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Environmental Water Analysis Applications Notebook

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Introduction

Ground and surface waters are the largest source of fresh water used for human consumption. Protecting the safety of these environmental waters is critical to ensure sustainable drinking water resources. Wastewater is typically discharged into ground and/or surface waters both by industry and water treatment facilities. These wastewaters carry a variety of toxic pollutants including heavy metals and organic toxins which are harmful to not only the environment but also to human health. The wastewater can also be recycled to be used as a source for drinking water. As a result, the water discharged by these facilities must be monitored for contaminants.

A prime example is wastewater produced from hydraulic fracturing (fracking) processes which contains salts (anions and cations), metals, and radioisotopes mobilized from the hydrocarbon-rich shale layer. When the high-pressure is used to crack the shale, wastewater flows to the surface where it is subject to different fates. Fracking wastewater can be disposed of into injection wells if no further use is required. If the wastewater will be used in subsequent fracking events, the water is first desalinated prior to mixing with additives. Lastly, wastewater can be sent to publicly owned treatment works (POTWs) to be treated prior to surface

water discharge. Due to the health and environmental safety concerns discussed above, anions, cations and organic acids found in fracking wastewaters that will not be directly disposed of should be monitored.

Under the U.S. Clean Water Act, the National Pollutant Discharge Elimination System (NPDES) permit program regulates point sources (ditches, pipes, etc.) that discharge pollutants into wastewaters. As a result, industrial, municipal, and other facilities must obtain permits if their discharges go directly to surface waters. Similarly, the European Commission Council Directive 91/271/EEC regulates urban wastewater discharges as well as discharges from certain industrial sectors.

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

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Anions

- Determination of Inorganic Anions in Wastewater by Ion Chromatography
- Determination of Inorganic Anions in Environmental Waters Using a Hydroxide-Selective Column
- Determination of Iodide in Seawater and Other Saline Matrices Using a Reagent-Free Ion Chromatography System with Suppressed Conductivity and UV Detection
- Determination of Total Nitrogen and Phosphorus in Wastewaters by Alkaline Persulfate Digestion Followed by IC
- Monitoring Anions and Cations During Desalination
- Fast Determinations of Inorganic Ions in Salton Sea Samples Using a High-Pressure IC System
- Determination of Anions in Fracking Flowback Water From the Marcellus Shale Using Automated Dilution and Ion Chromatography
- Determination of Anions in Acid Rain by Ion Chromatography Example 1
- Determination of Anions in Acid Rain by Ion Chromatography Example 2
- Determination of Inorganic Anions in Acid Rain Using a High-Pressure Modular Capillary Ion Chromatography System



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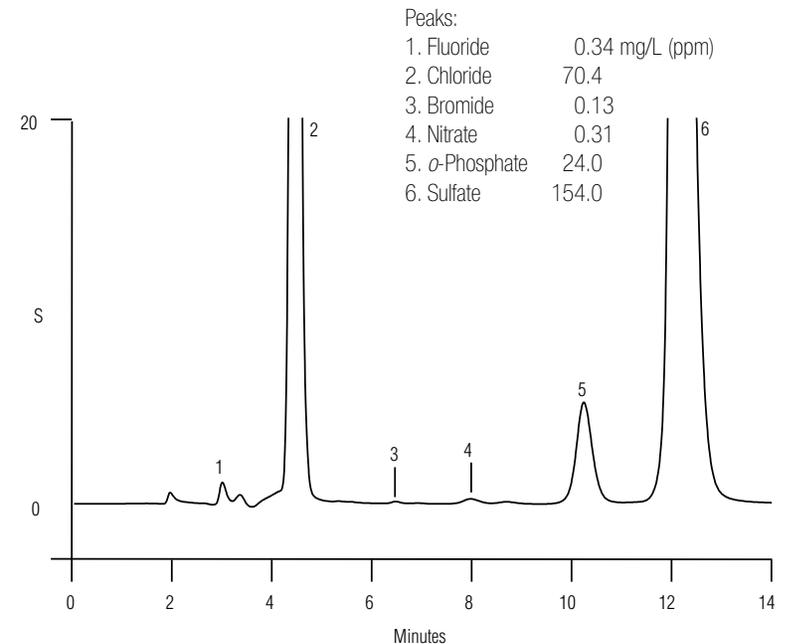
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The determination of common inorganic anions in environmental waters, such as wastewater and drinking, ground, and surface waters, is one of the most important applications of IC worldwide. Water quality in the U.S. is legislated through the Safe Drinking Water Act (SDWA) and the Clean Water Act (CWA). The SDWA ensures the integrity and safety of U.S. drinking water, and the goal of the CWA is to reduce the discharge of pollutants into U.S. waters. Many other industrialized countries have similar health and environmental standards and a considerable number of regulatory IC methods have been published worldwide (e.g., in Germany, France, Italy, and Japan) for the determination of anions in wastewater. In addition, many standards organizations (including ISO, ASTM, and AWWA) have validated IC methods for the determination of inorganic anions in wastewater.

Conditions

Columns:	Dionex IonPac AG14, 4 × 50 mm Dionex IonPac AS14, 4 × 250 mm
Eluent:	3.5 mM Sodium carbonate/1.0 mM Sodium bicarbonate
Run Time:	<14 min
Flow Rate:	1.2 mL/min
Injection Volume:	50 µL
Detection:	Suppressed conductivity, Dionex ASRS ULTRA suppressor (4 mm), recycle mode, 100 mA current
System Backpressure:	~1600 psi
Background Conductance:	~17 µS

In this application note, the determination of inorganic anions in wastewater and other environmental waters using conditions that are consistent with those in U.S. Environmental Protection Agency (EPA) Method 300.0. The Thermo Scientific™ Dionex™ IonPac™ AS14 column provides suitable performance for the determination of inorganic anions in low ionic strength wastewater samples and similar matrices, such as drinking, raw, and surface waters, as outlined in U.S. EPA Method 300.0.



Determination of inorganic anions in domestic wastewater using a Dionex IonPac AS14 column.

Inorganic Anions in Environmental Waters

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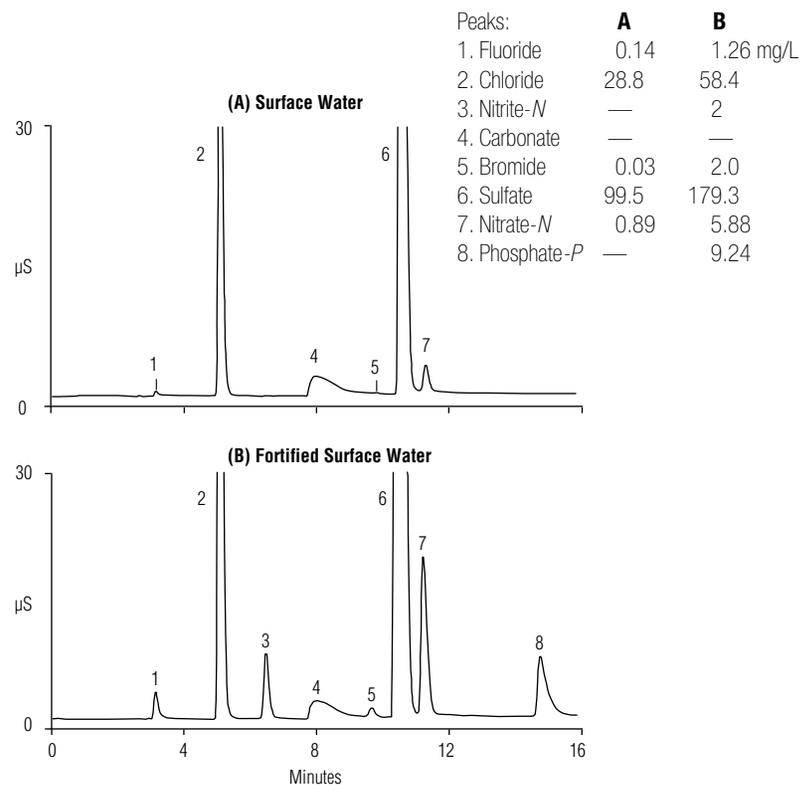
Traditionally, columns designed for use with carbonate/bicarbonate eluents have been used for determining inorganic anions in environmental samples. Columns that use hydroxide eluents (i.e., hydroxide-selective columns) have not been as widely used for routine analysis of inorganic anions in environmental waters due to the lack of appropriate selectivity and difficulty in preparing contaminant-free hydroxide eluents. The introduction of automated, electrolytic eluent generation has eliminated the difficulty in preparing hydroxide eluents.

