

100 mM sodium hydroxide diluent solution

All of the cyanide standards were prepared gravimetrically in 100 mM sodium hydroxide diluent. To prepare the 100 mM sodium hydroxide solution, add 1000.0 g of degassed deionized water into a 2 L precleaned eluent bottle. Rinse a 10 mL graduated, plastic pipette several times with deionized water and shake out the excess water. Using the pipette, remove 5.2 g of deionized water from the 2 L eluent bottle and discard it. Shake out or blow out with a pipette bulb the last remaining drops in the pipette. Using the same 10 mL pipette, add 8.0 g (~5.25 mL) of 50% (w/w) sodium hydroxide solution into the 2 L eluent bottle. Place the eluent bottle under ~4–5 psi of helium or other inert gas. Swirl the eluent bottle to thoroughly mix the diluent.

AS autosampler flush solution

Prepare the degassed deionized water in the same manner described in Eluent A.

Analysis of tap and river water samples

No target analytes were found in the tap and river water samples. Figure 6 shows chromatograms of a tap water sample detected with fluorescence using three parallel channels. Method recovery was investigated by determining the recoveries in a tap water sample spiked

at two concentrations (0.05 and 5 µg/L of each PAH). The recovery range was from 80 to 120%, demonstrating that this on-line SPE HPLC method combined with UV and fluorescence detection provides good selectivity and suitability for the determination of PAHs in water samples

Standard preparation

Warning: Cyanide is a poison by inhalation, contact, and ingestion. It generates the poisonous hydrogen cyanide gas at neutral or acidic pH. Solutions containing cyanide must be stabilized with base. Read and follow the material safety data sheet (MSDS) instructions for personnel handling, exposure, and disposal information. Also consult local safety personnel for regulations concerning the proper disposal of cyanide.

Cyanide standards

To prepare a 1000 mg/L stock solution, weigh 0.0377 g of reagent grade, sodium cyanide into a 20-mL polyethylene bottle. Add 100 mM sodium hydroxide diluent to a total weight of 20.00 g. Prepare an intermediate standard solution of 1.0 mg/L cyanide by pipetting 20 µL of the 1000 mg/L stock solution into a 20 mL polyethylene bottle and dilute with 100 mM sodium hydroxide to a final weight of 20.00 g.

To prepare 2.0, 3.0, 5.0, 10.0, 50.0, 100 µg/L working standards of cyanide from the 1.0 mg/L intermediate standard, pipette 40, 60, 100, 200, 1000, and 2000 µL, respectively, of the intermediate standard into 20 mL polyethylene bottles. Dilute these working standards with 100 mM sodium hydroxide to 20.00 g total weight. The stock solution and the intermediate standard are stable for more than a month when refrigerated. The working standards should be prepared daily.

Standards for interference and retention time determination experiments

To prepare individual 1000 mg/L stock solutions of the ions (bromide, iodide, sulfide, sulfite, thiocyanate, and thiosulfate) for the interference experiments, dissolve the amount of reagent grade compound (Table 2) in deionized water in a 20 mL polyethylene bottle and dilute to 20.00 g with deionized water. These stock standards will be diluted to 1.0 mg/L concentration for interference and retention time determination experiments.

Prepare separate intermediate standards of 1.0 mg/L from each of the 1000 mg/L stock solutions. Pipette 20 µL of the individual stock solution into a 20 mL polyethylene bottle and dilute with deionized water to 20.00 g total weight. To prepare 10 µg/L individual standards of bromide, iodide, sulfide, sulfite, thiocyanate, and thiosulfate, pipette 200 µL of the intermediate standard into a separate 20 mL polyethylene bottle and dilute with 100 mM sodium hydroxide to 20.00 g total weight. These standards were used to determine anion retention times.

To prepare 5 µg/L and 10 µg/L of separate cyanide standards spiked with 10 µg/L of the individual interference ion (bromide, iodide, sulfide, sulfite, thiocyanate, or thiosulfate), pipette 100 µL and 200 µL, respectively, of the 1.0 mg/L cyanide intermediate standard into individual 20 mL polyethylene bottles. Pipette 200 µL of the 1.0 mg/L intermediate standard of the interference ion (bromide, iodide, sulfide, sulfite, thiocyanate, or thiosulfate) into the 20 mL polyethylene bottle and dilute with 100 mM sodium hydroxide to 20.00 g total weight. These standards were used to evaluate possible interferences with cyanide determinations. Note: Sodium sulfide solutions degrade quickly. They should only be prepared from a new bottle of sodium sulfide, nonahydrate solid. Once exposed to air, sulfide rapidly breaks down to sulfite. All the sulfide solutions, including the 1000 mg/L, are unstable even when refrigerated. Low-level sulfide standards must be prepared every two days and tested at 10 °C. Intermediate sulfide standards must be prepared every 2 weeks. The 1000 mg/L sulfide solution showed long term stability only when it was preserved by freezing at -10 °C.

Table 2. Amount of compound used to prepare 20.00 g (~20 mL) of individual 1000 mg/L stock solutions.

Anion	Compound	Mass (g)
Bromide	Sodium bromide (NaBr)	0.0258
Iodide	Sodium iodide (NaI)	0.0362
Sulfide	Sodium sulfide, nonahydrate (Na ₂ S•9H ₂ O)	0.1498
Sulfite	Sodium sulfite (Na ₂ SO ₃)	0.0315
Thiocyanate	Sodium thiocyanate (NaSCN)	0.0279
Thiosulfate	Sodium thiosulfate pentahydrate (Na ₂ S ₂ O ₃ •5H ₂ O)	0.0440

Standards for metal interference experiments

To prepare 650 µg/L iron, and separate 300 µg/L of copper and nickel working standards from the 1000 mg/L iron, copper, and nickel reference standards, pipette 65 µL of the iron reference standard, and 30 µL each of the copper, and nickel reference standards, into individual 125 mL polypropylene bottles. Dilute these working standards with deionized water to 100.00 g total weight. These standards were used to evaluate the interference of dissolved metals on free cyanide determinations.

