Municipal water analysis applications summary

Inorganic anions and cations, toxic contaminants, disinfection byproducts

Introduction

Drinking water quality is a universal health concern with global impact. The water discharged by municipal waste water treatment plants and industrial facilities must be monitored to ensure strict compliance with global regulatory requirements. These agencies have developed standards for water analysis to assure that the community is consuming only safe drinking water.

The Safe Drinking Water Act (SDWA) is a US federal law which sets legal limits on the levels of certain contaminants in drinking water. Under the SDWA, the United States Environmental Protection Agency (EPA) enforces the National Primary Drinking Water Regulations (NPDWRS or primary standards) that apply to all public water systems. The NPDWR mandates maximum concentration levels of certain drinking water contaminants, also called “maximum contaminant levels” or “MCLs”. The EPA also provides a list of acceptable techniques for treating drinking water to reduce regulated contaminants to acceptably low levels.

In Europe, the Drinking Water Directive provides the essential quality standards. These quality standards were developed using guidelines from the World Health Organization (WHO) and the European Commission’s Scientific Advisory Committee. Member States of the European Union can add additional requirements for substance regulation; however they cannot set lower standards for these substances. Drinking water must be reported to the European Commission every three years.

Contaminant levels in drinking water are continuously subject to reassessment by the above regulatory bodies, both in regard to revised levels, as well as the addition of new contaminants to the list of existing regulated substances. For example, perchlorate has been recently identified as an environmental contaminant found in drinking water which impairs normal thyroid function by interfering with iodine uptake by the thyroid gland. In February, 2011 the EPA decided to develop a national primary drinking water regulation for perchlorate. This effort is still in progress.

Similarly, also in 2011 they recommended enhanced monitoring for Cr(VI) based on results from an independent survey showing that 35 drinking water samples exceeded the regulatory limits for Cr(VI). Hexavalent chromium (Cr(VI)) is of particular concern since it is a highly toxic carcinogen.

Thermo Fisher Scientific is committed to enhancing the quality of our global water resources. Our Thermo Scientific™ Dionex™ ion chromatography (IC) instruments are used by government and industry to provide solutions for environmental water testing for a wide range of regulated and emerging inorganic elements and organic compounds. These analytical instruments have evolved over many generations, each providing enhanced performance, greater reliability, and easier operation.

Ground and surface waters are a vital resource for a healthy environment. They are also the largest source of fresh water. These waters can comprise complex matrices that interfere with detection of analytes of interest. Another major challenge for qualifying drinking water is the analysis of disinfection byproducts (DBP). Drinking water is treated with disinfectants to remove potentially harmful bacteria. These disinfectants also react with ions and residual organic matter resulting in the formation of DBPs. DBPs are highly toxic, are regulated, and require mitigation of their concentrations prior to distribution of treated water. As the technology leader in ion chromatography, we have developed innovative techniques which overcome common challenges in the analysis of drinking water contaminants and DBPs.

As your partner for drinking water analysis, we promise to deliver the technology, experience, and support necessary for you to provide safe drinking water and ultimately protect our environment.

Ion analysis

Since the development of ion chromatography (IC) analysis over 30 years ago, Thermo Fisher Scientific has continually pioneered the development of new and innovative IC systems.

Our High-Pressure™ Ion Chromatography (HPIC™) systems include the Thermo Scientific Dionex ICS-5000+ HPIC system which is optimized for flexibility, modularity and ease-of-use, combining the highest chromatographic resolution with convenience. In addition, the Thermo Scientific Dionex ICS-4000 Capillary HPIC system is the world's first dedicated capillary Reagent-Free™ IC (RFIC™) system that is commercially available. The Dionex ICS-4000 system is always ready for the next analysis delivering high-pressure IC on demand.

Reagent-Free IC eliminates daily tasks of eluent and regenerant preparation, saving time, preventing errors, and increasing convenience. RFIC-EG systems use electrolytic technologies to generate eluent on demand from deionized water, and to suppress the eluent back to pure water, delivering unmatched sensitivity. RFIC-ER systems are designed to use carbonate, carbonate/ bicarbonate, or MSA eluents for isocratic separations.

At the heart of our ion chromatography systems is a unique set of column chemistries that provide high selectivities and efficiencies with excellent peak shape and resolution. Thermo Scientific™ Dionex™ IonPac™ polymeric columns address a variety of chromatographic separation modes including ion exchange, ion exclusion, reversed-phase ion pairing, and ion suppression. Our column chemistries are designed to solve specific applications, and we offer a variety of selectivities and capacities for simple and complex sample matrices. Additionally, our Dionex IonPac column line is available in standard bore, microbore and capillary formats for the ultimate application flexibility.

Learn more about our innovations in IC at
www.thermofisher.com/IC

The complete Thermo Scientific Dionex IC Systems Family

High-pressure ion chromatography

High-pressure ion chromatography systems allow continuous operation up to 5000 psi when configured as a Reagent-Free (RFIC) system for standard, microbore and capillary scale flow rates. As a result, these instruments can use high resolution 4 µm particle ion exchange columns which create higher back pressures. Higher backpressures are possible with the Dionex ICS-5000⁺ system and the Dionex ICS-4000 systems.

The Dionex IonPac 4 µm particle-size columns as in the case of reversed phase HPLC columns with smaller particles, higher system operating pressures are necessary to utilize these small particle columns. High pressure IC systems enable the use of the 4 µm columns, thus yielding yield fast separations with short (150 mm) columns, and high resolution with standard length (250 mm) columns.

Reagent-free ion chromatography (RFIC)

Advances in eluent generation and electrolytic suppression technologies are enabling a wider variety of applications and increased productivity. RFIC systems are available with eluent generation (RFIC-EG) or with eluent regeneration (RFIC-ER), such as the Thermo Scientific Dionex ICS-2100, the Dionex ICS-4000, and the Dionex ICS-5000⁺ systems. These systems combine automated eluent generators and electrolytically regenerated suppressors to electrolytically create the required eluents and regenerants used for IC applications. Laboratories using RFIC systems spend less time on equilibration, calibration, method verification, troubleshooting, and consistency checks because the systems minimize unintentional variations in the preparation of eluents and regenerants.

RFIC – eluent regeneration (RFIC-ER)

RFIC systems with eluent regeneration (RFIC-ER systems) are designed to use carbonate, carbonate/bicarbonate, or MSA eluents for isocratic separations. Eluent regeneration uses the suppressor to reconstitute the starting eluent, allowing use of a single 4 L bottle of eluent for up to four weeks. Because the system is a closed loop, it can run continuously, eliminating the need for recalibration or re-equilibration during the 28 days of non-stop operation. The same electrolytic process that suppresses eluent for detection is used to regenerate eluent for reuse.

After detection, suppressed eluent is passed through an analyte trap column to remove the analyte ions. The suppressed eluent is then returned to the suppressor to provide the water for electrolytic suppression. The effluent from the suppressor contains the eluent ions and H_2 and O_2 gases. A catalytic column recombines the oxygen and hydrogen to form water. The suppressor effluent is then returned to the eluent reservoir for reuse. Because the electrolysis gases are recombined stoichiometrically, no water is lost in the system and eluent concentration remains constant. A purification column is plumbed after the pump to further assure eluent purity. Stable eluent concentration yields reproducible results, with little variability in peak retention times or areas.

Continuous operation eliminates the need to re-equilibrate, and with no need to prepare eluent; all the operator has to do is load a sample. This means higher sample throughput and more time for operators to pursue other tasks. Pump maintenance is also reduced, because the flowing eluent has no opportunity to crystallize on pump surfaces. Eluent can be regenerated for up to four weeks for analysis of samples with low- to moderate-ionic strength, such as drinking water. Higher injection volumes or heavy workloads may require more frequent replacement or regeneration of trap and purification columns and eluent.

Learn more about our eluent regeneration solutions.

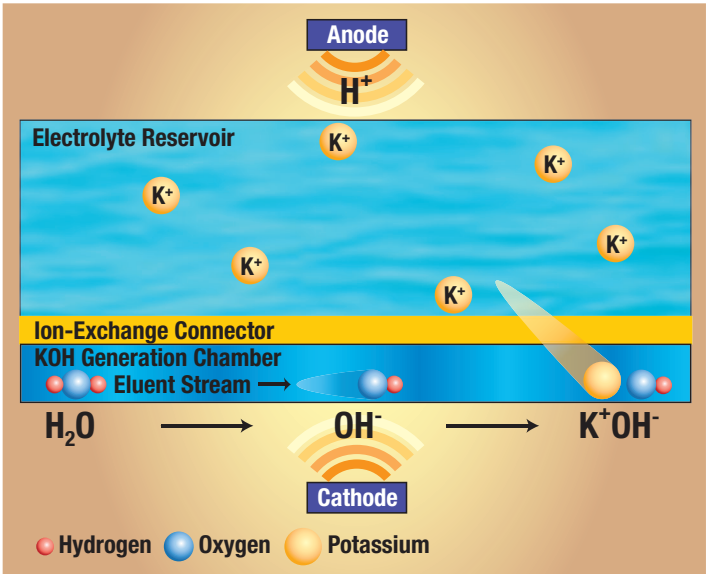
RFIC – eluent generation (RFIC-EG)

Eluent generation allows the automatic production of high-purity IC eluents. This is made possible through precise control of the electric current applied to the electrolysis of water to generate hydroxide and hydronium ions. Eluent generation eliminates the need to manually prepare eluents from concentrated acids and bases. The only routine reagent needed is deionized water. Furthermore, because the instrument pump seals and pistons only come in contact with deionized water, overall pump maintenance is significantly reduced.

With eluent generation, a pair of electrodes is positioned with an ion exchange membrane separating them; when a current is applied to the electrodes, electrolysis of water generates hydroxide at the cathode and hydronium at the anode. The ion exchange membrane prevents the species from recombining into water, and allows a counter-ion from the Eluent Generation Cartridge to migrate across the membrane to form the eluent. The eluent concentration is varied by changing the applied current to within a given range 0–100 mM or 0–200 mM (cap). This entire process can be done without the use of extra pumps, fittings, valves or any moving parts.

The Thermo Scientific Dionex Eluent Generator Cartridge (EGC) is at the core of the patented eluent generation technology used in RFIC-EG systems. A range of Dionex EGC cartridges are available for the production of hydroxide, carbonate, and methanesulfonic acid eluents. For cation-exchange applications, the Dionex EGC III MSA, Dionex EGC MSA (Capillary) and

Dionex EGC 500 MSA cartridges produce methanesulfonic acid eluents. For anion-exchange applications, the Dionex EGC III KOH, Dionex EGC NaOH, or Dionex EGC LiOH, Dionex EGC KOH (Capillary) and Dionex EGC 500 KOH cartridges produce potassium, sodium, or lithium hydroxide eluents. The Dionex EGC III K_2CO_3 cartridge produces carbonate-only eluent, and the Electrolytic pH Modifier (EPM III) can be added for generating carbonate/bicarbonate eluents.



The Dionex EGC III KOH cartridge consists of a KOH generation chamber and a K^+ electrolyte reservoir, connected by a cation exchange connector. A high-pressure connector permits the passage of K^+ ions from the K^+ electrolyte reservoir into the electroytic chamber.