The use of a Reagent-Free™ ion chromatograph (RFIC™) system with a Dionex IonPac AS18 column and electrolytic eluent generation is an improved approach to the routine determination of inorganic anions in environmental waters. Quantitative recoveries were obtained for all common inorganic anions spiked into typical environmental waters using the Dionex IonPac AS18 column. In addition, electrolytic generation of

potassium hydroxide eliminates the need to manually prepare eluents, increasing the level of automation, ease of use of the IC system, and data reproducibility. This approach to U.S. EPA Method 300.0 allows improved method performance for resolution, linearity, precision, and method detection limits (MDLs).

Conditions

Column:	Dionex IonPac AS18 Analytical, 4 × 250 mm Dionex IonPac AG18 Guard, 4 × 50 mm
Eluent:	22–40 mM KOH from 7–8 min
Eluent Source:	Thermo Scientific Dionex ICS-2000 System with Thermo Scientific Dionex CR-ATC Continuously Regenerated Anion Trap Column
Flow Rate:	1.0 mL/min
Temperature:	30 °C
Injection:	25 µL
Detection:	Suppressed conductivity, Dionex ASRS ULTRA Suppressor, 4 mm AutoSuppression, recycle mode 100 mA current System
Backpressure:	~2500 psi
Run Time:	20 min



Determination of inorganic anions in (A) surface water and (B) fortified surface water using the Dionex IonPac AS18 column.

Iodide in Seawater and Other Saline Matrices



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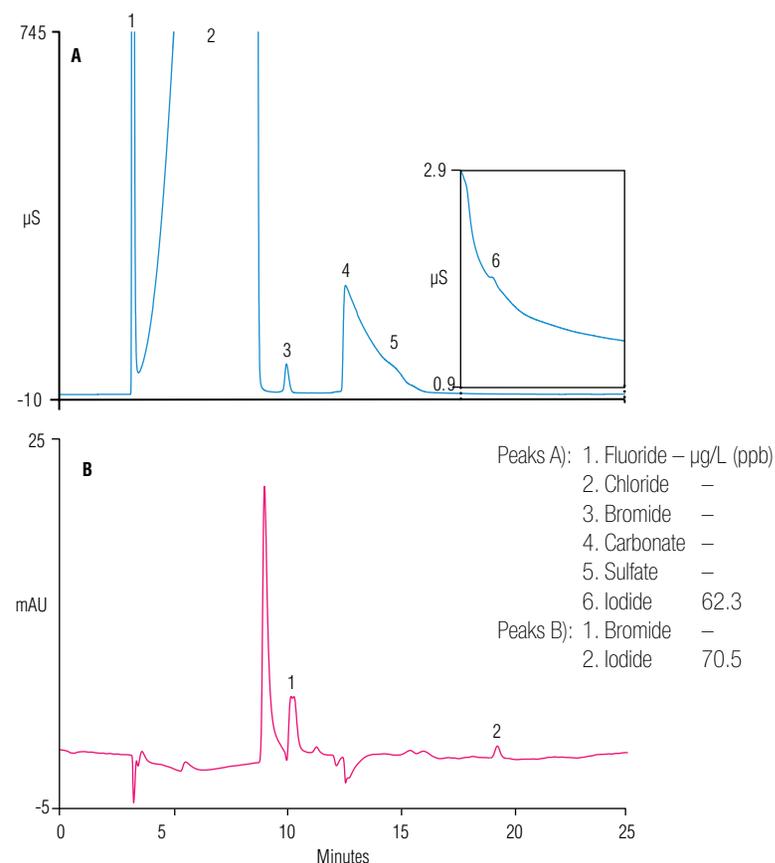
Iodine is an essential nutrient found in seawater, seafood, and iodine-enriched food, such as iodized table salt. The most common forms of iodine in the diet are iodide and iodate, with additional iodo-organic compounds providing a small fraction of bioavailable iodine. Iodine deficiency affects thyroid hormone production and leads to developmental diseases, goiter, and paralysis. Because iodide is an essential source of iodine, there is need to determine iodide in a variety of matrices. Seawater is a large natural source of iodide, typically containing 50–60 µg/L iodide with a wide range of observed concentrations.

Conditions

Columns:	Dionex IonPac AG20, 2 × 50 mm Dionex IonPac AS20, 2 × 250 mm
Eluent:	13 mM KOH from 0–10 min, 13–45 mM KOH from 10–15 min, 45 mM KOH from 15–25 min*
Eluent Source:	Thermo Scientific Dionex EGC II KOH Cartridge with Dionex CR-ATC Column
Flow Rate:	0.25 mL/min
Temperature:	30 °C (column & detector compartment)
Injection Volume:	10 µL
Detection:	A) Suppressed conductivity, Thermo Scientific™ Dionex™ ASRS™ 300 Anion Self-Regenerating Suppressor, 2 mm, external water mode, 28 mA suppressor current B) UV, 223 nm
Background Conductance:	<1 µS
Noise:	~0.5–1.0 nS (conductivity) ~0.05–0.10 mAU (UV)
System Backpressure:	~2600 psi

* The column equilibrates for 10 min at 13 mM KOH prior to injection.

In this application note, the use of the Dionex IonPac AS20 column with electrolytically generated hydroxide eluent to determine iodide in saline matrices. The high capacity of the Dionex IonPac AS20 column allows sample analysis without dilution or sample pretreatment. The method was shown to be accurate by recovering iodide in a variety of samples including natural seawater and iodized table salt.



Determination of iodide in seawater collected at Half Moon Bay, CA.

Nitrogen and Phosphorus in Wastewaters



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Although inorganic nitrate and phosphate can be determined directly in natural waters, this does not provide the wider environmental significance of the organic nitrogen and phosphorus fractions that contribute to the total nutrient loading in bodies of water. In water, nitrogen exists as inorganic and organic species. Inorganic nitrogen is present in the oxidized form (e.g., nitrite and nitrate) and reduced form (e.g., ammonia/ammonium and dinitrogen gas). Organic nitrogen is available in a variety of complex forms such as amino acids, proteins, humic acids, and urea. However, before being used as a nutrient, the organic nitrogen must first be converted to ammonia. Total nitrogen (TN) is the sum of all forms of nitrogen in the water sample.

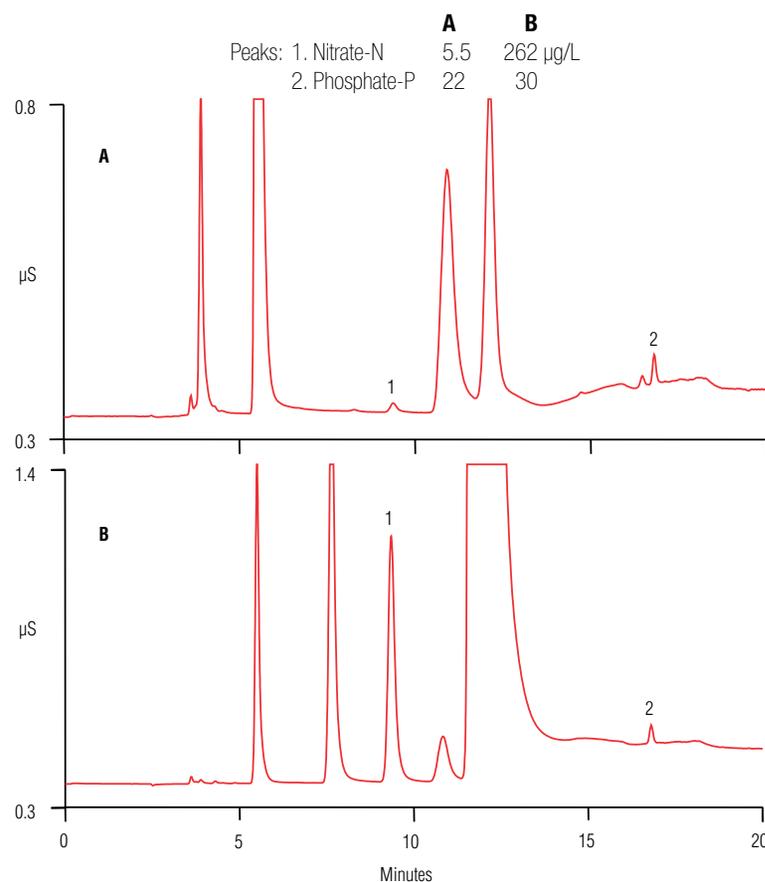
Phosphorus exists as inorganic orthophosphate, polyphosphate, and organic phosphate. Particulate phosphorus—found in suspension or sediment—consists of plants, animals, phosphorus in minerals, and