Procedure to remove dissolved metals

To remove the dissolved metals, treat the dissolved metal solutions with 2.5 cc capacity, Dionex OnGuard II H cartridges per the product manual instructions.¹⁵ These cartridges are designed to remove alkali and alkaline earth metals, and cationic transition metals. Fill a 5 mL disposable syringe with the dissolved metal solution, attach a new 2.5 cc Dionex OnGuard II H cartridge, and dispense the solution through the cartridge, at ~1 mL/min, and into a 10 mL graduated cylinder. Fill the syringe again and dispense through the same cartridge until the graduated cylinder contains 6 mL of filtrate. Discard this initial filtrate. Tare a 10 mL sample vial and dispense the filtrate into the sample vial. Continue to refill the syringe and dispense the filtrate until 7.80 g of filtrate is dispensed in the vial. Add ~2 drops of 50% sodium hydroxide to the filtrate. Mix thoroughly, and spike the solution with 100 µL of 1.0 mg/L of cyanide, resulting in 10 µg/L cyanide in ~100 mM sodium hydroxide. It is critical that the sodium hydroxide is added and mixed into the solution prior to adding the cyanide. As a control, we treated 10 µg/L of cyanide in 100 mM sodium hydroxide with a Dionex OnGuard II cartridge in the same manner as the dissolved metal solutions, except that no additional sodium hydroxide or cyanide was added. A new cartridge was used for each dissolved copper, iron, and nickel solution.

To prepare 10 mL each of the combined cyanide and metal solutions, spike the metal solutions with ~2 drops of 50% sodium hydroxide, mix thoroughly, and spike with 100 µL of 1.0 mg/L of cyanide to a final concentration of 10 µg/L cyanide. These samples were tested immediately.

Sample preparation

Cyanide is reactive and unstable, therefore drinking water samples should be stabilized as soon as possible and free cyanide determined as soon as possible. Oxidizing agents decompose cyanide. Also any free cyanide present at neutral pH will volatilize to hydrogen cyanide.

Because of these issues, the drinking and surface water samples were stabilized as soon as practical. Sunnyvale and San Jose municipal drinking water samples were stabilized within one hour of sampling by addition of a 50% (w/w) sodium hydroxide solution. The procedure is as follows: rinse the 10 mL graduated plastic pipette several times with deionized water and shake out the remaining drops. Using the same pipette, add 2.00 g (~1.5 mL) of 50% (w/w) sodium hydroxide solution into a 250 mL polypropylene bottle, add the municipal drinking water to 250.00 g total weight, then mix thoroughly.

Alamitos Creek (in the Almaden mining area of San Jose) and Twain Harte Valley were located farther away from the laboratory so sampling kits were prepared for the Alamitos Creek surface water, and the Twain Harte Valley drinking and surface water samples. The kit contained disposable vinyl gloves for chemical handling, 100 mL wide-mouth specimen containers to collect the samples, and the 250 mL sampling bottles to store and stabilize the samples, all placed into a large resealable bag. The 250 mL sampling bottles were prepared in a similar manner as those for the municipal drinking samples. At the sampling site, 250 mL of the drinking and surface water samples were collected and measured with the 100 mL specimen containers, transferred to the 250 mL sampling bottles, and then thoroughly mixed with 50% sodium hydroxide, as described above.

Spike recovery samples of 5 and 10 µg/L cyanide from both municipal drinking water samples and the Alamitos Creek surface water were prepared by pipetting 100 µL and 200 µL, respectively, of 1.0 mg/L cyanide standard into 20 mL polyethylene bottles, and diluting with the base-treated water sample to a total weight of 20.00 g.

To remove potentially interfering dissolved metals, portions of both municipal drinking waters, and Twain Harte drinking and surface water samples were treated with the Dionex OnGuard II H cartridges. These samples were prepared in the same manner as the dissolved metals samples, though without further sodium hydroxide additions. To prepare 5 and 10 µg/L cyanide spike recovery samples of the drinking and surface water samples and the cartridge-treated drinking and surface water samples, pipette 50 µL and 100 µL, respectively, of 1.0 mg/L cyanide in individual 20 mL polyethylene bottles, and dilute with water sample or treated water sample to 10.00 g total weight.

System preparation and setup

The setup for the individual modules, components, and system is thoroughly described in the Dionex ICS-3000 Operator's Manual,¹⁶ and Dionex ICS-3000 Installation Manual,¹⁷ and the Chromeleon "Help" menus.

Plumbing the chromatography system

Connect black PEEK (0.254-mm or 0.010-in. i.d.) tubing from Pump 1 to the gradient mixer (GM-4) and from the gradient mixer to position "P" on Injection Valve 1 inside the DC module. Connect the red PEEK (0.127-mm or 0.005-in. i.d.) tubing from Injection Valve 1, position "C" to the heat exchanger. Install the Dionex IonPac AS15 column set according to the Dionex IonPac AS15 Product Manual.¹⁸ Connect the red PEEK tubing exiting the System 1 heat exchanger to the column set. The free end will be installed into the ED cell. Install a 10- μ L loop in DC Injection Valve 1 in both "L" positions. Connect the AS Autosampler injection port tubing and the green PEEK (0.76-mm or 0.030-in. i.d.) tubing waste line to DC Injection Valve 1 positions "S" and "W", respectively.