RFIC-EG systems have redefined IC by making it possible to just add water to operate an IC system. These systems allow for a simpler and more reliable way to help deliver superior results while simultaneously saving time and labor. RFIC-EG systems facilitate drinking, waste, and groundwater analyses for regulatory compliance. Furthermore, they provide the accuracy and reproducibility needed for the analysis of high-purity water.

Learn more about our eluent generation solutions.

Capillary IC

Column size, injection volumes, and flow rates are scaled down by a factor of 10 to 100 in capillary IC. A capillary IC system can be left always ready to run samples as soon as they are prepared. This helps improve system stability and reduces the need for recalibration. A continuous mode of operation is possible because capillary IC systems only consume 15 mL of water a day, translating into 5.2 L a year.

The waste produced by a capillary IC system is dramatically minimized which in turn reduces disposal costs. When operated as a RFIC system, the Eluent Generation Cartridge lasts for 18 months under continuous operation. Using eluent generation, only water flows through the pumps which greatly extends the life of seals and decreases the cost of maintenance.

The Dionex ICS-5000⁺ and the Dionex ICS-4000 systems represent our latest innovations in capillary ion chromatography. As mentioned, they are also RFIC systems, which allow continuous operation up to 5000 psi when configured as an RFIC system.

Dionex ICS-4000 and ICS-5000⁺ capillary IC systems

Automated titration ion chromatography

The U.S. EPA lists a set of secondary water quality standards in addition to the National Primary Drinking Water Regulations. These secondary standards recommend maximum levels of certain analytes in drinking water that can cause a variety of issues ranging from poor taste and appearance to detrimental health effects. Some parameters, such as water hardness, can cause system problems related to scale formation. As a result, drinking water consumers and industries that use water may need to understand the secondary qualities of the water they source. Titration IC systems are fully automated to quickly and easily provide the levels of many of the secondary analytes suggested by the U.S. EPA.

Titration and IC can, as separate systems, provide the results of several different types of analyses automatically. Automated titration can be implemented to provide values such as conductivity, pH, alkalinity and carbonate hardness.

Ion chromatography provides automated analysis of the individual anion and cation concentrations. Operation of these systems separately involves loading at least two autosamplers and operating at least two instrument control/data acquisition software packages. Additionally there is the issue of data compilation. Utilizing separate IC and Titration data systems requires results from the ion chromatographs and the titrator to be combined by manual entry into a spreadsheet to calculate ion balance and permanent (or calcium) hardness. Unfortunately, this process of manual data entry increases required labor time and introduces the possibility of transcription errors, compromising the accuracy of results.

Manual versus automated sample handling time with water quality analysis

Manual operation	Manual labor time 10 samples (min)	Automated operation	Automated labor time 10 samples (min)	Time saved (min)
Measure 50 mL sample	4	Fill sample beakers	1	3
Add indicator	5	Automatic	0	5
Enter sample ID, situate beaker	3	Load autosampler	3	0
Titrate	20	Titrate	0	20
Rinse and change sample	5	Remove samples from autosampler	1	4
Total labor time, manual	37	Total labor time with automation	5	32

The METTLER TOLEDO™ titration system and liquid handler combined with two Dionex ion chromatography systems creates a fully automated water analysis system. The system can automatically dilute ion standards, removing the need for manual standards dilutions. These standards are then used to automatically build a multilevel calibration curve enabling analysis of a wide variety of drinking, surface, and ground water samples. The system can determine anions, cations, conductivity, and temperature values. Automated titration produces acid capacity numbers; total hardness and ion balance are automatically calculated for each sample.

The IC part of the solution comprises either two Dionex ICS-1100 systems or two Dionex ICS-1600 systems; one each for anion and cation analysis. IC analysis is performed simultaneously with titration for a completed analytical procedure in approximately 20 min. Additional features that enhance the productivity and accuracy of the system include automated, user selectable pH electrode calibration and verification. The Thermo Scientific Dionex AS-AP Autosampler provides high-performance, automated sample processing for ion chromatography applications. In addition, the Thermo Scientific Dionex AS-AP Sample Conductivity and pH Accessory—the first ever in an IC autosampler—allows in-line measurement of the sample conductivity and pH prior to injection. Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System (CDS) software triggers can then be used to either inject the sample or modify the sample prior to injection.

For anion analysis by IC, automatic selection of the correct sample loop size based on a conductivity value is also enabled. This eliminates time-consuming and costly rework of samples. Chromeleon Chromatography Data System software manages the entire system and integrates all results into a single convenient report ensuring automatic data analysis and reporting. The combined Titration IC system provides traceable ion analysis of water samples using the audit trail feature of Chromeleon CDS. Automating these analyses results in significant reductions in the amount of costly manual labor Results are more reliable and precise due to the removal of any need for manual data entry and calculations.

Learn more about our Titration IC solutions.

The METTLER TOLEDO titration system and liquid handler combined with two Dionex IC systems

Table of contents

Anions in municipal drinking water

Low-cost determination of anions in municipal drinking water using EPA Method 300.0 and the ICS-900 IC System

Brief of Application Brief 121

Summary

U.S. EPA 300.01 is an ion chromatography (IC) method approved by the United States Environmental Protection Agency (EPA) to determine inorganic anions (fluoride, chloride, nitrite, sulfate, bromide, nitrate, and phosphate) in municipal drinking water and wastewater. Three of these anions (fluoride, nitrite, and nitrate) are considered contaminants in drinking water under the EPA's National Primary Drinking Water Regulations. High levels of fluoride cause bone disease; nitrite and nitrate can cause birth defects. Chloride and sulfate are not harmful to public health but should not exceed 250 mg/L for the proper taste of water, according to the EPA's National Secondary Drinking Water Regulations. The EPA enforces these regulations to ensure use of approved analytical methods when analyzing water samples to meet federal monitoring requirements or to comply with drinking water regulations. The Thermo Scientific Dionex ICS-900 provides a complete solution for routine analysis of inorganic anions in water and meets all requirements specified in EPA method 300.0.

Download the full version of Application Brief 121

Equipment

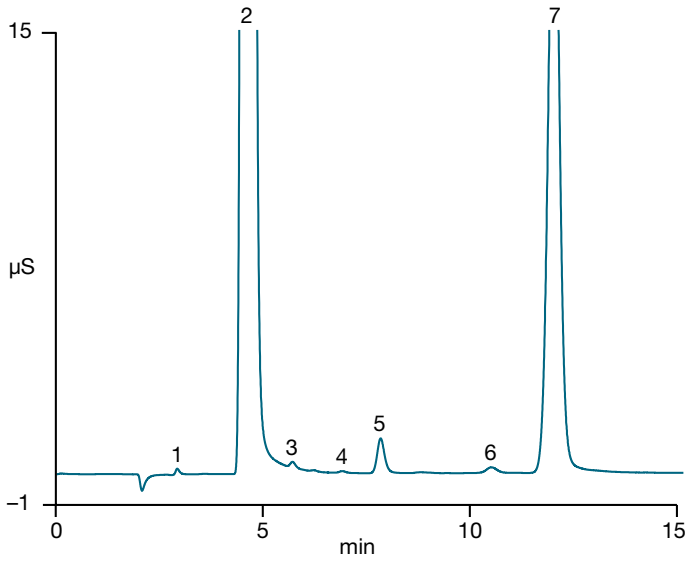
- Dionex ICS-900 System

Analysis

Ion-Chromatography

Results

See chromatogram at right.



Column:	Dionex IonPac AG22/AS22, 4 mm			
Eluent:	4.5 mM Sodium Carbonate 1.4 mM Sodium Bicarbonate			
Column Temp.:	Ambient			
Flow Rate:	1.2 mL/min			
Inj. Volume:	25 µL			
Detection:	Conductivity, Suppressed Conductivity Thermo Scientific™ Dionex™ AMMS™ 300 Anion MicroMembrane suppressor, 50 mM Sulfuric Acid			
Peaks (mg/L):	1. Fluoride	0.19	5. Nitrate	2.43
	2. Chloride	98.1	6. Phosphate	3.12
	3. Nitrite	0.54	7. Sulfate	48.2
	4. Bromide	1.22		

Separation of anions in municipal drinking water sample on the Dionex IonPac AS22 column using the Dionex ICS-900 system

Inorganic anions in municipal drinking and wastewater

Efficient and fast separations of inorganic anions in water samples using a 4 µm particle size microbore column with a high-pressure ion chromatography system

Brief of Technical Note 127

Summary

The Dionex IonPac AS18-4µm column is a newly developed high capacity column with AS18 chemistry using 4 µm resin particles. The Dionex IonPac AS18 column was developed as an alternative to the AS4A column; the Dionex IonPac AS18 is designed for use with EPA Method 300.0 (A) using electrolytically generated hydroxide eluents. These smaller resin particles allow an optimal combination of fast speed and high resolution. For the determination of inorganic anions in water samples, a comparison of the new 4 µm versus the previous 7.5 µm resin Dionex IonPac AS18 column is performed. This technical note presents the advantages of using the small-particle size microbore (2 mm i.d.) Dionex IonPac AS18-4µm column, in combination with the Dionex ICS-5000⁺ HPIC system, to obtain fast and efficient separation of inorganic anions in municipal drinking and wastewater samples.

Download the full version of Technical Note 127

Equipment

- Dionex High-Pressure ICS-5000⁺ HPIC Reagent-Free system including:
  - Dionex SP/DP Pump module
  - Dionex EG Eluent Generator module with high-pressure degas module
  - Dionex DC Detector/Chromatography module
  - Dionex AS-AP Autosampler
- Chromeleon CDS software ver. 6.8 or 7.1

Reagents and standards

- 18 MΩ-cm degassed deionized water
- Fisher Scientific reagents, ACS Grade

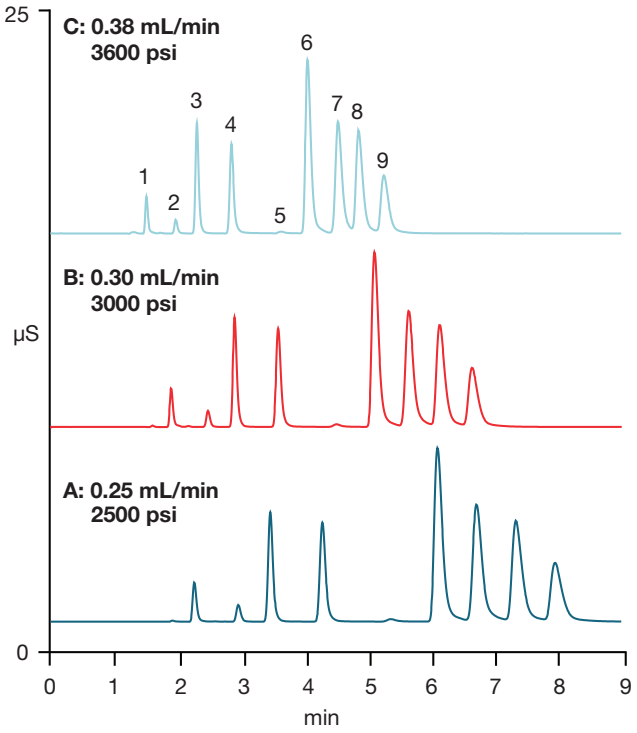
Conditions	
Columns	Dionex IonPac AG18-4µm column, 2 × 50 mm, Dionex IonPac AS18-4µm column, 2 × 150 mm
Eluent source	Dionex EGC 500 KOH Eluent Generator Cartridge, Thermo Scientific Dionex CR-ATC 500 Continuously Regenerated Anion Trap Column
Eluent	23 mM KOH
Flow rate	0.25, 0.30, 0.38 mL/min for flow rate experiments 0.38 mL/min for samples
Column temperature	30 °C
Injection volume	5 µL
Detection	Suppressed conductivity, Thermo Scientific™ Dionex™ ASRS™ 300 Anion Self-Regenerating Suppressor, recycle mode; 15, 18, 22 mA
Background conductance	< 1.0 µS
Noise	< 3 nS
System backpressure	2500–3600 psi