phosphate adsorbed on an iron oxyhydroxide mineral surface. Total phosphorus (TP) is a measure of all forms of phosphorus found in water.

This study demonstrates the simultaneous determination of TN and TP using a high-capacity hydroxide-selective Dionex IonPac AS19 Analytical Column with suppressed conductivity detection after alkaline persulfate digestion.

Conditions

Columns:	Dionex IonPac AG19 Guard, 2 × 50 mm Dionex IonPac AS19 Analytical, 2 × 250 mm
Eluent:	–6–10 min at 20 mM KOH, 10–12 min from 20 to 50 mM, 12–20 min at 50 mM
Eluent Source:	Dionex EGC III KOH cartridge with Dionex CR-ATC column
Flow Rate:	0.30 mL/min
Injection Volume:	5 µL (using a 5 µL sample loop in full-loop mode)
Detection:	Suppressed conductivity, Dionex AERS 500 suppressor (2 mm), recycle mode, 38 mA current
System Backpressure:	~2450 psi
Background Conductance:	~0.4 µS
Noise:	~0.5–1 nS/min, peak to peak
Run Time:	20 min





Inorganic Anions and Cations During Desalination

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Because of the growing demand for water and the limited supply of fresh water, desalination increasingly is being used to produce potable and irrigation water from salty or brackish water. A wide variety of desalination techniques are currently available and more are being developed. Most use distillation or membrane techniques. The performance of desalination processes is evaluated by monitoring the common anions and cations in the feed, intermediate, and final water product. For the final drinking water product, IC is approved for monitoring primary and secondary anions according to U.S. EPA Method 300.0 and Federal and State regulatory agencies ensure that U.S. National Primary and Secondary Drinking Water Standards are met.

This application note demonstrates the Dionex IonPac AS18 and Dionex IonPac CS12A columns with electrolytically generated hydroxide and MSA eluents simultaneously determining anions and cations in saline and drinking water matrices. The capacities of the Dionex IonPac AS18 and Dionex IonPac CS12A columns allow sample analysis with minimal sample pretreatment. The RFIC-EG system allows continuous operation of the instrument with minimal maintenance. The methods were shown to be accurate by the good recovery of anions and cations in a wide variety of samples including natural and artificial seawater and drinking water. These methods are robust for all ion-monitoring needs of a typical desalination facility and support a varying range of matrices from seawater to drinking water.

Conditions

Anion Determinations

Columns:	Dionex IonPac AG18, 2 × 50 mm Dionex IonPac AS18, 2 × 250 mm
Eluent:	22 mM KOH from 0–7 min, 22–40 mM KOH from 7–8 min, 40 mM KOH from 8–18 min*
Eluent Source:	Dionex EGC II KOH Cartridge with Dionex CR-ATC Column
Injection Volume:	4 µL
Flow Rate:	0.25 mL/min
Detection:	Suppressed conductivity, Dionex ASRS Suppressor, 2 mm, recycle mode, suppressor current 15 mA
Background Conductance:	<1 µS

Cation Determinations

Columns:	Dionex IonPac CG12A-5 µm, 3 × 30 mm Dionex IonPac CS12A-5 µm, 3 × 150 mm
Eluent:	20 mM MSA
Eluent Source:	Thermo Scientific Dionex EGC II MSA Cartridge with Dionex CR-ATC Column
Injection Volume:	10 µL
Flow Rate:	0.50 mL/min
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ CSRS™ 300 Cation Self-Regenerating Suppressor, 2 mm, recycle mode, suppressor current 30 mA
Background Conductance:	<0.5 µS

* The column equilibrates for 2 min at 22 mM KOH prior to injection.





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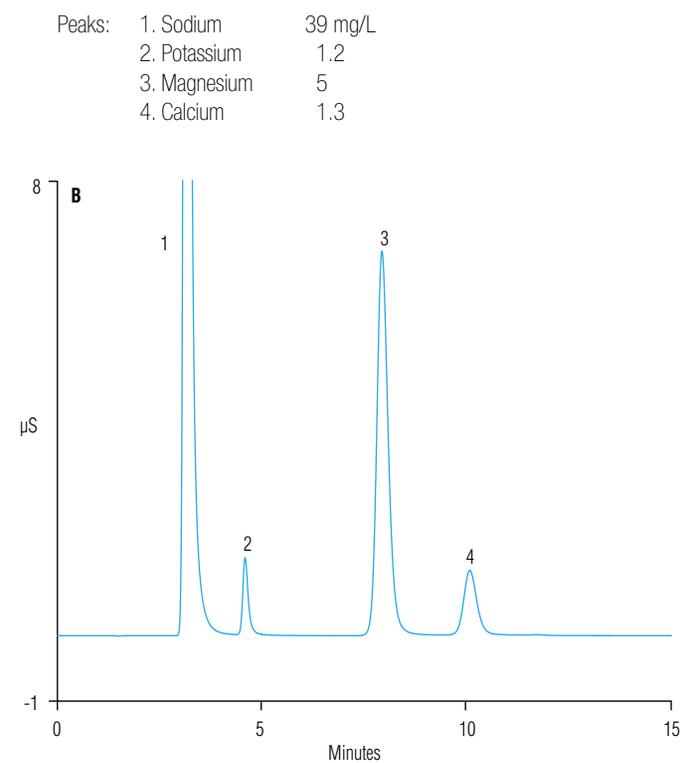
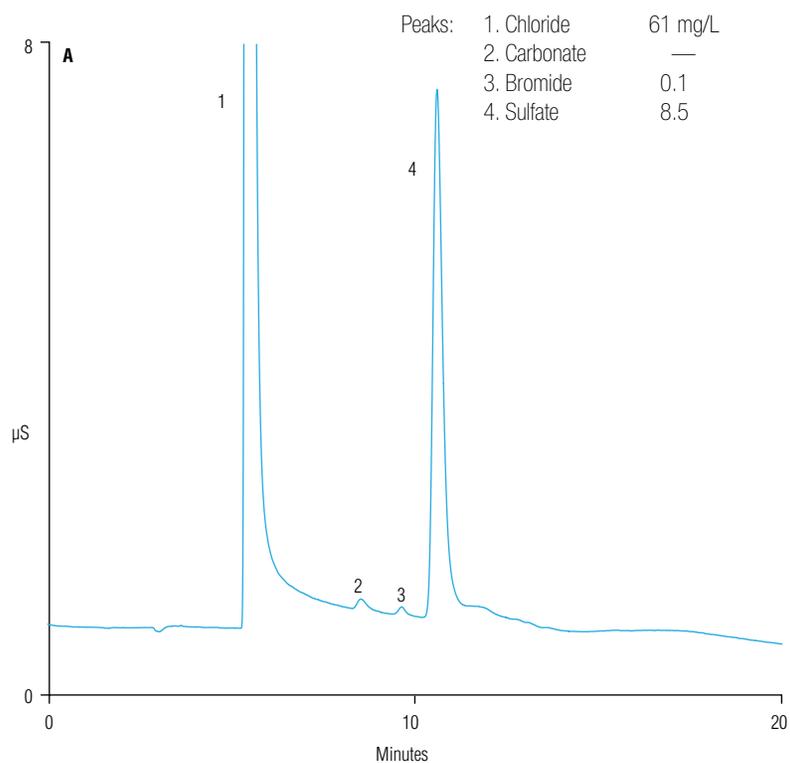
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San Francisco, CA bay water: determination of common (A) inorganic anions using the Dionex IonPac AS18 column and (B) cations using the Dionex IonPac CS12A column.