Configuring the AS Autosampler

Configure the Dionex AS Autosampler and connect the sample prep and sample syringes according the Dionex AS Autosampler Operator's Manual.¹⁹ Enter the loop size (10 μ L) in loop size V1, on the AS front panel, under Menu and Plumbing Configuration. Select the syringe sizes of the sample prep and the sample syringe from the pull down menus, under Menu and System Parameters. Also select "Normal" sample mode and "Enable" Wait function, under Menu and System Parameters.

Configuring the system

With all the power on and the Chromeleon monitor program running, open the Chromeleon Server Configuration program. To configure the system, first create a cyanide timebase and then add the devices: Dionex ICS-3000 DP or Thermo Scientific Dionex SP pump module, the Thermo Scientific Dionex DC module, and Dionex AS Autosampler. If a SP single pump is used, assign the Pump 1 to the cyanide timebase, (right click on SP module, select properties, select Devices tab, and select the cyanide timebase on pull-down menu for the pump). If a Dionex DP dual pump is used, select Devices tab, click off the share boxes ("share eluent bottles", and "share waste bottle"). Also insert a "pump off" command in the program for Pump 2. Verify that the AS device has the same options (e.g., sample preparation, temperature tray control, etc.)

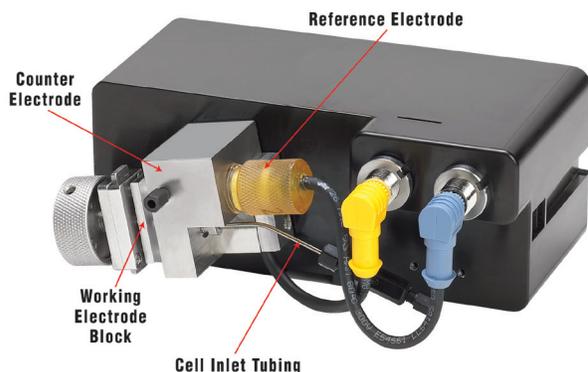


Figure 1. Amperometry cell.

as listed on the Dionex AS Autosampler module (Installed Options under Module Setup Menu). Save and check the configuration before leaving the program.

Electrochemical detector and amperometry cell

Install the electrochemical detector ED and amperometry cell (Figure 1)²⁰ and calibrate the combination pH-Ag/AgCl reference electrode. Install the ED module in the middle DC chamber, above Injection Valve 1. Remove the storage cap from the reference electrode but leave the storage cap O-ring in place on top of the reference electrode. The storage cap O-ring will be used again when the reference electrode is removed someday and sealed into the storage cap. It does not interfere with the installation of the reference electrode. Rinse the KCl storage solution off the reference electrode, pat dry, and place the reference electrode in pH 7 buffer. Open Chromeleon and connect to the cyanide timebase. Click on the Chromeleon Panel icon, expand the cyanide timebase panel, and select the EC Detector tab. Connect the blue lead of the reference electrode to the ED black port. Check the cell on/off button to ensure that the cell is turned off. The pH electrode remains active regardless of the cell power. Click on the "Calibration" button which opens the ED Wellness Panel. Follow the calibration instructions in the "instructions" button or in the Dionex ICS-3000 Operator's Manual. Wait for the pH reading to stabilize, then press the "pH Offset Cal" button and wait while it calculates the pH offset. After the reference electrode is finished reading, remove, rinse, and pat it dry. Place the reference electrode in pH 10 buffer and wait until the reading is stable. Enter the "10.00" in the pH slope buffer value, press the "pH slope Cal." button, and wait while it calculates the slope intercept. When the slope intercept is calculated, save, upload the new calibration values, and close the ED Wellness Panel.

Assemble the electrochemical cell. Check that the reference electrode O-ring on the bottom of the reference electrode is in place (install one if it is missing or damaged). Gently screw the reference electrode into the electrochemical cell body. Tighten to a snug fit (finger-tight, do not use tools). Install the Certified Disposable Silver Electrode in the electrochemical cell, according to the Disposable Electrode Ag Installation Guide²¹ received with the electrodes. Install the electrochemical cell into the ED. Connect the red PEEK tubing exiting the columns to the cell inlet and direct the cell outlet tubing to waste.

Results and discussion

The cyanide waveform

The cyanide waveform is a three-potential waveform using E1, E2, and E3. These voltages are applied at the designated times during a 1 sec. waveform. E1, detection and integration potential, is -0.10 V vs Ag/AgCl and maintained from 0.00 to 0.90 sec. E2 is -1.0 V vs Ag/AgCl at 0.91 sec, and E3 is -0.30 V vs Ag/AgCl from 0.93 to 1.00 sec. E2 and E3 clean and restore the working electrode (Table 1). The waveform is applied continuously when the amperometric cell is turned on.

Chromatography

In the publication that reported the above waveform, the recommended eluent concentration was 62.5 mM sodium hydroxide.¹¹ In this experiment, the eluent concentration used was 63 mM sodium hydroxide, however, a 200 mM sodium hydroxide solution was prepared because it is easier to prepare consistently and requires less frequent

preparation. The pump was programmed to proportion this prepared 200 mM sodium hydroxide solution to create the desired eluent concentration. Cyanide elutes at 5.8 min. Figure 2 shows both 5 and 10 µg/L cyanide standards in 100 mM sodium hydroxide.

Interferences

In the EPA methods, sulfide and sulfide-generating compounds are cited most often as potential interferences. Sulfide complexes with free cyanide to form thiocyanate. Other interferences cited are nitrate, nitrite, and chlorine. Copper and other transition metals complex with cyanide, preventing the measurement of free cyanide. Copper (II) rapidly oxidizes cyanide to cyanogen gas and copper (I) cyanide precipitates. Acidification will volatilize cyanide to the poisonous hydrogen cyanide gas, thus preventing it from being measured as free cyanide. In this application, electroactive anions (iodide, thiosulfate, bromide, thiocyanate, and sulfide) are potential interferences, that is, anions detected using a silver working electrode and this waveform.