Analysis

High-Pressure Ion Chromatography

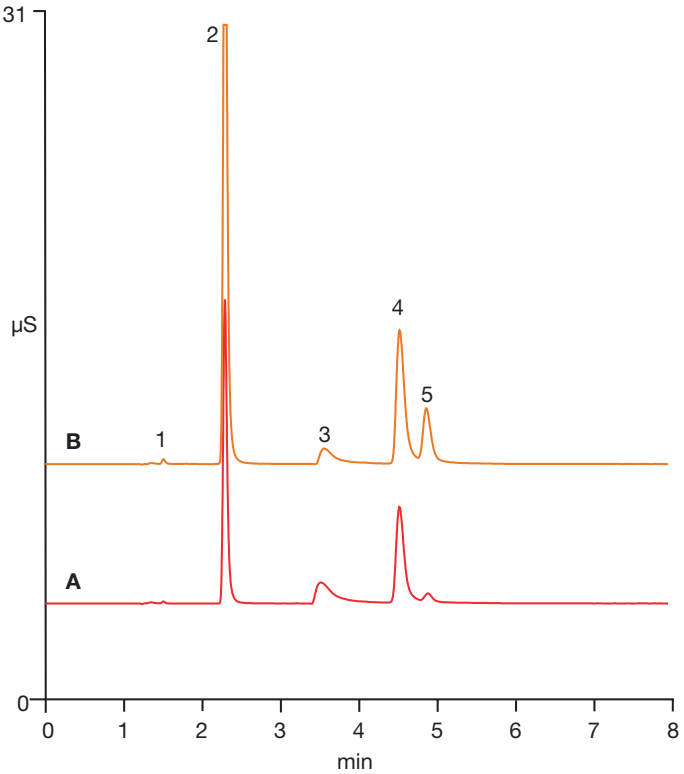
Results

See chromatograms below.



Peaks (mg/L):				
1. Fluoride	0.5	6. Bromide	10.0
2. Chlorite	1.0	7. Sulfate	10.0
3. Chloride	3.0	8. Nitrate	20.0
4. Nitrite	5.0	9. Chlorate	10.0
5. Carbonate	–		

Comparison of the separation of inorganic anions using increasing flow rates



Samples:	A: Municipal Drinking Water B: Municipal Wastewater
Sample Prep:	A: 5-fold diluted, filtered 0.45 µm B: 10-fold, filtered 0.45 µm

Peaks (mg/L):	A	B
1. Fluoride	0.11	0.56
2. Chloride	36.0	160
3. Carbonate	–	–
4. Sulfate	38.0	110
5. Nitrate	2.90	43.0

Determination of inorganic anions in municipal drinking and waste water samples

Inorganic anions in municipal drinking waters

Determination of inorganic anions in municipal water using high-pressure modular capillary ion chromatography

Brief of Technical Note 132

Summary

Ion chromatography (IC) has been widely adopted to determine inorganic anions in environmental waters, including surface, ground, drinking and wastewaters. This Technical Note describes the determination of inorganic anions in municipal drinking water samples using the Dionex ICS-5000⁺ HPIC capillary system in combination with the Dionex IonPac AS18-4µm column, which is the column of choice for compliance monitoring of water samples in accordance with U.S. EPA Methods 300.0 (A) and 300.1.

Download the full version of Technical Note 132

Equipment

- Dionex High-Pressure ICS-5000⁺ HPIC capillary Reagent-Free system including:
  - Dionex ICS-5000⁺ DP Dual Pump module with high-pressure capillary pumps
  - Dionex ICS-5000⁺ EG Eluent Generator module
  - Dionex ICS-5000⁺ DC Detector/ Chromatography module with a Dionex IC Cube and high-pressure degas cartridge
  - Dionex AS-AP Autosampler
- Chromeleon CDS software

Reagents and standards

- 18.2 MΩ-cm degassed deionized water
- Anion Standard solution made from 1000 mg/L stocks

Samples

- Municipal drinking and wastewater diluted as indicated with deionized water

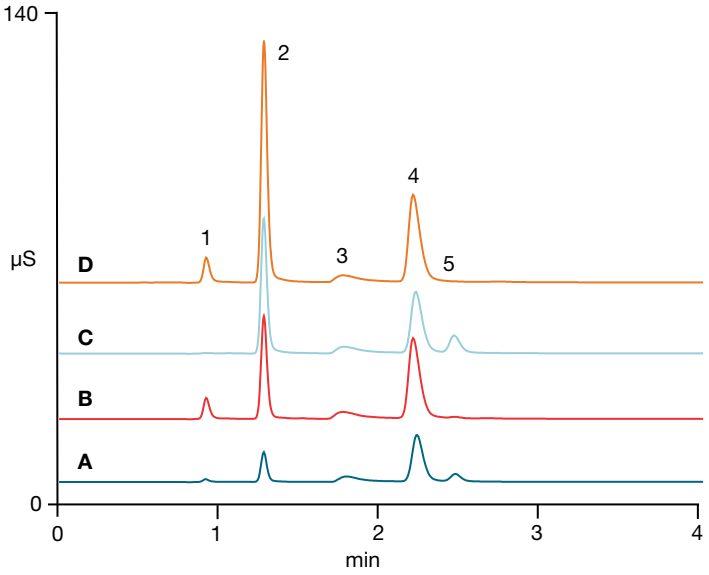
Conditions	
Column	Dionex IonPac AS18-4µm column, 0.4 × 150 mm
Eluent source	Dionex EGC-KOH Cartridge (Capillary) with Dionex CR-ATC Continuously Regenerated Anion Trap Column
Eluent	23 mM KOH
Flow rate	0.025 mL/min
Column temperature	30 °C
Injection volume	0.4 µL
Detection	Suppressed conductivity, Thermo Scientific™ Dionex™ ACES 300 Anion Capillary Electrolytic suppressor, recycle mode
Background conductance	0.3–0.5 µS
Noise	Less than 3 nS
System backpressure	~ 3500 psi

Analysis

Capillary Ion Chromatography

Results

See chromatograms below.

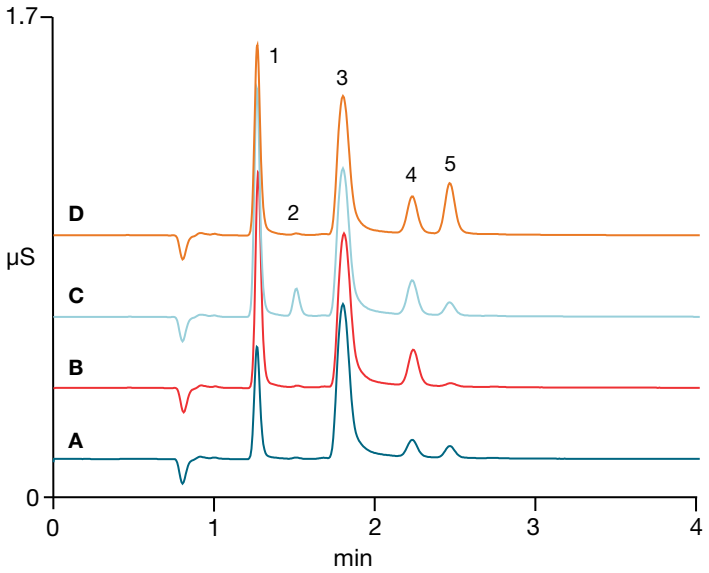


Sample Prep: A, C: Diluted 5-fold
B, D: Undiluted

Peaks (mg/L):

	A	B	C	D
1. Fluoride	0.61	0.95	0.05	1.14
2. Chloride	8.36	5.76	38.80	13.90
3. Carbonate	–	–	–	–
4. Sulfate	50.10	18.00	66.90	20.30
5. Nitrate	7.09	0.16	17.00	–

Determination of inorganic anions in municipal drinking water samples using high-pressure capillary IC



Sample Prep: Diluted 1000-fold, filtered, 0.2 µm

Peaks (mg/L):

	A	B	C	D
1. Chloride	76.5	146.0	154.0	130.0
2. Nitirite	1.5	2.1	37.4	1.6
3. Carbonate	–	–	–	–
4. Sulfate	41.6	88.9	84.8	91.8
5. Nitrate	28.8	7.2	31.7	128.0

Determination of inorganic anions in municipal wastewater using high-pressure capillary IC

Inorganic cations in municipal wastewater

Fast determinations of inorganic cations in municipal wastewater using high-pressure capillary IC

Brief of Technical Note 121

Summary

Analysis of cations and anions are important to municipal drinking water and wastewater treatment plants for compliance monitoring. Cationic determinations are necessary as part of the water monitoring program's secondary water specification of acceptable taste. In municipal wastewater, cation determinations ensure that no environmental effects occur as a result of discharging high-salt concentrations into the water system. This application demonstrates the advantages of high-pressure capillary IC using the high capacity Dionex IonPac CS16 capillary cation-exchange column to provide high sample throughput by simply increasing the flow rate on a high-pressure capable Dionex ICS-5000⁺ HPIC capillary IC, saving time and money.

Download the full version of Technical Note 121

Equipment

- Dionex ICS-5000⁺ HPIC Reagent-Free capillary system including:
  - Dionex ICS-5000⁺ SP Single Pump module with high-pressure capillary pumps
  - Dionex ICS-5000⁺ EG Eluent Generator module
  - Dionex ICS-5000⁺ DC Detector/Chromatography module with Dionex IC Cube and high-pressure degas cartridge
  - Dionex AS-AP Autosampler
- Chromeleon CDS software

Reagents and standards

- 18 MΩ-cm degassed deionized water
- Thermo Scientific Dionex Combined Six Cation II Standard (Dionex P/N 046070)

Conditions	
Columns	Dionex IonPac CS16 column, 0.5 × 250 mm
Eluent source	Dionex EGC-MSA capillary cartridge with Dionex CR-CTC Continuously Regenerated Cation Trap Column
Eluent	30 mM MSA
Flow rate	A: 0.010 mL/min; B: 0.030 mL/min
IC cube temp.*	40 °C
Compartment temperature	15 °C
Injection volume	0.4 µL
Detection	Suppressed conductivity, Thermo Scientific™ Dionex™ CCES™ 300 Cation Capillary Electrolytic Suppressor, recycle mode; A: 8 mA; B: 13 mA
Background conductance	0.3–0.8 µS
Noise	< 0.3 nS
System backpressure	A: 1250 psi; B: 3720 psi

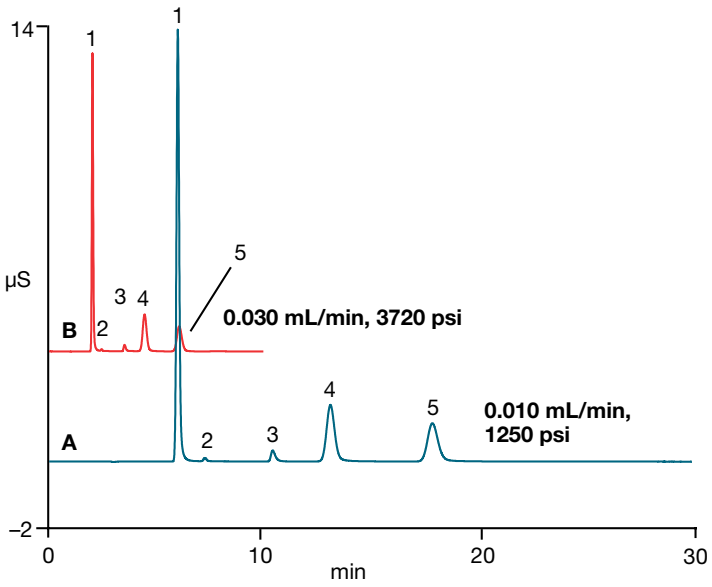
* The Dionex IC Cube heater controls the separation temperature by controlling the column cartridge temperature. The original term of "column temperature" refers to the temperature in the bottom DC compartment which is not used for capillary IC

Analysis

Capillary Ion Chromatography

Results

See chromatogram below.