Inorganic Ions in Salton Sea Samples



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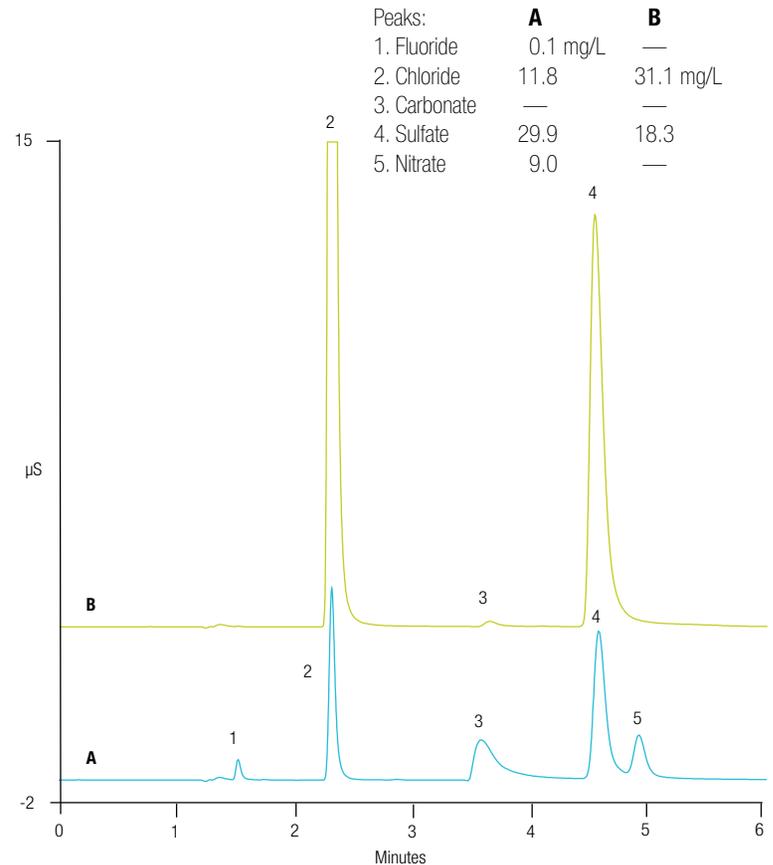
Monitoring salinity levels in estuarine ecosystems is critical to preventing salinity-specific diseases. In the Chesapeake and Delaware bays, for example, pathogens infecting oysters are restricted to areas that fall within discrete salinity levels. IC can be used to measure ions in high salinity water samples.

Conditions

Column:	Dionex IonPac AG18-4 μ m, 2 \times 30 mm Dionex IonPac AS18-4 μ m, 2 \times 150 mm
Eluent Source:	Dionex EGC 500 KOH Eluent Generator Cartridge Dionex CR-ATC 500 Column
Eluent:	23 mM KOH
Flow Rate:	0.25, 0.35, 0.45, 0.55 mL/min for flow rate experiments 0.55 mL/min for samples
Injection Volume:	5 μ L
Column Temperature:	30 $^{\circ}$ C
Detection:	Suppressed conductivity, Dionex ASRS 300 Suppressor, recycle; 15, 26, 32 mA
Background Conductance:	1–2 μ S
Noise:	2–3 nS
System Backpressure:	2000*–4300 psi

* Backpressure tubing was added prior to the injection valve for those conditions running below <2000 psi to bring the total system pressure above 2000 psi needed for the Dionex EGC 500 cartridge.

In this study we demonstrate the fast separations of inorganic anions and cations in a Salton Sea surface water sample and a neighboring municipal drinking water sample using ion-exchange chromatography on a high pressure capable Thermo Scientific Dionex ICS-5000+ HPIC system.



Fast anion determinations of the Salton Sea area water samples using high-pressure IC.

Anions in Fracking Flowback Water



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Fracking has been in use since the 1940s primarily for oil extraction, however recent refinements to this process have enabled the exploitation of energy resources that had previously been inaccessible. While fracking provides financial benefits to both local and national economies, it has not been without controversy. Inadvertent spills or the dumping of fracking flowback (fracking solution that returns to the surface via the well bore) into unlined collection ponds can contaminate groundwater.

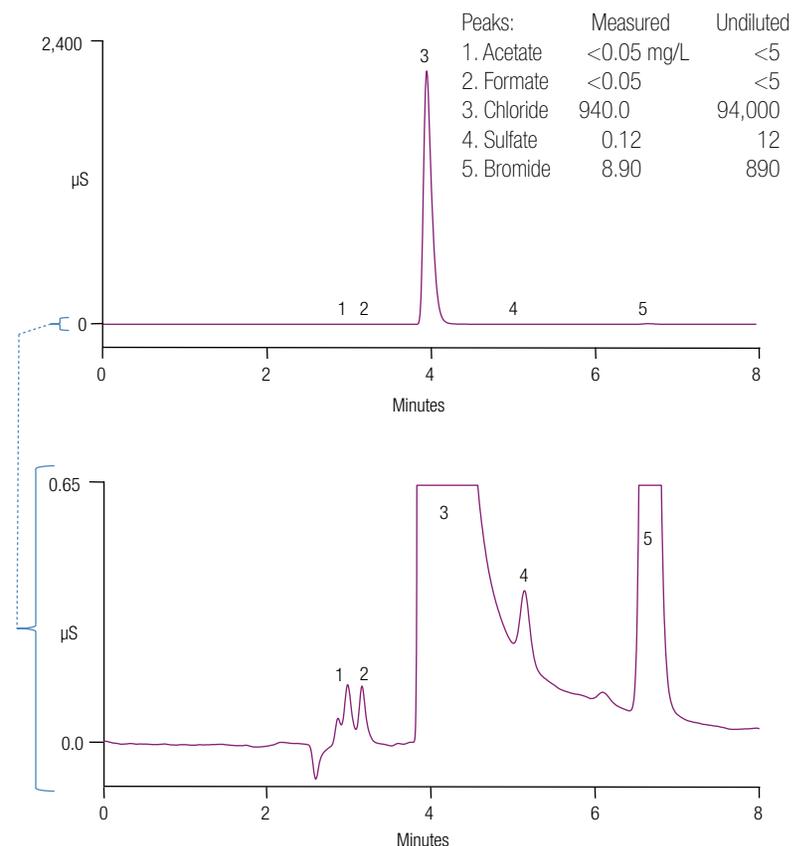
This technical note demonstrates that by using the Thermo Scientific Dionex AS-AP Sample Conductivity and pH Accessory in combination with Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data

Conditions

Columns:	Dionex IonPac AG18/AS18, 4 mm
Eluent Source:	Thermo Scientific Dionex EGC III KOH Cartridge
Eluent:	23 mM or 39 mM KOH
Flow Rate:	1.0 mL/min
Column Temperature:	30 °C
Injection Volume:	25 µL
Detection:	Suppressed conductivity, Dionex ASRS Suppressor*, recycle mode, 57 mA (23 mM KOH); 97 mA (39 mM KOH)
Background Conductance:	<2 µS
Noise:	<3 nS
System Backpressure:	2100 psi

* Equivalent or improved results can be obtained using the Thermo Scientific™ Dionex™ AERS™ 500 Anion Electrolytically Regenerated Suppressor.

System (CDS) software, samples containing high levels of anions, such as fracking flowback wastewater, can be automatically diluted prior to injection if a preset conductivity maximum is exceeded. This prescreening eliminates repeat analysis, thereby reducing eluent waste, extending column life, and providing fast, accurate results.