Possible anionic interferences

We determined the interference effects of the non-oxidized and partially oxidized sulfur-containing anions (sulfide, thiosulfate, thiocyanate, and sulfite), bromide, and iodide by analyzing solutions of 10 µg/L cyanide and 20 µg/L of each potential interfering anion. Table 3 shows that the free cyanide concentrations were not significantly affected by any of the anions. The free cyanide concentration did show a small decrease with sulfide and a small increase with thiocyanate. Sulfide and thiocyanate are not expected at high concentrations in drinking water. Because sulfide is not desirable in drinking water due to its disagreeable odor and taste, it is typically removed from municipal water systems by oxidation during the sanitation process. Noise levels always increase during the thiosulfate experiments

Column: Dionex IonPac AS15 and Guard, 2 mm
 Eluent: 63 mM Sodium hydroxide (31.5% Eluent B, 200 mM sodium hydroxide)
 Column Temp: 30 °C
 Tray Temp: 10 °C
 Flow Rate: 0.25 mL/min
 Inj. Volume: 10 µL
 Detection: PAD, Cyanide waveform
 Peaks: (A) (B)
 1. Cyanide 5.0 9.7 µg/L
 Samples: Standards A & B are in 100 mM sodium hydroxide

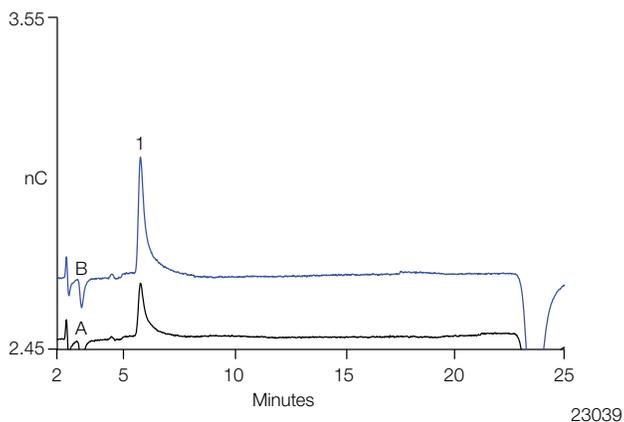


Figure 2. Cyanide standards in 100 mM sodium hydroxide.

Table 3. Effect of bromide, iodide, sulfide, sulfite, thiocyanate, and thiosulfate on cyanide recovery.

Anion ^a	Compound
None	102.9 ± 2.3%
Bromide	99.9 ± 2.6%
Iodide	99.9 ± 2.9%
Sulfide	95.9 ± 1.7%
Sulfite	97.7 ± 2.5%
Thiocyanate	110.1 ± 2.6%
Thiosulfate	99.9 ± 2.7%

n=10 for each experiment.

^a The concentration of each anion was 20 µg/L.

because thiosulfate is a reducing agent and interacts with the working electrode. These experiments confirmed that the cyanide waveform detects thiosulfate, sulfide, and bromide under these conditions. Although sulfite, thiocyanate, and iodide are also potential interfering anions to cyanide, they were not detected by this waveform (sulfite and thiocyanate) or elute under these conditions (iodide). For applications requiring resolution of cyanide from sulfide, the Dionex IonPac AS7²² column set should be selected.^{9,10}

Metal interferences

We determined the effects of dissolved iron, copper, and nickel on free cyanide determinations. We treated a 10 µg/L cyanide standard with each of the dissolved metal solutions and compared these three solutions to an untreated standard. We selected the iron concentration (600 µg/L) based on the expected levels in drinking water.²³ We arbitrarily set the copper and nickel concentrations for this experiment to 300 µg/L, 50% of the iron levels. We also treated the metal solutions with Dionex OnGuard II H cartridges to remove the metals and then added these solutions to cyanide standards.

The results show that copper and nickel reduce free cyanide concentrations. In the iron solution, the free cyanide concentration loss was comparable to the control, 7–10% over 3 days (Figure 3). After 92 h, only 28% of free cyanide remained in the copper solution (Figure 4). In the nickel solution, the free cyanide decreased to 75% within 20 h and then stabilized for the remainder of the 3 day experiment (Figure 5).

The cyanide control sample was not affected (102% ± 1% recovery) by the Dionex OnGuard II H treatment. Most importantly, the free cyanide concentration is as stable as the untreated standard when the metal-containing solutions are treated with the Dionex OnGuard II H cartridges (7–10% loss over three days) and, therefore, the cartridges effectively remove the dissolved metals. The results also confirm that the free cyanide concentration declines over three days and that the samples should be analyzed as soon as possible.

Column: Dionex IonPac AS15 and Guard, 2 mm Column Temp: 30 °C
 Eluent: 63 mM Sodium hydroxide Tray Temp: 10 °C
 (31.5% Eluent B, 200 mM sodium hydroxide) Flow Rate: 0.25 mL/min
 Inj. Volume: 10 µL
 Detection: PAD, Cyanide waveform

Samples: A) 286 µg/L of copper spiked with sodium hydroxide and 10 µg/L of cyanide
 B) 286 µg/L of copper treated with Dionex OnGuard II H cartridge, spiked with sodium hydroxide and 10 µg/L of cyanide
 C) 10 µg/L of cyanide in 100 mM sodium hydroxide
 D) 10 µg/L of cyanide in 100 mM sodium hydroxide, treated with Dionex OnGuard II H cartridge

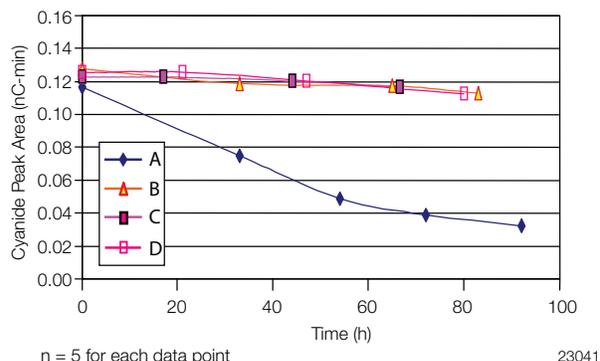


Figure 4. Effect of dissolved copper on free cyanide (10 µg/L).