Peaks (mg/L):

1. Sodium	96.0
2. Ammonium	0.80
3. Potassium	11.6
4. Magnesium	38.0
5. Calcium	52.9

Fast, high-pressure cation separations in a wastewater sample using capillary IC

Nitrite and nitrate in wastewater

Determination of nitrite and nitrate in wastewater using capillary IC with UV detection

Brief of Application Update 185

Summary

Ion chromatography with suppressed conductivity detection is an effective technique to simultaneously determine common inorganic anions in environmental water and drinking water. However in some samples such as mineral water, wastewater, and brine, accurate quantification of some anions present at low concentrations can be challenging due to the high ionic strength of the sample. Ion chromatography with UV detection provides an alternate approach for determining nitrite and nitrate without compromising sensitivity. By combining suppressed conductivity with UV detection, the suppressor reduces the background noise. The Dionex Capillary RFIC system delivers fast turnaround by reducing eluent preparation, system startup, and equilibration times. This method is a solution for nitrite analysis when high concentrations of chloride can mask the presence of nitrite.

Download the full version of Application Update 185

Equipment

- Dionex ICS-5000 Capillary IC system* including:
  - DP Dual Pump module (Capillary)
  - Dionex EG Eluent Generator module with Thermo Scientific Dionex EGC Cartridge (Capillary) and Thermo Scientific Dionex CR-TC Continuously Regenerated Trap Column (Capillary)
  - Thermo Scientific Dionex DC Detector/Chromatography Compartment with Thermo Scientific™ Dionex™ IC Cube™ module and Capillary CD Conductivity Detector
  - Dionex AS-AP Autosampler
  - Thermo Scientific Dionex ICS-Series VWD Variable Wavelength Detector with PEEK capillary cell (P/N 076072)
- Chromeleon CDS Software

*Dionex ICS-4000 system and Dionex ICS-5000+ system can be used for equivalent results

Reagents and standards

- Nitrite, 1000 mg/L (Fisher Scientific P/N AS-NO29-27)
- Nitrate, 1000 mg/L (Fisher Scientific P/N AS-NO3N9-2y)

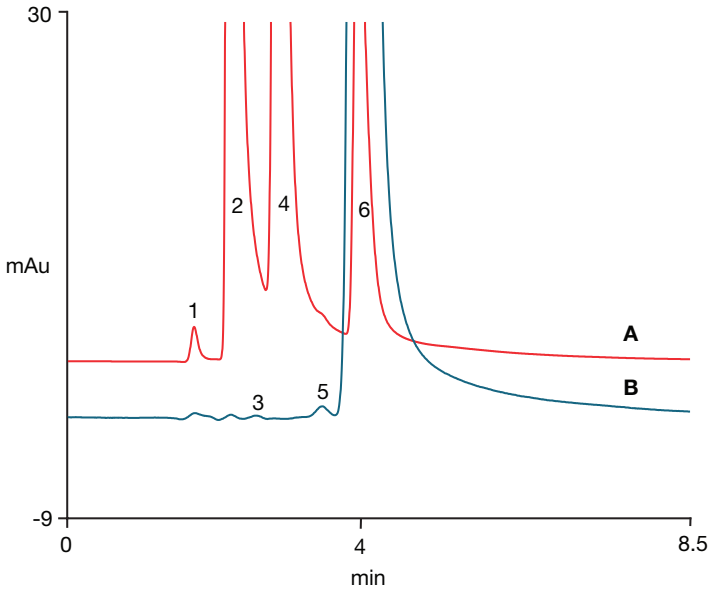
Conditions	
Column	Dionex IonPac AS18-Fast column, 0.4 × 150 mm
Eluent	33 mM KOH
Flow rate	0.015 mL/min
Injection volume	0.4 µL
Detection	A: Suppressed conductivity, Dionex ACES 300 suppressor, recycle mode B: UV, 210 nm, capillary

Analysis

Capillary Ion Chromatography

Results

See chromatogram on next page.



Peaks (mg/L):	A	B
1. Fluoride	—	—
2. Chloride	—	—
3. Nitrite	—	0.030
4. Sulfate	—	—
5. Bromide	—	—
6. Nitrate	—	48.7

Separation of inorganic anions in a municipal wastewater sample spiked with 0.030 mg/L nitrite

Oxyhalides and bromide in municipal and bottled water

Determination of trace concentrations of oxyhalides and bromide in municipal and bottled waters using a hydroxide-selective column with a reagent-free ion chromatography system

Brief of Application Note 167

Summary

Previous methods developed for determining low µg/L concentrations of bromate by direct injection have focused primarily on using columns specifically designed for carbonate eluents. Columns designed for use with hydroxide eluents have not been widely used for the determination of trace bromate in environmental waters due to their lack of appropriate column selectivity and the difficulty in preparing contaminant-free hydroxide eluents. The introduction of electrolytic eluent generation has not only eliminated the difficulty in preparing hydroxide eluents, but has simplified the development of optimized methods. In this application note, we use the Dionex IonPac AS19 column, a column specifically designed for use with hydroxide eluents and developed with an optimized selectivity for the determination of trace disinfection byproducts (DBPs) and bromide in environmental waters.

Download the full version of Application Note 167

Equipment

- Thermo Scientific Dionex ICS-2000 RFIC* System** consisting of:
  - Eluent Generator
  - Column Heater
  - Pump with Degasser
  - Dionex EluGen EGC II KOH Cartridge (P/N 058900)
  - Dionex CR-ATC Continuously Regenerated Anion Trap Column (P/N 060477)

*Also applicable to other RFIC systems

**Dionex ICS-5000+ HPIC system can be used for equivalent or improved results

- Thermo Scientific Dionex AS50 Autosampler
- Chromeleon CDS Software

Reagents and standards

- Deionized water, Type I reagent grade, 18 MΩ-cm resistivity or better
- Sodium and Potassium salts, A.C.S. reagent grade or better, for preparing anion standards (VWR or other)
- Fluoride standard 1000 mg/L, 100 mL (P/N 037158)
- Chloride standard 1000 mg/L, 100 mL (P/N 037159)
- Sulfate standard 1000 mg/L, 100 mL (P/N 037160)
- Bromide standard 1000 mg/L, 100 mL (Ultra Scientific, VWR P/N ICC-001)
- Sodium Chlorite, 80% (Fluka Chemical Co.)
- Sodium Bromate (EM Science, VWR P/N EM SX0385-1)
- Ethylenediamine, 99% (Sigma-Aldrich)

Conditions	
Columns	Dionex IonPac AS19 Analytical column, 4 × 250 mm P/N 062885) Dionex IonPac AG19 Guard column, 4 × 50 mm (P/N 062887)
Eluent	10 mM KOH from 0 to 10 min, 10–45 mM from 10 to 25 min*
Eluent source	Dionex ICS-2000 EG Cartridge with Dionex CR-ATC Trap Column
Flow rate	1.0 mL/min
Temperature	30 °C
Injection volume	250 µL
Detection	Suppressed conductivity, Thermo Scientific™ Dionex™ ASRS™ ULTRA II Suppressor, 4mm (P/N 061561), Autosuppression, Recycle Mode, 130 mA current
Background onductance	<1 µS
System backpressure	~2200 psi
Run time	30 min

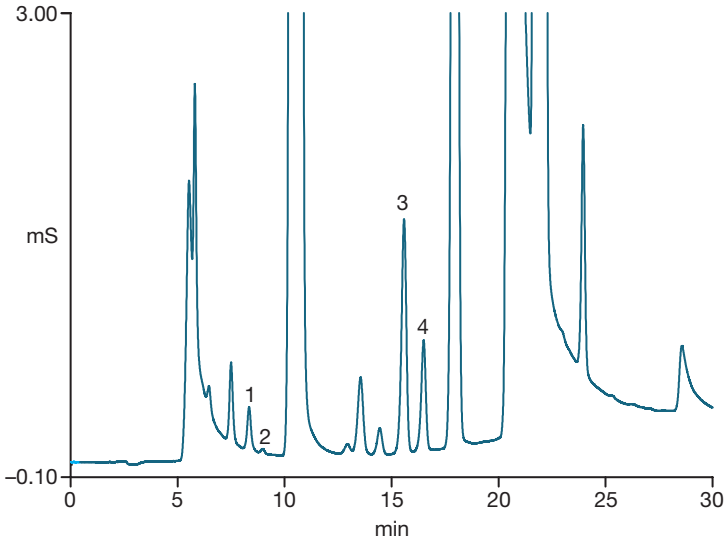
*Method returns to 10 mM KOH for 3 min prior to injection

Analysis

Reagent-Free IC

Results

See chromatogram at right.



Column: Dionex IonPac AG19 and AS19 columns, 4 mm
Eluent: 10 mM KOH 0–10 min, 10–45 mM 10–25 min
Eluent Source: Dionex ICS-2000 EG cartridge with Dionex CR-ATC trap column
Flow Rate: 1 mL/min
Temp.: 30 °C
Inj. Volume: 500 mL
Detection: Dionex ASRS ULTRA II suppressor, 4 mm recycle mode, 130 mA

Peaks (mg/L):

1. Chlorite	23
2. Bromate	5
3. Chlorate	190
4. Bromide	77

Determination of DBP anions and bromide spiked in drinking water using a 500 µL injection volume. Recoveries of trace oxyhalides and bromide spiked into environmental waters.

Disinfection byproduct anions and bromide in drinking water

Determination of disinfection byproduct anions and bromide in drinking water using a reagent-free ion chromatography system followed by postcolumn addition of an acidified on-line generated reagent for trace bromate analysis

Brief of Application Note 171

Summary

Considerable efforts have focused on developing improved analytical methods for determining trace concentrations of inorganic DBPs in drinking water to meet current regulatory requirements. Traditionally, IC with suppressed conductivity detection has been used to determine chlorite, bromate, and chlorate in environmental waters, as described in EPA Method 300.0 (B). In this application note, we demonstrate the performance of the Dionex IonPac AS19 column for EPA Method 326.0. This method allows quantification of bromate to 1 µg/L by suppressed conductivity detection with a hydroxide eluent and 0.5 µg/L using postcolumn reaction with UV detection.