Determination of inorganic anions and organic acids in fracking flowback (F10).



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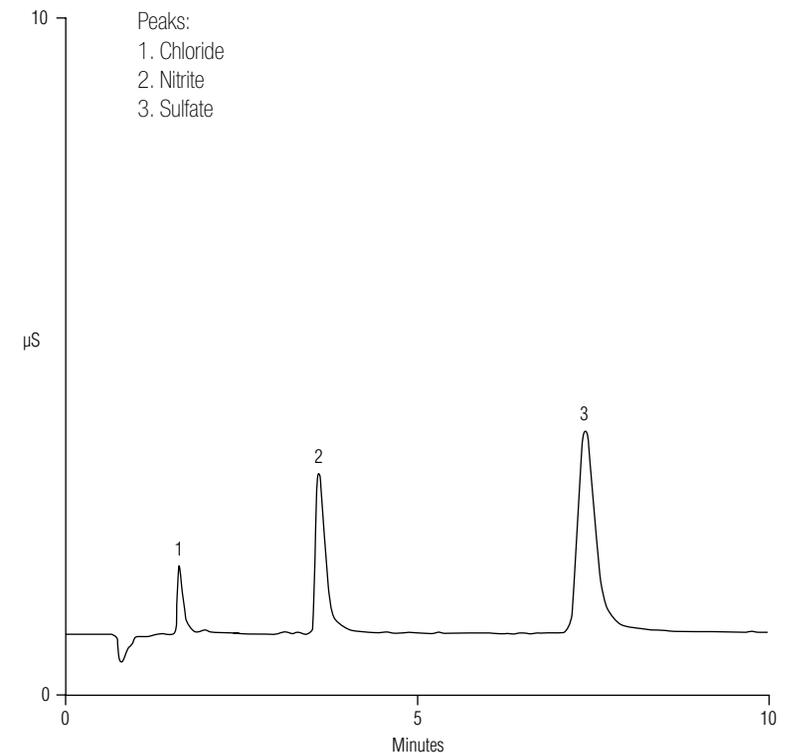
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The increase in the acidity of wet and dry depositions has been attributed to the increase of SO₂ and NO₂ emissions from stationary and mobile sources. The problem of acid rain has reached dramatic proportions throughout many regions of the United States, Canada, Europe, and Japan. Monitoring of air and rain are essential for assessing the effects of pollutants on global ecology.

Conditions

Columns:	Dionex IonPac AS4A-SC 2 mm analytical and Dionex IonPac AG4A-SC 2 mm guard (Alternatively, 4-mm analytical and guard columns may be used; changes in experimental conditions are noted below in parentheses.)
Eluent:	1.8 mM Sodium carbonate/1.7 mM Sodium bicarbonate
Flow Rate:	0.5 mL/min (2.0 mL/min for 4 mm columns)
Injection Volume:	5.0 µL (20 µL for 4 mm columns)
Detection:	Suppressed conductivity
Suppressor:	Dionex ASRS suppressor, 2 mm (or 4 mm), AutoSuppression mode; or Dionex AMMS suppressor, 2 mm (or 4 mm)
Regenerant:	50 mN Sulfuric acid with AMMS
Regenerant Flow:	5–7 mL/min for AMMS

This application note describes the established ion chromatographic method for the determination of anions in rainwater samples extended to use of microbore columns and accessories. A rainwater standard from the National Institute of Standards and Technology (NIST) was analyzed, verifying the accuracy of the method.



Analysis of simulated rainwater, SRM 2694-II.



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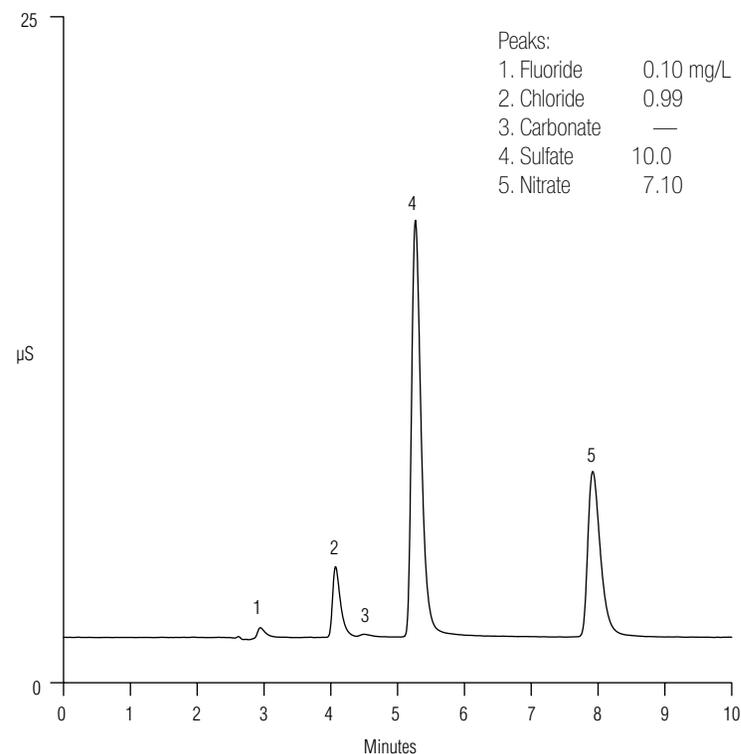
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The increase in acidity of wet and dry atmospheric depositions has been attributed to the increase of SO₂ and NO_x emissions from stationary and mobile pollution sources. The acid rain problem has reached dramatic proportions throughout many regions of the United States, Europe, Canada, and Japan. Monitoring air and rain is essential, not only for assessing the effects of pollutants on global ecology, but also for monitoring the progress of pollution abatement efforts. IC with suppressed conductivity detection offers excellent precision and accuracy for the measurement of such pollutants and has been specified by the U.S. EPA as the method of choice for determining inorganic anions in wet deposition samples.

Conditions

Columns:	Dionex IonPac AS18 Analytical, 4 × 250 mm Dionex IonPac AG18 Guard, 4 × 50 mm
Eluent:	38 mM potassium hydroxide
Eluent Source:	Dionex ICS-2000 System with Dionex CR-ATC Column
Flow Rate:	1.0 mL/min
Temperature:	30 °C
Injection:	25 µL
Detection:	Suppressed conductivity, Dionex ASRS Ultra Suppressor, 4 mm, AutoSuppression™ recycle mode 100 mA current
Background Conductance:	~1.4 µS
System Backpressure:	~2500 psi
Run Time:	10 min

In this application update, the use of IC with a Dionex IonPac AS18 column and electrolytic eluent generation provides a rapid, isocratic analysis for the determination of inorganic anions in rainwater. The results obtained from the simulated rainwater samples were comparable to the values reported by High Purity Standards and were well within the certified standard deviation ranges. In addition, electrolytic generation of potassium hydroxide eliminates the need to manually prepare eluents, increasing the IC system's level of automation, ease of use, and analytical performance.



Analysis of simulated rainwater.



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Acid rain is a global problem due to its adverse impact on plants, aquatic animals, infrastructures, and human health. It is primarily produced by the reaction of water in the atmosphere with SO₂ and NO_x released from natural occurrences including volcanic eruptions and lightning strikes.