Column: Dionex IonPac AS15 and Guard, 2 mm Column Temp: 30 °C
 Eluent: 63 mM Sodium hydroxide Tray Temp: 10 °C
 (31.5% Eluent B, 200 mM sodium hydroxide) Flow Rate: 0.25 mL/min
 Inj. Volume: 10 µL
 Detection: PAD, Cyanide waveform

Samples: A) 601 µg/L of iron spiked with sodium hydroxide and 10 µg/L of cyanide
 B) 601 µg/L of iron treated with Dionex OnGuard II H cartridge, spiked with sodium hydroxide and 10 µg/L of cyanide
 C) 10 µg/L of cyanide in 100 mM sodium hydroxide
 D) 10 µg/L of cyanide in 100 mM sodium hydroxide, treated with Dionex OnGuard II H cartridge

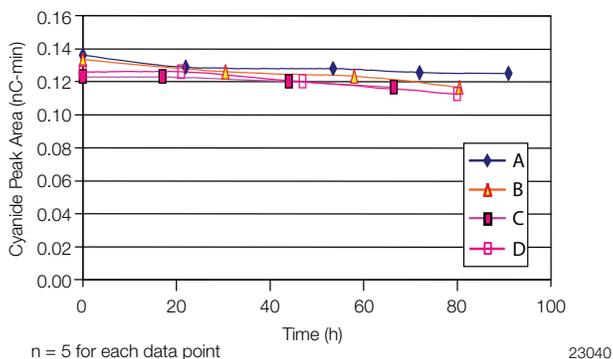


Figure 3. Effect of dissolved iron on free cyanide (10 µg/L).

Column: Dionex IonPac AS15 and Guard, 2 mm Column Temp: 30 °C
 Eluent: 63 mM Sodium hydroxide Tray Temp: 10 °C
 (31.5% Eluent B, 200 mM sodium hydroxide) Flow Rate: 0.25 mL/min
 Inj. Volume: 10 µL
 Detection: PAD, Cyanide waveform

Samples: A) 299 µg/L of nickel spiked with sodium hydroxide and 10 µg/L of cyanide
 B) 299 µg/L of nickel treated with Dionex OnGuard II H cartridge, spiked with sodium hydroxide and 10 µg/L of cyanide
 C) 10 µg/L of cyanide in 100 mM sodium hydroxide
 D) 10 µg/L of cyanide in 100 mM sodium hydroxide, treated with Dionex OnGuard II H cartridge

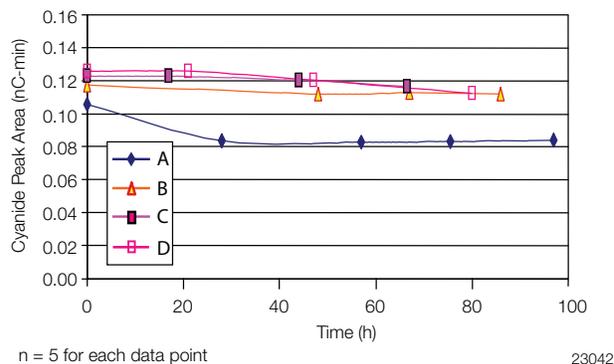


Figure 5. Effect of dissolved nickel on free cyanide (10 µg/L).

Method Qualification

The cyanide method was qualified prior to determining cyanide in real drinking water samples by determining the linearity over a 50-fold concentration range, typical noise, the method detection limit (MDL), reproducibility, and ruggedness. The linearity of cyanide response was determined by measuring cyanide in six replicates each of six standards (2.0, 3.0, 5.0, 10.0, 50.0, and 100 µg/L). The calibration results showed good linearity over this concentration range ($r^2 > 0.999$).

For each of the five disposable electrodes, the noise was determined over two 60 min runs, when no sample was injected, by measuring the noise in 1 min intervals from 5 to 60 min. The noise value determined by this experiment was 7.0 ± 1.8 pC ($n = 10$). The method detection limit (MDL) was defined as the peak in a standard with a peak height that is three times the noise level. For this application, the MDL was 1.0 µg/L. The signal to noise ratio of the 2.0 µg/L cyanide standard was 16.3 ± 4.8 ($n = 10$).

The reproducibility and ruggedness of the cyanide method was determined over 140 injections, ~62 h. During this study, cyanide in 10 µg/L cyanide standards was measured and the same standards spiked in copper, iron, and nickel metal solutions pretreated with Dionex OnGuard II H cartridge. Deionized water injections were inserted between the sample groups. The results (Figures 6 and 7) showed that retention time and peak areas were stable over 62 h of the experiment. The retention time and peak area reproducibilities were 5.78 ± 0.027 min and 0.1232 ± 0.0016 nC-min, respectively.

Determination of cyanide in drinking water and surface water

The characteristics of most drinking water change with the season. Free cyanide concentrations and spike recoveries of 5 and 10 µg/L cyanide in the City of Sunnyvale water were determined throughout the course of the application experiments and some changes were observed.