Download the full version of Application Note 171

Equipment

- Dionex ICS-3000 RFIC System* consisting of:
  - DP Dual Pump or SP Single Pump
  - DC Dual Compartment with a CD conductivity detector and an Automation manager (PN 061962) equipped with a RCH-1 Postcolumn Reaction Heater (P/N 079944)
  - VWD UV/Vis Absorbance Detector with a PEEK™ analytical flow cell (PN 6074.0200)
  - Dionex AS Autosampler
  - EG Eluent Generator with a Dionex EluGen EGC II KOH Cartridge (P/N 058900)
  - Dionex CR-ATC Continuously Regenerated Anion Trap Column (P/N 060477)

*Dionex ICS-5000+ HPIC system can be used for equivalent or improved results

  - PC10 Postcolumn Pneumatic Delivery Module (P/N 050601)
  - Knitted Reaction Coil, 500 µL, potted (for RCH-1) (P/N 039349)
  - PEEK Mixing Tee (P/N 048227)
  - Four 4 L plastic bottle assemblies (P/N 063292)
  - Three bottles for external water mode of suppression
  - One bottle for 0.3 N sulfuric acid for the online conversion of KI to I_3^-
- Chromeleon CDS Software
- Polystyrene Autoselect vials with caps and septa,10 mL (P/N 055058)
- Nalgene Filter Unit, 0.2 µm nylon membrane, 1000 mL (VWR P/N 28198-514)

Reagents and standards

- Deionized water, Type I reagent grade, 18 MΩ-cm resistivity or better
- Potassium Iodide (KI) (VWR P/N BDH0264-500g)
- Ammonium Molybdate Tetrahydrate $[(NH_4)_6Mo_7O_{24}•4H_2O]$ (Sigma-Aldrich, A7302)
- Ethylenediamine (EDA) (Aldrich, 24,072-9)

- Sulfuric Acid, 36 N (J.T. Baker® Instra-Analyzed® 9673-33)
- Bromide Standard, 1000 mg/L, 100 mL (Ultra Scientific, VWR P/N ICC-001)
- Sodium Chlorite (NaClO2) (Fluka 71388, 80% pure)
- Bromate Standard, 1000 mg/L, 100 mL (Ultra Scientific, VWR P/N ICC-010)
- Sodium Bromate ($NaBrO_3$) (EM SX 03785-1)
- Sodium Chlorate ($NaClO_3$) (Aldrich, 24,414-7)
- DL-Malic Acid, Disodium salt (Sigma-Aldrich, M6773)

Conditions	
Columns	Dionex IonPac AS19 Analytical column, 4 × 250 mm (P/N 062885) Dionex IonPac AG19 Guard column, 4 × 50 mm (P/N 062887)
Eluent	10 mM KOH from 0–10 min, 10–45 mM from 10–25 min 45 mM from 25–30 min*
Eluent source	Dionex EG50 cartridge with Dionex CR-ATC trap column
Flow rate	1.0 mL/min
Temperature	30 °C
Injection volume	250 µL
Detection	Suppressed conductivity, Dionex ASRS 300 suppressor, 4mm (P/N 064554), Autosuppression, external water mode, 112 mA current
Background conductance	<1 µS
System backpressure	~2200 psi
Run time	30 min
Postcolumn reaction conditions	
UV detection	Absorbance at 352 nm (deuterium lamp)
PCR flow	0.26 M potassium iodide at 0.3 mL/min
AMMS III	0.3 N sulfuric acid at 2.5 mL/min
Postcolumn heater temperature	80 °C
UV noise	<0.1 mAU

*Method returns to 10 mM KOH for 3 min prior to injection

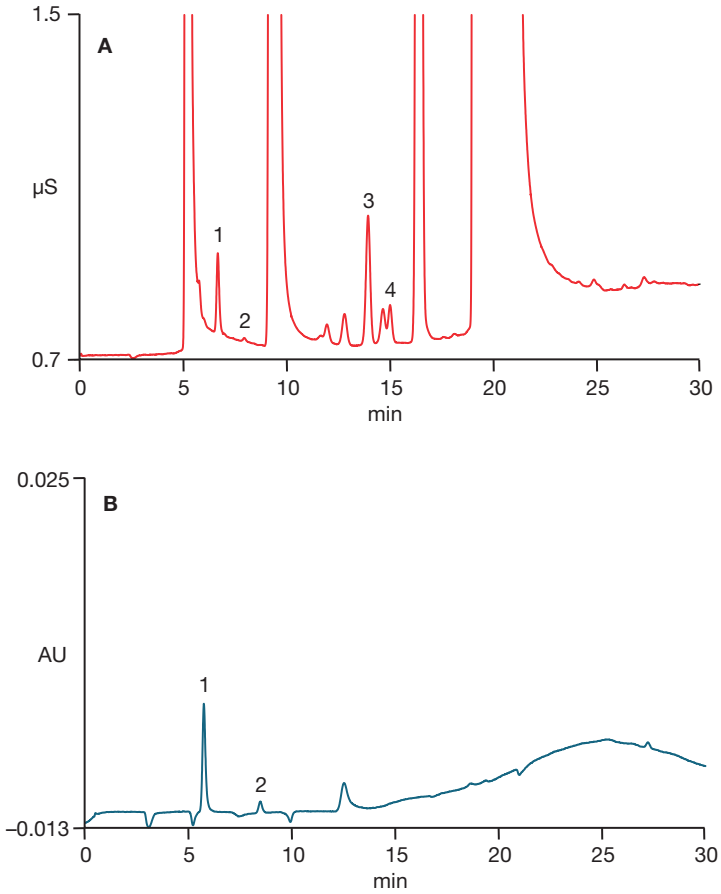
Analysis

Reagent-Free IC

Results

See table below and chromatogram on next page.

Analyte	Amount found (µg/L)	Amount added (µg/L)	Recovery (%)
Tap water A			
Chlorite	4.6	6.9	95.9
Bromate (conductivity)	0.32	1.0	95.5
Bromate (UV/Vis)	0.35	1.0	98.1
Chlorate	74.7	80.1	97.5
Bromide	34.6	39.9	95.4
Tap water B			
Chlorite	< MDL	4.6	108.0
Bromate (conductivity)	2.4	3.0	102.8
Bromate (UV/Vis)	2.8	3.0	94.7
Chlorate	62.4	69.7	96.7
Bromide	17.5	19.9	92.3
Bottled water A-1			
Chlorite	< MDL	4.9	105.3
Bromate (conductivity)	9.5	9.7	101.1
Bromate (UV/Vis)	10.8	9.7	97.3
Chlorate	< MDL	6.2	99.8
Bromide	19.0	19.9	95.0
Bottled water A-2			
Chlorite	<MDL	6.4	95.9
Bromate (conductivity)	8.7	9.7	95.7
Bromate (UV/Vis)	8.5	9.7	98.4
Chlorate	< MDL	6.4	107.6
Bromide	3.2	6.4	111.8
Bottled water B			
Chlorite	< MDL	4.9	108.3
Bromate (conductivity)	< MDL	1.0	102.4
Bromate (UV/Vis)	< MDL	1.0	104.5
Chlorate	< MDL	5.2	101.5
Bromide	10.4	9.9	90.8



Column:	Dionex IonPac AG19/AS19 columns, 4 mm
Eluent:	10 mM potassium hydroxide for 0–10 min, 10–45 mM potassium hydroxide for 10–25 min 45 mM potassium hydroxide for 25–30 min
Eluent Source:	Dionex EG50 cartridge with Dionex CR-ATC trap column
Temperature:	30 °C
Flow Rate:	1 mL/min
Inj. Volume:	250 µL
Detection:	A: Dionex ASRS 300 suppressor, 4 mm, external water mode B: Absorbance, 352 nm
Postcolumn Reagent:	Acidified potassium iodide (KI)
PCR Flow Rate:	0.3 mL/min
Postcolumn Heater:	80 °C

Peaks µg/L (ppb):

A		B	
1. Unknown	—	1. Unknown	—
2. Bromate	2.4	2. Bromate	2.8
3. Chlorate	62.4		
4. Bromide	17.5		

Determination of trace DBP anions and bromide in tap water B using suppressed conductivity detection and UV absorbance after PCR with acidified iodide

Bromate in municipal and natural mineral water

Determination of sub-µg/L bromate in municipal and natural mineral waters using preconcentration with two-dimensional ion chromatography and suppressed conductivity detection

Brief of Application Note 187

Summary

Determining low concentrations of bromate in high-ionic-strength matrices using suppressed conductivity detection is subject to potential interferences and loss of sensitivity. Although postcolumn reaction methods do not generally suffer from interferences by common anions, column overloading with high-ionic-strength samples can still cause peak broadening and an associated loss of response. In this application note, we demonstrate the use of a two-dimensional (2-D) IC system for the determination of trace concentrations of bromate in municipal and natural mineral waters with high-ionic-strength matrices. This 2-D IC method achieves bromate detection limits equivalent to or better than postcolumn addition methods. The 2-D IC method avoids the cost and disposal of the chemicals required for postcolumn configurations and simplifies the experimental setup.

Download the full version of Application Note 187

Equipment

- Dionex ICS-3000 RFIC System* consisting of:
  - DP Dual Pump module
  - EG Eluent Generator module with a dual setup
  - DC Detector/Chromatography module (single or dual temperature zone configuration)
- Dionex AS Autosampler with a 5 mL syringe (P/N 053915), 8.2 mL sampling needle assembly (P/N 061267)
- Dionex EluGen EGC II KOH cartridges (P/N 058900)

*Dionex ICS-5000+ HPIC system can be used for equivalent or improved results

- Two Dionex CR-ATC Continuously Regenerated Anion Trap Columns (P/N 060477)
- Four 4 L plastic bottle assemblies for external water mode of operation
- Chromeleon CDS Software, version 6.8

Reagents and standards

- Deionized water, Type I reagent grade, 18 MΩ-cm resistivity or better
- Bromate standard (1000 mg/L, ULTRA Scientific, N. Kingstown, RI, USA, VWR P/N ULICC-010)
- Sodium bromate ($NaBrO_3$) (EM Science, EMD Millipore, Billerica, MA, USA, SX0385-1)
- Sodium chloride (NaCl) (J.T. Baker®, Center Valley, PA, USA VWR P/N JT3625-1)
- Sodium nitrate ($NaNO_3$) (Fisher Scientific S343-500)
- Sodium bicarbonate ($NaHCO_3$) (EM Science SX0320-1)
- Sodium sulfate (Na_2SO_4) (Sigma-Aldrich, St. Louis, MO, USA, 29,931-3)
- Sodium phosphate, dibasic, anhydrous (Na_2HPO_4) (J.T. Baker 4062-1)