Some human activities, such as burning coal in power plants and exhaust from motor vehicles can also contribute to the contamination of SO₂ and NO_x in the air. Because of widespread deterioration of the environment caused by acid rain, many countries have regulations to enforce the reduction of SO₂ and NO_x released to the air. The monitoring of air and

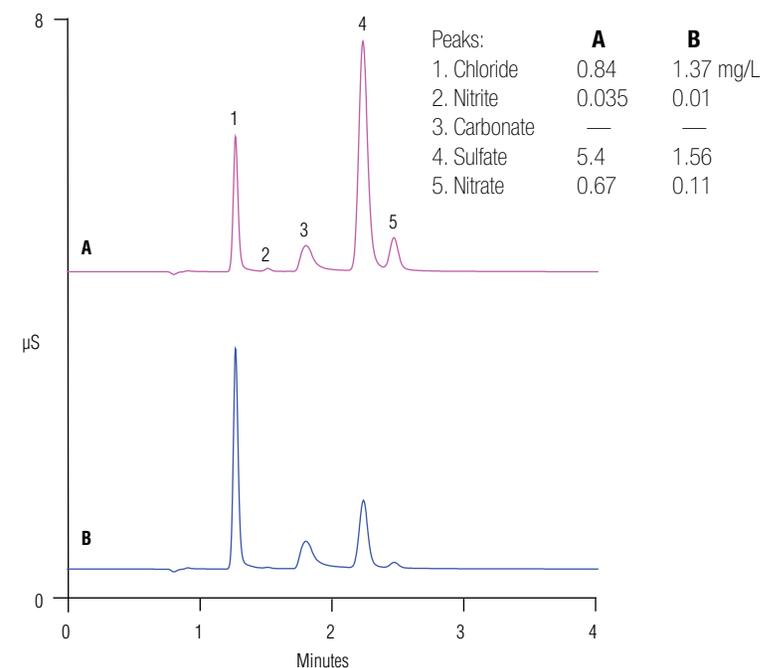
rain has become an important task worldwide for investigating the effects of pollutants on global ecology and assessing the progress of pollution abatement measures.

This technical note demonstrates that the high-pressure Dionex ICS-5000+ Capillary HPIC system provides a solution to high throughput sample analysis. The high-pressure capability facilitates fast analysis using higher flow rates. Combined with the benefits of Dionex IonPac AS18-4µm column, good resolution is achieved while the analysis time is shortened.

Conditions

Columns:	Dionex IonPac AG18-4µm, 0.4 × 50 mm Dionex IonPac AS18-4µm, 0.4 × 150 mm
Eluent Source:	Dionex EGC-KOH Cartridge (Capillary)
Eluent:	23 mM KOH
Flow Rate:	A: 0.010, 0.020, and 0.025 mL/min for standard solutions B: 0.025 mL/min for samples
Column Temperature:	30 °C*
Injection Volume:	0.4 µL
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ ACES™ 300 Anion Capillary Electrolytic Suppressor, recycle mode, 9 mA (0.015 mL/min); 10 mA (0.02 mL/min); 13 mA (0.025 mL/min)
Background Conductance:	1–2 µS
Noise:	2–3 nS
System Backpressure:	1600 psi (0.010 mL/min) 3100 psi (0.020 mL/min) 3800 psi (0.025 mL/min)

* The temperature of the capillary column is specified by setting the temperature of the Thermo Scientific Dionex IC Cube module because the capillary column is installed in the Dionex IC Cube module, not in the bottom column compartment, which used for analytical scale columns.



Determination of inorganic anions in rain samples using the Dionex IonPac AS18-4µm column.

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Cations

- Determination of Diethanolamine and Triethanolamine in Surface Finishings, Wastewater, and Scrubber Solutions
- Determination of Inorganic Cations and Ammonium in Environmental Waters by Ion Chromatography Using the Dionex IonPac CS16 Column
- Determination of Cations in Hydraulic Fracturing Flowback Water from the Marcellus Shale
- Fast Determinations of Inorganic Cations in Municipal Wastewater Using High-Pressure Capillary IC



Diethanolamine and Triethanolamine in Surface Finishing, Wastewater, and Scrubber Solutions

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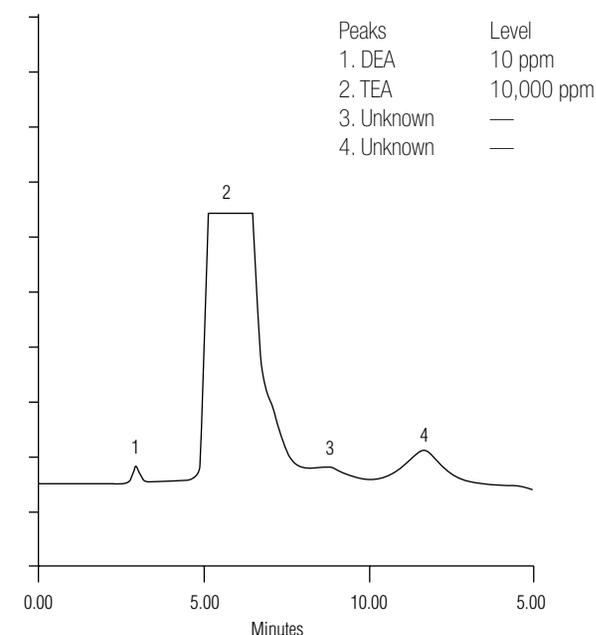
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Alkanolamines are important in the chemical and pharmaceutical industries for production of emulsifying agents and the manufacturing of laundry additives and dyes. The analysis of alkanolamines is also important in metal surface finishing and in wastewater effluents. They are commonly used in acid gas removal systems (scrubbers) in both oil refineries and natural gas plants. Monitoring soil and water samples in and around a refinery can identify sources of scrubber leaks before any serious losses or environmental contamination occurs.

Conditions

Column:	Dionex OmniPac PAX-500 Analytical and Guard								
Eluent 1:	300 mM sodium hydroxide								
Eluent 2:	10% v/v acetonitrile/water								
Eluent Proportion:	50% E1/50% E2								
Flow Rate:	1 mL/min								
Detector:	PED or PAD II, Gold working electrode Solvent compatible cell Ag/AgCl Reference electrode Range 30K nA <table border="0"><thead><tr><th>Potential</th><th>Time</th></tr></thead><tbody><tr><td>E1 = 0.08 V</td><td>T1 = 540 mS</td></tr><tr><td>E2 = 1.00 V</td><td>T2 = 420 mS</td></tr><tr><td>E3 = -0.08 V</td><td>T3 = 420 mS</td></tr></tbody></table>	Potential	Time	E1 = 0.08 V	T1 = 540 mS	E2 = 1.00 V	T2 = 420 mS	E3 = -0.08 V	T3 = 420 mS
Potential	Time								
E1 = 0.08 V	T1 = 540 mS								
E2 = 1.00 V	T2 = 420 mS								
E3 = -0.08 V	T3 = 420 mS								
Sample Loop:	50 µL								
Sample Preparation:	Dilute in 150 mM sodium hydroxide								

In this application update, determination of total alkanolamines is accomplished using liquid chromatography on Thermo Scientific™ Dionex™ OmniPac™ columns and Pulsed Amperometric Detection (PAD). The blended isocratic eluent is 150 mM sodium hydroxide solution containing 5% acetonitrile. The sodium hydroxide in the eluent maintains a high pH thus suppressing ionization of the alkanolamines. Under these conditions, the alkanolamines are retained and separated by a reversed-phase mechanism on the Dionex OmniPac PAX-500 analytical column.



Diethanolamine determination in an oil refinery triethanolamine scrubber solution.

Inorganic Cations and Ammonium in Environmental Waters



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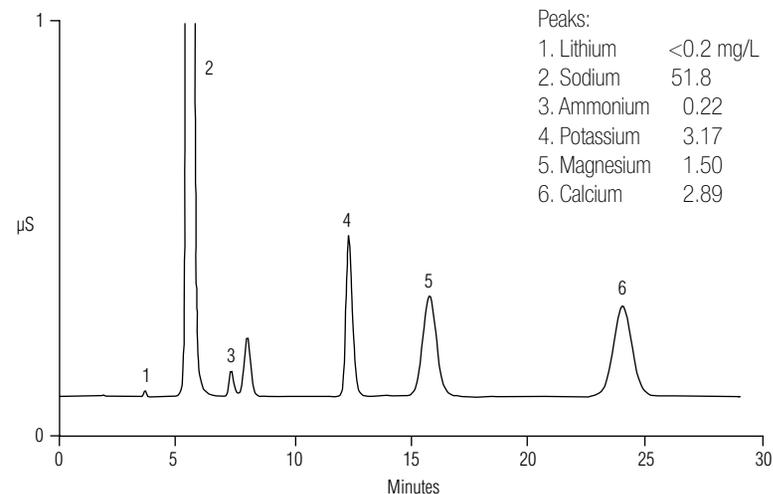
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Ammonia is a colorless, pungent gas. It is highly soluble in water, where it exists in equilibrium between a molecular form associated with water and the ionized form (the ammonium cation, NH_4^+). The extent of its toxicity to aquatic life depends upon the extent of dissociation, which in turn depends upon temperature and pH. Ammonia can enter environmental waters as a product of anaerobic decomposition of nitrogen-containing compounds or from waste streams containing ammonia. Ammonium cation is routinely measured in the U.S. for wastewater discharge compliance monitoring and in the EU and Japan in both wastewater and drinking water.