Sunnyvale drinking water sampled during the summer showed good spike recovery of cyanide for 5 µg/L and 10 µg/L of spiked cyanide ($91.5 \% \pm 1.0\%$ [$n=10$] and $98.2 \% \pm 1.7\%$ [$n=10$], respectively). Cyanide also showed good recovery when 10 µg/L of sulfide was added ($91.9\% \pm 1.7\%$). Spiking cyanide into 100 mM sodium hydroxide yielded similar recoveries. No free cyanide was measured in the unspiked samples of City of Sunnyvale drinking water (Figure 8).

Column: Dionex IonPac AS15 and Guard, 2 mm
Eluent: 63 mM Sodium hydroxide (31.5% Eluent B, 200 mM sodium hydroxide)
Column Temp: 30 °C
Tray Temp: 10 °C
Flow Rate: 0.25 mL/min
Inj. Volume: 10 µL
Detection: PAD, Cyanide waveform

Samples: A)286 µg/L of copper solution treated and spiked with 10 µg/L cyanide
B)299 µg/L of nickel solution treated and spiked with 10 µg/L cyanide
C)601 µg/L of iron solution treated and spiked with 10 µg/L cyanide
D)Control, 10 µg/L in 100 mM sodium hydroxide
E)Treated Control

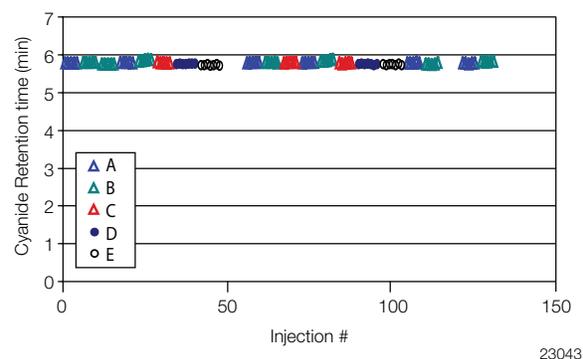


Figure 6. Retention time stability of 10 µg/L cyanide.

Column: Dionex IonPac AS15 and Guard, 2 mm
Eluent: 63 mM Sodium hydroxide (31.5% Eluent B, 200 mM sodium hydroxide)
Column Temp: 30 °C
Tray Temp: 10 °C
Flow Rate: 0.25 mL/min
Inj. Volume: 10 µL
Detection: PAD, Cyanide waveform

Samples: A)286 µg/L of copper solution treated and spiked with 10 µg/L cyanide
B)299 µg/L of nickel solution treated and spiked with 10 µg/L cyanide
C)601 µg/L of iron solution treated and spiked with 10 µg/L cyanide
D)Control, 10 µg/L in 100 mM sodium hydroxide
E)Treated Control

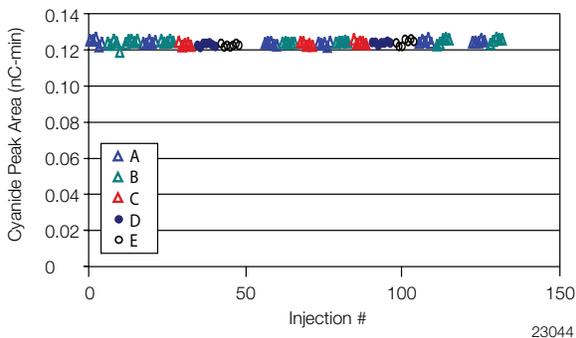


Figure 7. Peak area stability of 10 µg/L cyanide.

Column: Dionex IonPac AS15 and Guard, 2 mm
 Eluent: 63 mM Sodium hydroxide (31.5% Eluent B, 200 mM sodium hydroxide)
 Column Temp: 30 °C
 Tray Temp: 10 °C
 Flow Rate: 0.25 mL/min
 Inj. Volume: 10 µL
 Detection: PAD, Cyanide waveform
 Peaks:
 1. Unknown
 2. Unknown
 3. Chloride
 4. Cyanide 10 µg/L
 5. Bromide
 Samples: A)City of Sunnyvale drinking water, sampled during the summer and treated with sodium hydroxide
 B)Sample A Spiked with 10 µg/L cyanide

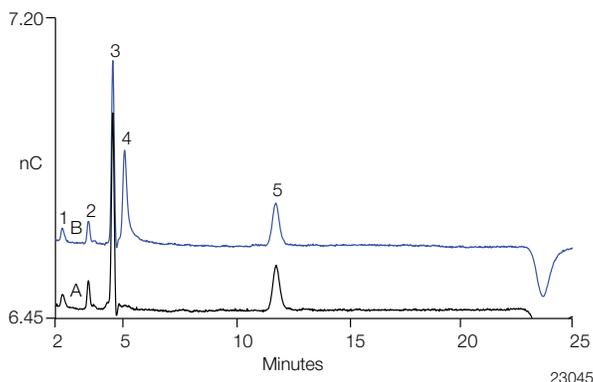


Figure 8. City of Sunnyvale drinking water with and without spiked cyanide.

The method described in this document was used to measure free cyanide and the recovery of cyanide from two drinking water samples sampled in the fall (City of San Jose and City of Sunnyvale) and one surface-water sample (Alamitos Creek in the old Almaden mining region of San Jose) that were collected in the fall. The results showed no initial concentrations of free cyanide and variable recovery of cyanide spikes (Table 4). Only the Alamitos Creek surface water sample (Figure 9) exhibited acceptable recovery. The City of Sunnyvale drinking water had poor recovery at both spike levels (9.6% ± 3.0% and 55.5% ± 2.8%, for 5 µg/L and 10 µg/L cyanide, respectively). The City of San Jose drinking water samples had mixed results (74.3% ± 11.8% recovery of 5 µg/L and 99.6% ± 0.5% for 10 µg/L cyanide). These cyanide recovery results for the City of Sunnyvale sample were contradictory to the initial results (Figure 8). It is possible that City of Sunnyvale drinking water changed since the initial sampling. The cyanide recovery from

Table 4. Recovery of cyanide from untreated drinking water and surface water samples^a

Concentration of cyanide spike (µg/L)	City of Sunnyvale drinking water ^a	City of San Jose drinking water	Alamitos Creek in Almaden
5	9.6 ± 3.0%	74.3 ± 11.8%	102.0 ± 1.3%
10	55.5 ± 2.8%	99.6 ± 0.5%	97.3 ± 2.4%

n= 5 for each sample.
^aSampled during the fall months.