First-dimension conditions	
Columns	Dionex IonPac AG19 Guard column, 4 × 50 mm (P/N 062887) Dionex IonPac AS19 Analytical column, 4 × 250 mm (P/N 062885)
Eluent	10 mM potassium hydroxide 0–12 min,[a] step to 65 mM at 12 min, 65 mM 12–35 min[b]
Eluent source	Dionex EGC II KOH cartridge with Dionex CR-ATC trap column
Flow rate	1 mL/min
Temperature	30 °C (lower compartment) 30 °C (upper compartment)
Injection volume	1000 µL
Detection	Suppressed conductivity, Dionex ASRS ULTRA II suppressor, 4mm (P/N 064554), autosuppression, external water mode (flow rate: 3–5 mL/min) Current setting: 161 mA
System backpressure	~2300 psi
Expected background conductance	<0.5 µS
Noise	~1–2 nS/min peak-to-peak
Run time	35 min

[a]The step change described here should occur after the valve on system #2 has switched from the load to inject position

[b]The method equilibrates for 5 min at 10 mM KOH prior to injection

Second-dimension conditions	
Columns	Dionex IonPac AG24 Guard column, 2 × 50 mm (P/N 064151) Dionex IonPac AS24 Analytical column, 2 × 250 mm (P/N 064153)
Eluent	10 mM potassium hydroxide 0–24 min, step to 65 mM at 24 min, 65 mM 24–35 min[b]
Eluent source	Dionex EGC II KOH cartridge with Dionex CR-ATC trap column
Flow rate	0.25 mL/min
Temperature	30 °C (lower compartment) 30 °C (upper compartment)
Cut volume	2 mL (on the concentrator column)
Concentrator	Dionex IonPac TALC-ULP1 column, 5 × 23 mm (P/N 061400)
Detection	Suppressed conductivity, Dionex ASRS ULTRA II suppressor, 2 mm, auto-suppression, external water mode (flow rate: 1–3 mL/min), Current setting: 41 mA
System backpressure	~2400 psi
Expected background conductance	<0.5 µS
Noise	~2–3 nS/min peak-to-peak
Run time	35 min

[b]The method equilibrates for 5 min at 10 mM KOH prior to injection

Bromate recoveries from fortified reagent water, LSSM, and municipal drinking water matrices

Matrix	Amount found (µg/L)	Amount added (µg/L)	Replicates	Average recovery (%)	Peak area precision (RSD)
Reagent water	—	0.5	7	101.5	1.98
		5.0	7	105.6	0.66
LSSM[a]	—	0.5	7	96.1	5.75
		5.0	7	106.7	1.66
Drinking water A	0.45	0.5	7	98.2	6.06
		5.0	7	104.5	1.71
Drinking water B	1.19	0.5	7	98.7	2.51
		5.0	7	105.6	1.91

[a]LSSM = Laboratory synthetic sample matrix containing 100 mg/L each of chloride, sulfate, and bicarbonate and 10 mg/L each of nitrate-N and phosphate-P

Hexavalent chromium in drinking water

Sensitive determination of hexavalent chromium in drinking water

Brief of Application Update 179

Summary

Chromates are oxyanions (e.g., CrO_4^{2-}, $Cr_2O_7^{2-}$) of chromium in oxidation state +6. All hexavalent chromium Cr(VI) compounds are strong oxidizing agents and considered toxic and potentially carcinogenic. Hence, chromates are regulated in the environment and are a primary drinking water contaminant in the United States. For example, in 1999, the state of California established a public health goal (PHG) of 0.2 µg/L (ppb) for Cr(VI) and 2.5 µg/L for total chromium. The PHG is based on an estimated one-in-one-million lifetime cancer risk level. This method allows modifications to the existing U.S. EPA Method 218.6 to allow sufficient sensitivity for determining hexavalent chromium (i.e., Cr(VI) as CrO_4^{2-}) at the proposed California PHG level of 0.02 µg/L.

Download the full version of Application Update 179

Equipment

- Thermo Scientific Dionex ICS-2100, Dionex ICS-1600, Dionex ICS-1100,* or Dionex ICS-5000+ system including:
  - SP Single Pump or DP Dual Pump module**
  - DC Detector/Chromatography module**
  - Injection loop, 1000 µL
  - Reaction coil, 125 µL (P/N 053640), 375 µL (P/N 043700)
  - Sample syringe, 5 mL
  - Dionex ICS Series VWD UV-vis Absorbance Detector (P/N 069117, 4 wavelength or P/N 069116, single wavelength) with PEEK semi-micro flow cell, 2.5 µL, 7 mm (Victrex P/N 6074-0300) or PEEK standard flow cell, 11 µL, 10 mm (Victrex P/N 6074.0200)

- Postcolumn Delivery Configuration:
  - DP** or PC10 Postcolumn Pneumatic Delivery Package or the Thermo Scientific Dionex AXP Auxiliary Pump (P/N 063973) or Thermo Scientific Dionex AXP-MS Metering Pump (P/N 060684)
- Dionex AS-AP Autosampler
- Chromeleon CDS software
- Eluent Organizer, including 2 L plastic bottles (P/N 072057) and pressure regulator (P/N 038201)
- Polypropylene injection vials with caps (0.3 mL vial kit, P/N 055428)
- Nalgene™ 125 mL HDPE narrow mouth bottles (VWR P/N 16057-062)
- Nalgene 250 mL HDPE narrow mouth bottles (VWR P/N 16057-109)
- Nalgene 250 mL 0.2 µm nylon filter units (VWR P/N 28199-371)
- Nalgene 1000 mL 0.2 µm nylon filter units (VWR P/N 28198-514)

*With addition of the optional column heater

**For the Dionex ICS-3000 or ICS-5000+

Reagents

- Prepare all solutions from analytical reagent-grade chemicals (when commercially available). Note: There is a possibility of the presence of trace levels of chromate in some commercially available chemicals.
- Deionized (DI) water, 18 MΩ or better
- Ammonium sulfate (Mallinckrodt General P/N AR 7725)
- Ammonium hydroxide (Sigma P/N A6899)
- Sulfuric acid, 95–98% (J.T. Baker® Instra-Analyzed® P/N 9673)
- Methanol, HPLC grade (Fisher Optima P/N A454-4)
- Potassium dichromate (J.T. Baker P/N 4765-01)
- Sodium and potassium salts, A.C.S. reagent grade, for preparing the anion standards

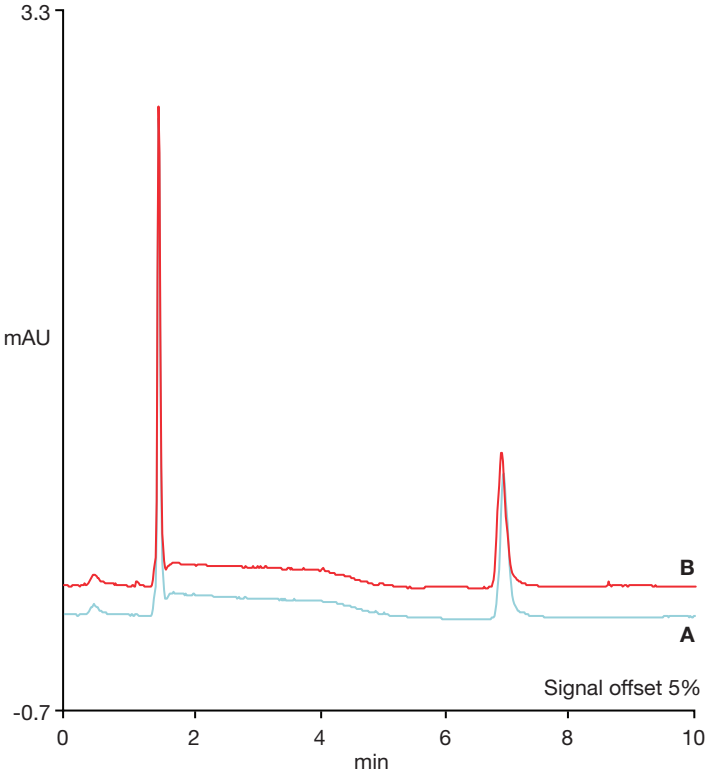
Conditions	
Columns	Dionex IonPac AG7 Guard, 2 × 50 mm (PN 063099), Dionex IonPac AS7 Analytical, 2 × 250 mm (PN 063097)
Eluent	250 mM Ammonium sulfate and 100 mM ammonium hydroxide
Eluent flow rate	0.36 mL/min
Injection volume	1000 µL (Full loop)
Temperature	30 °C
Back pressure	1700–2000 psi
Postcolumn reagent (PCR)	2 mM diphenylcarbazide, 10% methanol, 1 N sulfuric acid
PCR flow rate	0.12 mL/min
Detection	Visible absorbance, 530 nm
Noise	6–8 µAU
Run time	10 min

Analysis

Reagent-Free Ion Chromatography

Results

See chromatogram at right.



Column: Dionex IonPac AG7, 2 × 50 mm, Dionex IonPac AS7, 2 × 250 mm
Eluent: 250 mM $(NH_4)_2SO_4$, 100 mM NH_4OH
Flow: 0.36 mL/min
Inj. Vol.: 1000 µL
Postcolumn
Reagent: 2 mM diphenylcarbazide, 10% methanol, 1 N sulfuric acid
Reaction Coil: 125 µL
UV Cell: Semi-micro (PEEK), 2.5 µL

A: 0.1 µg/L Cr(VI) in DI water
B: 0.1 µg/L Cr(VI) in HIW

Determination of chromate (0.1 µg/L) in A) DI water and B) HIW on a Dionex ICS-2100 system. Postcolumn reagent delivered by an AXP pump. Flow cell: semi-micro (PEEK).

Metal cyanide complexes in water samples

Determination of metal cyanide complexes by ion chromatography with on-line sample preconcentration and UV absorbance detection

Brief of Application Note 161

Summary

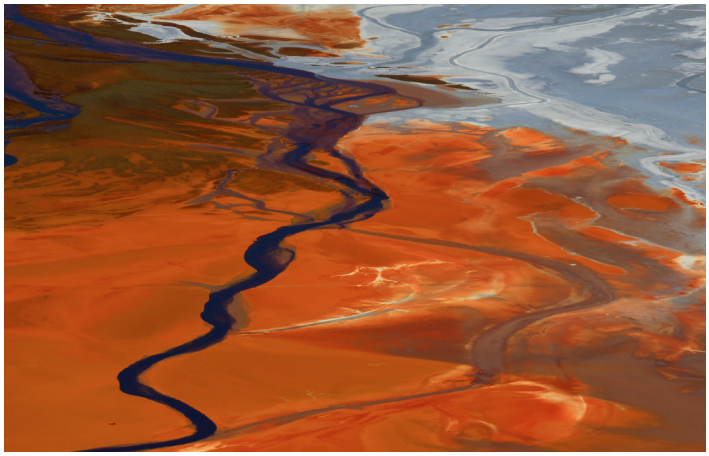
Several methods measure free cyanide, but rely on some operational definition to distinguish between weak and strong cyanide complexes. Ion chromatography resolves each individual metal cyanide complex during an automated, 30 min separation. IC thus allows a precise differentiation of complexes of limited toxicity from those of greater toxicity. This method of on-line preconcentration allows determination of metal cyanide complexes at µg/L concentrations in a variety of environmental water matrices. This method provides good recoveries for the gold, iron, and cobalt cyanide complexes in all matrices studied, and for the nickel cyanide complex in all matrices except wastewater. This method shows increased bias for the silver and copper cyanide complexes, especially in higher-ionic strength matrices.