Conditions

Columns:	Dionex IonPac CS16 Analytical, 5 × 250 mm Dionex IonPac CG16 Guard, 5 × 50 mm
Eluent:	26 mM MSA
Eluent Source:	Thermo Scientific Dionex EG40 Eluent Generator
Flow Rate:	1.5 mL/min
Temperature:	30 °C
Injection:	10 µL
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ CSRS™ ULTRA Cation Self-Regenerating Suppressor (4 mm), AutoSuppression recycle mode, current setting 100 mA
Background:	<1 µS
Noise:	~0.2 nS peak-to-peak
Backpressure:	~2300 psi
Run Time:	30 min

In this application note, the Dionex IonPac CS16 column with a 26 mM MSA eluent and suppressed conductivity detection was used to determine inorganic cations and ammonium at concentrations ranging from 0.1–80 mg/L. The high capacity of the Dionex IonPac CS16 column enables the analysis of a wide range of environmental waters, and also resolves trace ammonium in the presence of a 10,000-fold higher concentration of sodium.



Determination of inorganic cations and ammonium in industrial wastewater with the Dionex IonPac CS16 column.

Cations in Hydraulic Fracturing Flowback Water



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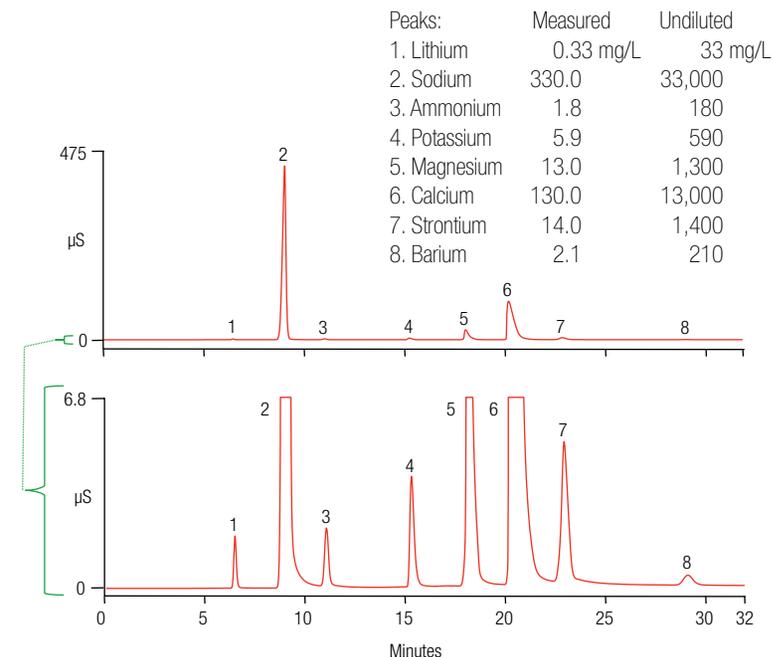
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Hydraulic fracturing requires large quantities of water because each well may be fractured multiple times and each fracturing event can require hundreds of thousands of gallons of fluid. This large consumption puts great stress on the local water resources, particularly in arid regions, making recycling an increasingly attractive option compared to disposing wastewater by pumping it into disposal wells. Cations such as calcium, barium, and strontium are especially important because they contribute to scaling problems in water pumps, pipes, etc. resulting in poor performance of recycled water for future fracturing events. Knowing the composition of anions and cations in flowback wastewater can also be used to adjust the treatment plan if surface water discharge is the final goal.

Conditions

Columns:	Dionex IonPac CG16 Guard, 5 × 50 mm Dionex IonPac CS16 Separation, 5 × 250 mm
Eluent Source:	Dionex EGC III MSA Cartridge
Gradient:	20–30 mM MSA (0–10 min), 30–55 mM MSA (10–18 min), 55 mM MSA (18–32 min), 20 mM MSA (32–38 min)
Flow Rate:	1 mL/min
Column Temperature:	40 °C
Injection Volume:	25 µL
Detection:	Suppressed conductivity, Dionex CERS Suppressor recycle mode, 161 mA
Background Conductance:	<0.2 µS
Noise:	<1 nS
System Backpressure:	2150 psi

This application note demonstrates that after diluting hydraulic fracturing flowback water, the concentration of cations can be accurately determined using the Dionex ICS-5000+ HPIC system with a capillary or standard bore Dionex IonPac CS16 column. The chromatographic conditions were optimized so that all the cations analyzed eluted with baseline resolution within 32 min. The most abundant analyte was sodium, followed by calcium, strontium, magnesium, potassium, barium, ammonium, and lithium. Wastewater from progressively later recovery volumes showed a gradual increase in cation concentrations indicative of more extensive association with the shale layer that was targeted for hydrocarbon extraction.



Determination of cations in fracturing flowback water (F4) using a standard bore column.

Inorganic Cations in Municipal Wastewater



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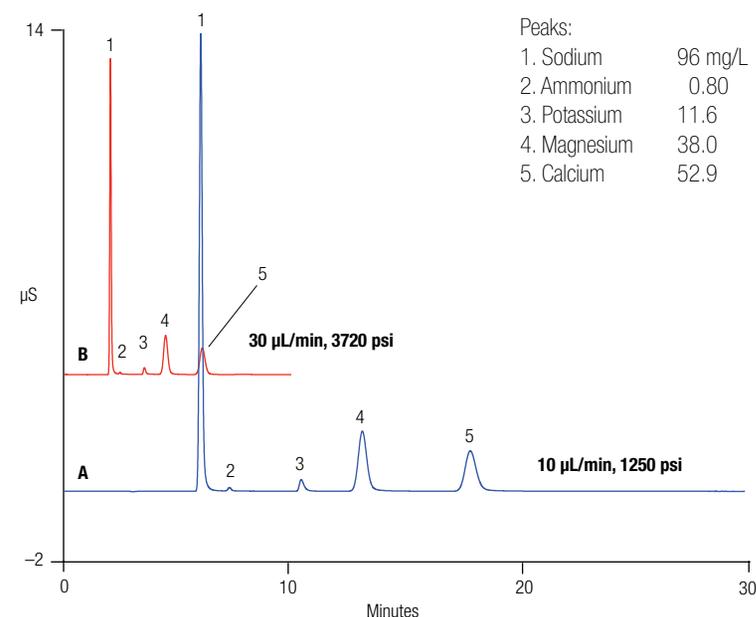
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Analysis of cations and anions are important to municipal drinking water and wastewater treatment plants for compliance monitoring. Cationic determinations are necessary as part of the water monitoring program's secondary water specification of acceptable taste. In municipal wastewater, cation determinations ensure that no environmental effects occur as a result of discharging high-salt concentrations into the water system.

Conditions	
Columns:	Dionex IonPac CS16, 0.5 × 250 mm
Eluent Source:	Dionex EGC-MSA Capillary Cartridge with Thermo Scientific Dionex CR-CTC Continuously Regenerated Cation Trap Column
Eluent:	30 mM MSA
Flow Rate:	A: 10 μ L /min B: 30 μ L /min
IC Cube Temperature:*	40 °C
Compartment Temperature:	15 °C
Injection Volume:	0.4 μ L
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 μ S conductance
Noise:	<0.3 nS
System Backpressure:	A: 1250 psi; B: 3720 psi

* The Thermo Scientific Dionex IC Cube module 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.

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.