Column: Dionex IonPac AS15 and Guard, 2 mm
 Eluent: 63 mM Sodium hydroxide (31.5% Eluent B, 200 mM sodium hydroxide)
 Column Temp: 30 °C
 Tray Temp: 10 °C
 Flow Rate: 0.25 mL/min
 Inj. Volume: 10 µL
 Detection: PAD, Cyanide waveform
 Peaks:
 1. Unknown
 2. Unknown
 3. Cyanide 10 µg/L
 4. Bromide
 Samples: A)Alamitos Creek region of San Jose treated with sodium hydroxide
 B)Spiked with 10 µg/L cyanide

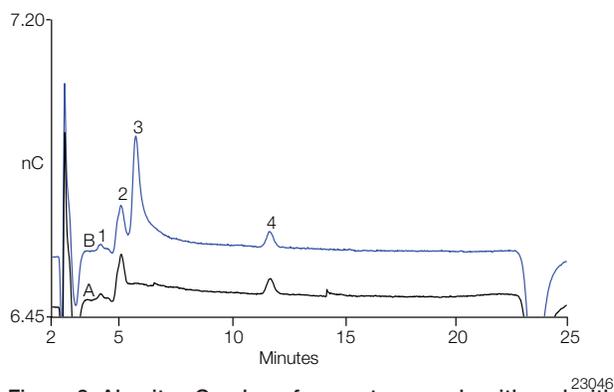


Figure 9. Alamitos Creek surface water sample with and without cyanide.

the City of San Jose drinking water over time showed a trend similar to those observed with metal interferences (Figure 10). Therefore, the samples were treated with the Dionex OnGuard II H cartridges and the recovery experiments repeated. Drinking and surface water samples were also analyzed from Twain Harte Valley, an old gold mining region. The results (Table 5) show good recovery for all samples (Figures 11–13) and good stability (> 84% of the initial peak response) for 31 h (not shown). No free cyanide was measured in any of the drinking or surface water samples.

Table 5. Recovery of cyanide in treated water samples^a (Dionex OnGuard II H Cartridges)

Concentration of cyanide spike (µg/L)	City of Sunnyvale drinking water ^a	City of San Jose drinking water	Twain Harte Valley drinking water	Twain Harte Valley Flume
5	80.6 ± 5.5%	87.3 ± 6.4%	95.9 ± 2.5%	81.1 ± 3.2%
10	99.5 ± 2.8%	99.4 ± 2.5%	96.8 ± 3.1%	93.1 ± 1.5%

n= 5 for each sample.
^aSampled during the fall months.

Column: Dionex IonPac AS15 and Guard, 2 mm
 Eluent: 63 mM Sodium hydroxide (31.5% Eluent B, 200 mM sodium hydroxide)
 Column Temp: 30 °C
 Tray Temp: 10 °C
 Flow Rate: 0.25 mL/min
 Inj. Volume: 10 µL
 Detection: PAD, Cyanide waveform

Column: Dionex IonPac AS15 and Guard, 2 mm
 Eluent: 63 mM Sodium hydroxide (31.5% Eluent B, 200 mM sodium hydroxide)
 Column Temp: 30 °C
 Tray Temp: 10 °C
 Flow Rate: 0.25 mL/min
 Inj. Volume: 10 µL
 Detection: PAD, Cyanide waveform

Samples: A)City of San Jose drinking water, treated with sodium hydroxide and 10 µg/L cyanide

Samples: A)City of Sunnyvale drinking water (sampled in the fall), treated
 B) Sample A spiked with 10 µg/L cyanide

Peaks:
 1. Unknown
 2. Chloride
 3. Cyanide 10 µg/L
 4. Unknown

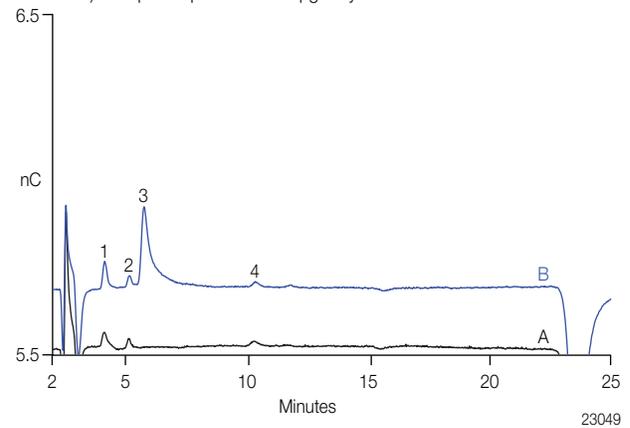
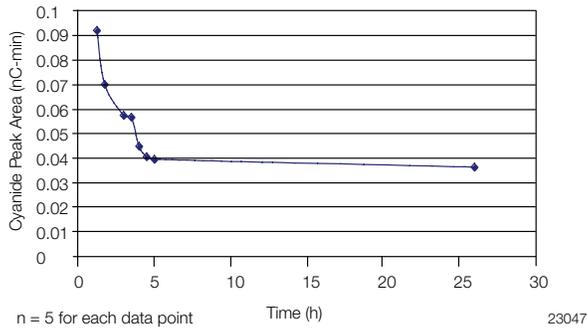


Figure 10. Degradation of cyanide spiked into the city of San Jose drinking water.