Download the full version of Application Note 161

Equipment

- Thermo Scientific Dionex ICS-2500* IC system consisting of:
  - Thermo Scientific Dionex GS50 Gradient Pump
  - Thermo Scientific Dionex AD25 Absorbance Detector
  - Thermo Scientific Dionex AS50 AutoSelect, PEEK, with Chromatography Compartment and Chemistry Switching Option
  - Thermo Scientific Dionex AS50 Dual-Valve Needle Assembly (P/N 061267-01)
  - Thermo Scientific Dionex Sample PREP Syringe, 10 mL (P/N 055068)
- Chromeleon CDS Workstation

*Dionex ICS-5000+ system can be used for equivalent or improved results



- Thermo Scientific Dionex DQP-1 Sample/Reagent Pump (P/N 035250)
- Syringe filters (Gelman IC Acrodisk® 0.2 µm, PN 4483)
- Storage bottles, amber HDPE (VWR IRN301-0125 or 16172-144)
- Thermo Scientific Dionex Trap Columns, Metal-Free MFC-1, 2 each (P/N 037017)
- Thermo Scientific Dionex Vial Kit 10-mL polystyrene (P/N 055058)

Reagents and standards

- Copper cyanide (AlfaAesar 12135)
- Potassium dicyanoargentate (I) (AlfaAesar 12551)
- Potassium dicyanoaurate (I) (AlfaAesar 12552)
- Potassium ferrocyanide (II) trihydrate (Aldrich 22,768-4)
- Potassium hexacyanocobaltate (III) (AlfaAesar 23126)
- Potassium tetracyanonickelate (II) hydrate (Strem 93-2836)
- Sodium cyanide, 99.99% (Aldrich 43,159-1)
- Sodium hydroxide solution 50% w/w (Fisher SS254)
- Sodium perchlorate monohydrate, HPLC-grade (Fisher S490)

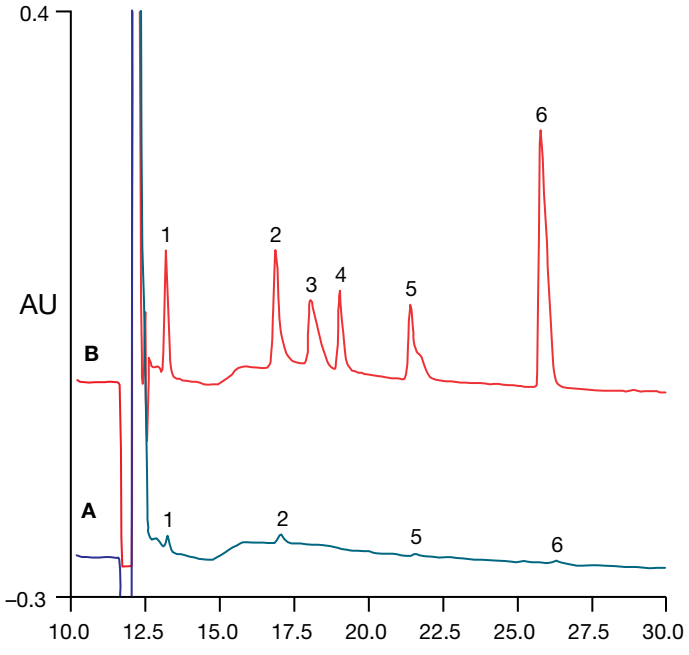
Conditions	
Columns	Dionex IonPac AS11 Analytical column, 2 × 250mm Dionex IonPac AG11 Guard column, 2 × 50 mm, 2 each Dionex IonPac ATC-3 column
Temperature	30 °C
Injection volume	5 mL
Detection	Absorbance at 215 nm
Expected system backpressure	850 psi
Noise	1–5 mAU
Run time	32 min
Flow rate	0.25 mL/min
Eluent	A: 20 mM sodium hydroxide/150 mM sodium cyanide B: 20 mM sodium hydroxide/300 mM sodium perchlorate C: 20 mM sodium hydroxide

Analysis

Ion Chromatography

Results

See chromatogram at right.



Peaks (µg/L):	A	B
1. $[Ag(CN)_2]^-$	8.6	79.5
2. $[Cu(CN)_3]^{2-}$	1.1	16.1
3. $[Au(CN)_2]^-$	—	83.7
4. $[Ni(CN)_4]^{2-}$	—	60.8
5. $[Fe(CN)_6]^{4-}$	0.1	10.1
6. $[Co(CN)_6]^{3-}$	1.2	10.7

Anion-exchange separation with preconcentration and absorbance detection at 215 nm of metal cyanide complexes in drinking water from a municipal well. Drinking water matrix blank (A) and matrix spiked with metal cyanide complexes (B) as shown.

Cyanide in municipal drinking water

Determination of cyanide in drinking water by ion chromatography with pulsed amperometric detection (ICE-PAD)

Brief of Application Note 173 (AN71020)

Summary

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. 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. 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 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.

Download the full version of Application Note 173 (AN71020)

Equipment

- Thermo Scientific Dionex ICS-3000* IC system consisting of:
  - Single Gradient Pump (SP) or Dual Gradient Pump (DP) module with degas option and gradient mixer (P/N 049135)
  - Detector and Chromatography Module (DC) with a single temperature zone and one injection valve
  - Electrochemical Detector ED (P/N 061718) with an electrochemical cell containing a combination pH-Ag/AgCl reference electrode (cell and reference electrode, P/N 061756, reference electrode P/N 071879) and a Certified Disposable Silver (Ag) working electrode (Package of 6 electrodes, P/N 063003)
  - Dionex AS Autosampler with Sample Tray Temperature Controlling option and 1.5 mL sample tray

*Dionex ICS-5000+ system can be used for equivalent or improved results

- Chromeleon CDS software version 6.7 or higher
- Filter unit, 0.2 µm 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 (vial kit, P/N 061696)
- 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 Dionex 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 MΩ-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)

Used for experiments that determined retention times and possible interferences:

- Copper reference standard, Certified 1000 ppm ±1% (Fisher Chemical, P/N SC194-100)
- Iron reference standard, Certified 1000 ppm ±1% (Fisher Chemical, P/N SI124-100)
- Nickel reference standard, Certified 1000 ppm ±1% (Fisher Chemical, P/N SN70-100)
- Sodium bromide, anhydrous (Aldrich, P/N 310506)
- Sodium iodide, anhydrous (Aldrich, P/N 383112)
- Sodium sulfide, nonahydrate, >99.99% (Aldrich, P/N 431648)
- Sodium sulfite, anhydrous (Aldrich, P/N 239321)
- Sodium thiocyanate, (Aldrich, P/N 251410)
- Sodium thiosulfate, pentahydrate (Aldrich, P/N 2929)

Conditions	
Columns	Dionex IonPac AS15 Analytical column, 2 × 250 mm Dionex IonPac AG15 Guard column, 2 × 50 mm
Flow rate	0.25 mL/min
Eluent	A: 63 mM Sodium hydroxide (31.5%) B: 200 mM sodium hydroxide)
Column temperature	30 °C
Tray temperature	10 °C
Injection volume	10 µL (PEEK sample loop, P/N 042949), full-loop injection
Detection	Pulsed Amperometric Detection (PAD)
Electrodes	Reference: pH-Ag/AgCl electrode (P/N 061879) in AgCl mode
Working electrode	Certified disposable Ag working
Background	3–13 nC versus Ag/AgCl[a]
Backpressure	~1100 psi
Noise	<7 pC
Run time	25 min
Syringe speed	4
Flush volume	250 µL

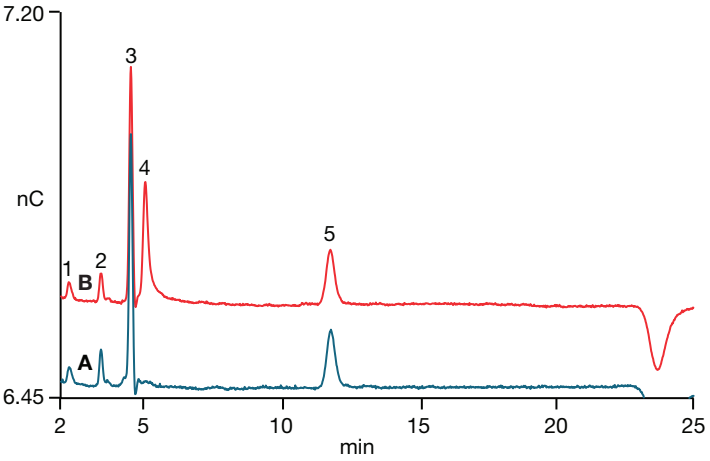
[a]The disposable silver electrodes have a background specification of –45 to +55 nC versus Ag/AgCl with a recommended waveform

Analysis

ICE-PAD

Results

See chromatogram below.



City of Sunnyvale drinking water with and without spiked cyanide

Columns:	Dionex IonPac AS15 and Guard Columns, 2 mm
Eluent:	63 mM Sodium hydroxide (31.5% Eluent B, 200 mM sodium hydroxide)
Column Temp:	30 °C
Tray Temp:	10 °C
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
Flow Rate:	0.25 mL/min
Inj. Volume:	10 µL
Detection:	PAD, Cyanide waveform
Peaks (µg/L):	1. Unknown — 2. Unknown — 3. Chloride — 4. Cyanide 10 5. Bromide —

Total cyanide in municipal waste and drinking water

Determination of total cyanide in municipal wastewater and drinking water using ion exclusion chromatography with pulsed amperometric detection (ICE-PAD)

Brief of Application Note 227

Summary

Total cyanide is defined by the US EPA as free cyanide ion and complex cyanides that are converted to hydrocyanic acid (HCN) during strong acid digestion. For drinking and surface waters, the US EPA has established a maximum contamination level (MCL) of 200 µg/L free cyanide determined by a total cyanide assay. In wastewater, the EPA specifies cyanide discharge limits by industry and size of the facility. In this Application Note, the authors describe a method with PAD using a Pt disposable working electrode and a waveform optimized for determination of total cyanide in drinking and wastewater. The authors also demonstrate linearity, detection limits, accuracy, and precision for determination of total cyanide in drinking water and wastewater samples using the EPA-approved MICRO DIST™ system and ICE-PAD.