Figure 12. Treated city of Sunnyvale drinking water with and without 10 µg/L of cyanide.

Column: Dionex IonPac AS15 and Guard, 2 mm
 Eluent: 63 mM Sodium hydroxide (31.5% Eluent B, 200 mM sodium hydroxide)
 Column Temp: 30 °C
 Tray Temp: 10 °C
 Flow Rate: 0.25 mL/min
 Inj. Volume: 10 µL
 Detection: PAD, Cyanide waveform

Column: Dionex IonPac AS15 and Guard, 2 mm
 Eluent: 63 mM Sodium hydroxide (31.5% Eluent B, 200 mM sodium hydroxide)
 Column Temp: 30 °C
 Tray Temp: 10 °C
 Flow Rate: 0.25 mL/min
 Inj. Volume: 10 µL
 Detection: PAD, Cyanide waveform

Samples: A)City of San Jose drinking water, treated
 B)Sample A spiked with 10 µg/L cyanide

Peaks:
 1. Unknown
 2. Chloride
 3. Unknown
 4. Cyanide 10 µg/L
 5. Bromide

Samples: A)Twain Harte Valley drinking water, treated
 B)Sample A spiked with 10 µg/L cyanide

Peaks:
 1. Unknown
 2. Chloride
 3. Unknown
 4. Cyanide 10 µg/L
 5. Unknown
 6. Unknown

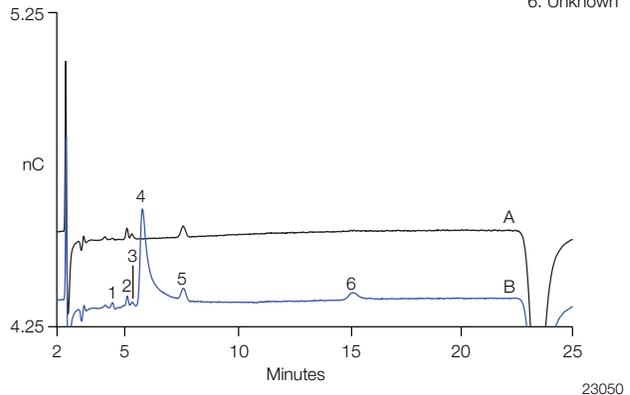
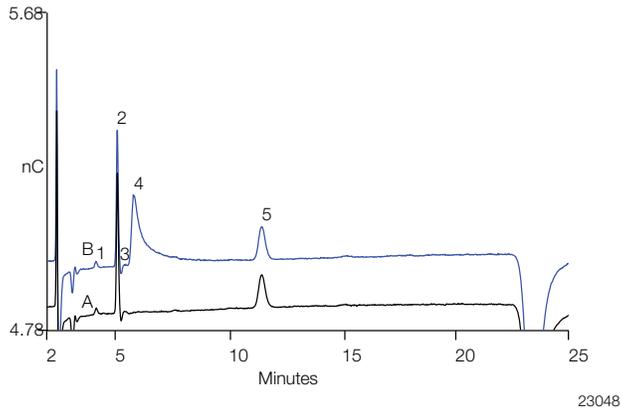


Figure 11. Treated city of San Jose drinking water with and without 10 µg/L of cyanide.

Figure 13. Treated city of Twain Harte Valley drinking water with and without 10 µg/L of cyanide.

Disposable silver working electrodes

In a published application using a waveform to determine iodide, the disposable silver working electrodes exhibited comparable or better reproducibility, linearity, and sensitivity than the conventional working silver electrodes.²⁴ The authors reported that they also saved time by discarding the disposable silver working electrodes at 80% of the peak response rather than re-polishing the conventional working electrode. In this study, the lifetimes of five disposable silver working electrodes were evaluated during the interference experiments, method qualification, and the testing of the municipal drinking water samples. Each electrode was installed, tested, and removed after two weeks of continuous use.

The average peak areas of 10 µg/L cyanide in 100 mM sodium hydroxide were compared over the five electrodes. The average peak area was 0.1206 ± 0.0038 nC-min, less than 1% variation. All five of the disposable silver working electrodes exceeded the 14 day lifetime specification (> 80% of the peak response). Three of the five disposable silver working electrodes were only removed after three weeks so that another electrode could be tested. The other two electrodes showed > 10 pC of noise during the last few days of operation.

Conclusion

Free cyanide can be determined in drinking water by IC-PAD. This method exhibits good sensitivity (MDL of 1 µg/L) and recovery, and exhibits linearity from 2 to 100 µg/L. This method can tolerate basic pH solutions, therefore it is believed that this method can determine cyanide in samples prepared for total cyanide determinations without dilution or neutralization of pH 13 distillation samples.

Transition metals can interfere with free cyanide determinations in drinking water. Dissolved transition metals are often present in drinking water and, therefore, it is prudent to eliminate this possible interference by treating the water samples with Dionex OnGuard II H cartridges prior to analysis. Cyanide determinations in drinking water should always include spike recovery to ensure accurate determinations.

Precautions

Warning: Cyanide is a poison. Never add cyanide to any solutions that have not been stabilized with base to a pH > 9. Read and follow all safety precautions, handling, and waste disposal information prior to handling or using cyanide. Cyanide solutions cannot be poured into the water system without treatment. Consult your local safety representative for waste handling.

The Eluent Generator is not recommended as the eluent source for this application because of undesirably high noise levels.

Drinking and surface water samples should be stabilized immediately with sodium hydroxide. Cyanide solutions in 100 mM sodium hydroxide are stable for about one week. In drinking water samples without metal or other cation interferences, cyanide is stable for about three days.

Sodium hydroxide will etch glass and will foul the silver electrode, therefore, use only plastic pipettes, vials, and bottles for this application.

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