Download the full version of Application Note 227

Equipment

- Dionex ICS-3000* IC system consisting of:
  - Single Gradient Pump (SP) module with degas option
  - Detector and Chromatography Module (DC) with single or dual heating zone, and 6-port injection valve
  - Electrochemical Detector ED (P/N 061718)
  - Dionex AS Autosampler with Sample Tray Temperature Controlling option and 1.5 mL sample tray
  - Dionex AS Autosampler with Sample Tray Temperature Controlling option, and 10 mL sample tray
  - An electrochemical cell containing a combination pH–Ag/AgCl reference electrode (cell and reference electrode, P/N 061756) and a disposable (Pt) working electrode (P/N 064440 package of six)

*Dionex ICS-5000+ can be used for equivalent or improved results

- Chromeleon CDS 6.8 Workstation
- Vial Kit, 10 mL polystyrene with caps and septa (P/N 055058)
- Knitted reaction coil, 375 µL, (P/N 043700) with two PEEK™ unions (¼-28 thread female to 10-32 thread female, P/N 042806)
- MICRO DIST System for sample distillation (Lachat Instruments/Hach Company, P/N MDD001) with user filled tube kit (Hach Company, P/N A17117 package of 100), heating block, protective gloves, test tube racks, and a small mechanical press
- Filter unit for vacuum filtration, 0.2 µm nylon (Nalgene™ Media-Plus with 90 mm filter, Nalge Nunc International, P/N 164-0020) or equivalent nylon filter
- Vacuum pump
- Syringe filter (Pall Life Sciences, GHP Acrodisc® 25 mm with 0.45 µm GHP membrane, P/N 4560T) or filter unit for sample filtration, 0.45 µm nylon (Nalgene Media-Plus with 50 mm filter, Nalge Nunc International, P/N 153-0045) or equivalent nylon filter
- PEEK Tubing:
  - Red (0.127 mm or 0.005 in i.d., P/N 052310 for 5 ft) tubing used for liquid line connections from injection valve to the guard and analytical columns, and cell.
  - Yellow (0.76 mm or 0.003 in i.d., P/N 052301 for 5 ft) tubing used for system backpressure loop.
- 50 µL PEEK sample loop (P/N 042950)

Reagents and standards

- Deionized water, Type 1 reagent grade, 18.2 MΩ-cm resistivity, freshly degassed by ultrasonic agitation and applied vacuum
- Use only ACS reagent grade chemicals for all reagents and standards
- Magnesium chloride, hexahydrate (VWR, P/N JT2444-1)
- Methanesulfonic acid (Aldrich, P/N 64280; Dionex, P/N 033478)
- pH 7 (yellow) buffer solution (VWR International, P/N BDH5046)
- pH 4 (red) buffer solution (VWR International, BDH5018)
- Sodium cyanide, anhydrous (Aldrich, P/N 20,522-2)
- Sodium hydroxide, 50% (w/w) (Fisher Chemicals, P/N SS254-500)
- Sulfuric acid (VWR, P/N JT9681-33)

For Interference Experiments

- Ammonium chloride (Aldrich, P/N 213330, FW 53.49)
- Sodium cyanate (Aldrich, P/N 185086, FW 65.01)
- Sodium sulfide, nonahydrate, > 99.99% (Aldrich, P/N 431648, FW 240.18)
- Sodium thiocyanate, (Aldrich, P/N 251410, FW 81.07)
- Sodium nitrate (Aldrich, P/N SS506, FW 84.99)
- Sodium sulfate (Aldrich, P/N 239313, FW 142.04)

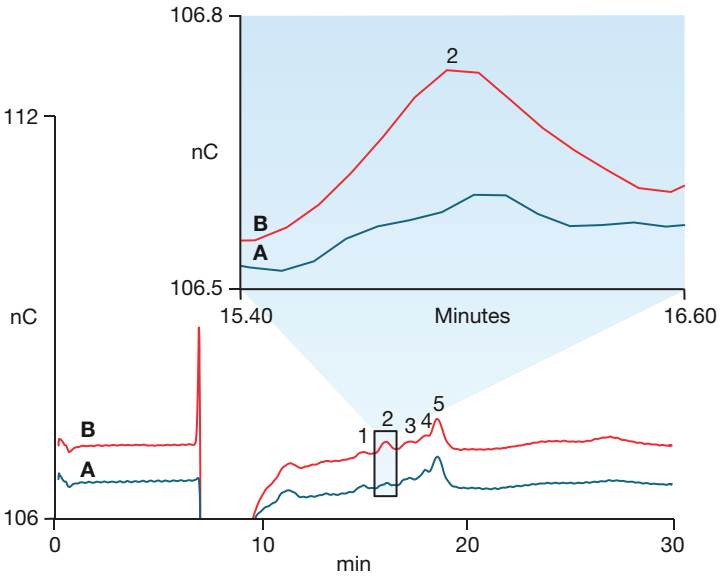
Conditions	
Columns	Dionex IonPac ICE-AG1 Guard column, 4 × 50 mm (P/N 067842) Dionex IonPac ICE-AS1 Analytical column, 4 × 250 mm (P/N 064198)
Flow rate	0.2 mL/min
Eluent	50 mM Methanesulfonic acid
Column temperature	30 °C
Tray temperature	10 °C
Injection volume	50 µL
Detection	Pulsed Amperometric Detection (PAD)
Reference electrode	pH-Ag/AgCl electrode (P/N 061879) in AgCl mode
Working electrode	Disposable Platinum
Typical background	70–120 nC
Typical system backpressure	2200 psi
Noise	20–30 pC
Typical pH	1.2–1.3
Run time	30 min

Analysis

ICE-PAD

Results

See chromatogram and table on next page.



Columns:	Dionex IonPac ICE-AG/S1 columns, 4 mm
Eluent:	50 mM Methanesulfonic acid
Temperature:	30 °C
Flow Rate:	0.2 mL/min
Inj. Volume:	50 µL
Detection:	PAD, Pt (Disposable)
Sample Prep.:	Micro Dist acid digestion
Samples:	A: Municipal drinking water + base B: Sample A + 1 µg/L cyanide

Peaks (µg/L):	A	B
1. Unknown	—	—
2. Cyanide	0.67	1.61
3–5. Unknown	—	—

Comparison of (A) Municipal drinking water, and (B) Sample A with 1 µg/L cyanide added

Average cyanide determinations over three days

Sample	Amount found (µg/L)[a]	Amount added (µg/L)	Average recovery[a] (%)
100 mM sodium hydroxide	<LOD	1.06	110 ± 6.4
Filtered 100 mM sodium hydroxide	<LOD	5.02	102 ± 1.0
10-fold dilution of certified cyanide wastewater sample (4.0 µg/L total cyanide)	4.25 ± 0.07	4.99	102 ± 0.9
Municipal drinking water	0.67 ± 0.02	0.99	97.4 ± 2.0
Filtered municipal wastewater effluent without base	<LOD	Not Tested	—
Filtered municipal wastewater effluent with base	5.99 ± 0.09	4.97	99.5 ± 1.0

[a]n = 6

Perchlorate in drinking water

Determination of perchlorate in drinking water using reagent-free ion chromatography

Brief of Application Update 148

Summary

Perchlorate (ClO_4^-) is an environmental contaminant and has been found in drinking, ground, and surface waters in several states in the U.S. However, most contaminated sites appear to be geographically confined, particularly in the western U.S., and linked to identifiable sources, such as military installations and manufacturing sites. Because perchlorate targets the thyroid gland at sufficiently high concentrations, in 1998 the EPA's Office of Groundwater and Drinking Water placed this anion on its Contaminant Candidate List (CCL) for drinking water. Currently, the EPA has not established any enforceable health regulations for perchlorate in drinking water or related matrices, although some states have set individual action levels. This application update demonstrates an approved approach compared to Application Note 134 for the determination of perchlorate in environmental samples using U.S. EPA Method 314.0. The results meet or exceed the performance requirements specified in U.S. EPA Method 314.0.

Download the full version of Application Update 148

Equipment

- A Dionex ICS-2000* RFIC System was used in this work. The Dionex ICS-2000 system is an integrated ion chromatograph that includes:
  - Eluent Generator
  - Column Heater
  - Pump Degas
  - Dionex EluGen EGC II KOH Cartridge (P/N 058900)
  - Dionex CR-ATC Continuously Regenerated Anion Trap Column (P/N 060477)

* Equivalent or improved results can be achieved using the Dionex ICS-2100 system

- Dionex AS50 Autosampler
- Dionex Chromeleon 6.5 CDS Workstation
- Suppressor External Regen Installation Kit for ExternalWater Mode (P/N 038018)
- Conductivity Meter (Thermo Scientific™ Orion,™ Model 105)
- This application update is also applicable to other RFIC systems.

Reagents and standards

- Deionized (DI) water, Type I reagent grade, 18 MΩ-cmresistance or better
- Sodium Perchlorate ($NaClO_4$) (Aldrich 41,024-1)
- Sodium Chloride (NaCl) (J. T. Baker; VWR P/N JT3625-1)
- Sodium Sulfate (Na_2SO_4) (Aldrich 29,931-3)
- Sodium Carbonate Monohydrate ($Na_2CO_3 \bullet H_2O$) (Fisher S262-3)

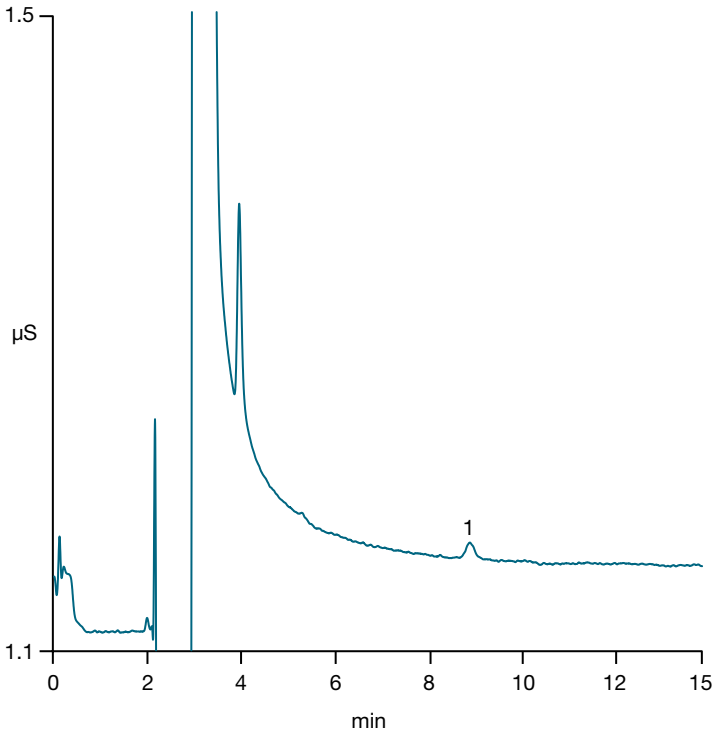
Conditions	
Columns	Dionex IonPac AS11 Analytical, 4 × 250 mm (P/N 055376) Dionex IonPac AG16 Guard, 4 × 50 mm (P/N 055377)
Eluent	65 mM potassium hydroxide
Eluent source	Dionex ICS-2000 EG with Dionex CR-ATC column
Flow rate	1.2 mL/min
Temperature	30 °C
Injection volume	1000 µL (with 10 µL cut volume from a 1100 µL sample loop)
Detection	Suppressed conductivity, Dionex ASRS ULTRA II suppressor, 4 mm, auto-suppression external water mode Power setting, 193 mA
System backpressure	~2500 psi
Background conductance	~1–2 µS µS
Noise	~1–2 nS/min peak-to-peak
Run time	15 min

Analysis

Ion Chromatography

Results

See chromatogram at right.



Columns: Dionex IonPac AG16/AS16, 4 mm
Eluent: 65 mM KOH
Eluent Source: Dionex ICS-2000 EG with Dionex CR-ATC column
Temperature: 30 °C
Flow Rate: 1.2 mL/min
Inj. Volume: 1000 µL
Detection: Dionex ASRS ULTRA II suppressor external water mode
Peaks (µg/L): 1. Perchlorate 1

Determination of 1 µg/L perchlorate in deionized water

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