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

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Perchlorate

- Determination of Low Concentrations of Perchlorate in Drinking and Groundwaters Using Ion Chromatography
- Determination of Perchlorate in Environmental Waters by Ion Chromatography Coupled with Electrospray Mass Spectrometry (IC-MS)
- Improved Determination of Trace Perchlorate in Drinking Water Using 2D-IC



Low Concentrations of Perchlorate in Drinking and Groundwaters

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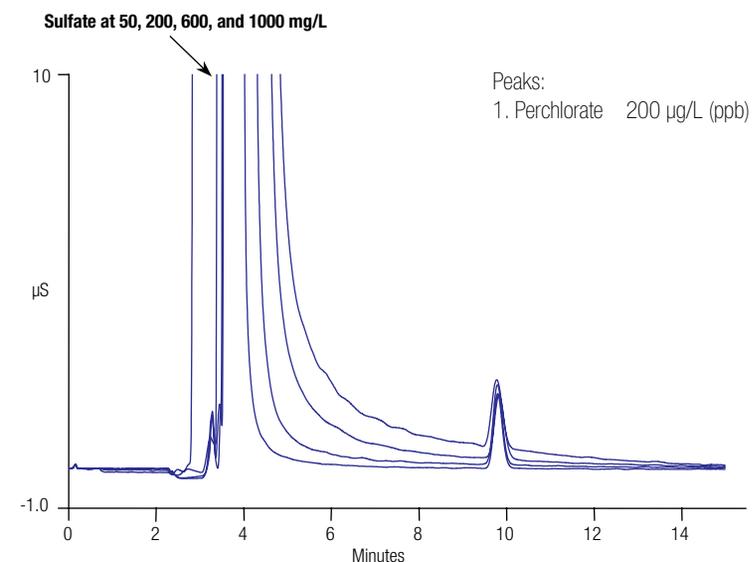
Ammonium perchlorate is a key ingredient in solid rocket propellants. Perchlorate has recently been found in drinking water wells in regions of the U.S. where aerospace material, munitions, or fireworks were developed, tested, or manufactured. Perchlorate poses a human health concern because it can interfere with the thyroid gland's ability to utilize iodine to produce thyroid hormones. Current data suggest that 4 to 18 $\mu\text{g/L}$ (ppb) is an acceptable exposure level. The determination of perchlorate at trace levels is a difficult analytical task and IC represents the only viable means for the quantification of such low concentrations of perchlorate.

In this application note, the method described can be used to determine low $\mu\text{g/L}$ concentrations of perchlorate in drinking and groundwaters. The use of IC with the Dionex IonPac AS5 or Dionex IonPac AS11 columns has

Conditions

Columns:	Dionex IonPac AS16 Analytical, 4 × 250 mm Dionex IonPac AG16 Guard, 4 × 250 mm
Eluent:	65 mM potassium hydroxide
Eluent Source:	Dionex EG40 eluent generator
Flow Rate:	1.2 mL/min
Temperature:	30 °C
Sample Volume:	1000 μL
Detection:	Suppressed conductivity, Dionex ASRS ULTRA Suppressor, AutoSuppression, external water mode; Power setting – 300 mA
System Backpressure:	2600 psi
Background Conductance:	1–4 μS
Run Time:	12 min

previously been shown to provide an interference-free method for the analysis of perchlorate in modest-ionic-strength drinking water and groundwater samples; the Dionex IonPac AS16 column yields improved results. The Dionex IonPac AS16 column is compatible with the Dionex EG40 eluent generator, and its higher capacity makes it most appropriate for the analysis of perchlorate in higher ionic-strength samples. This application can also be run using a Dionex IonPac AS11 column as described in Application Note 121: Analysis of Low Concentrations of Perchlorate in Drinking Water and Groundwater by Ion Chromatography.



Effect of sulfate on perchlorate recovery on the Dionex IonPac AS16 column.

[Download Application Note 134: Determination of Low Concentrations of Perchlorate in Drinking and Groundwaters Using Ion Chromatography](#)

[Download Application Note 121: Analysis of Low Concentrations of Perchlorate in Drinking Water and Groundwater by Ion Chromatography](#)



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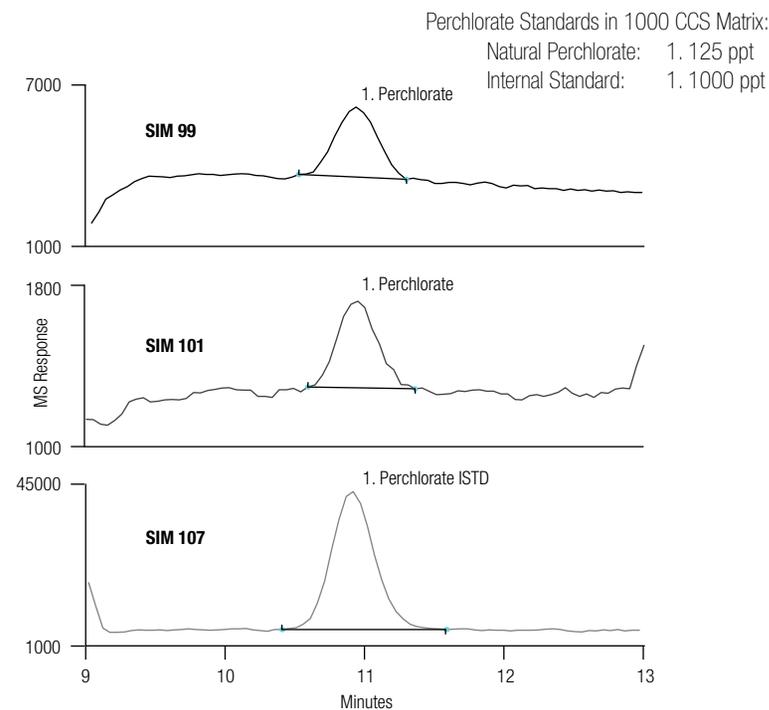
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Perchlorate has been used as an oxidizer in rockets, munitions, and fireworks since the 1950s, and has been found to cause thyroid dysfunction in humans. The California Department of Health Services first reported the determination of perchlorate in drinking water in 1997. The U.S. EPA has stated that defense facilities from Los Angeles to Cape Cod have discharged large amounts of perchlorate onto the ground, contaminating groundwater in many places. In 2001, it was reported that perchlorate (ClO_4^-) was migrating into groundwater in California from a landfill site for civilian and military explosives. In 2002, the U.S. EPA recommended a maximum containment level (MCL) for perchlorate of 1 ppb in drinking water.

Conditions

Column:	Dionex IonPac AS16, 250 × 2 mm i.d.
Suppressor:	Dionex ASRS ULTRA II Suppressor, 2 mm, external water, 70 mA
GS50 Eluent:	45 mM KOH
AXP-MS Eluent:	50/50 v/v acetonitrile/water
Flow Rate:	0.3 mL/min, GS50 Gradient Pump and Dionex AXP-MS Auxiliary Pump
Temperature:	28 °C
Matrix Diversion Time:	2–9 min
Injection Volume:	100 μL
Detection:	1. Suppressed Conductivity 2. MS
MS Conditions:	Mode, –ESI Cone voltage, 70 V Probe voltage, –3 kV Probe temp., 450 °C SIM channels, 99, 101, and 107 m/z SIM parameters, span 0.3 amu, dwell time 1 s
Run Time:	13 min
Expected System Backpressure:	2100–2350 psi
Expected Background Conductance:	<1.5 μS

In this application note, the use of IC-MS to determine perchlorate in environmental waters is demonstrated. The method uses a 2 mm column format, a stable-labeled internal standard, a matrix diversion valve to eliminate the need for offline sample pre-treatment, and a mass spectrometer with an electrospray interface as the detector. The mass spectrometer is a more selective detector than conductivity in that it monitors the mass/charge ratio (m/z) of the analyte. The m/z ratio provides peak identification information for perchlorate at both 99 and 101 m/z due to the relative isotopic abundance of ^{35}Cl and ^{37}Cl .



High ionic matrix spiked with 125 ppt perchlorate and internal standard.



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Perchlorate is identified as an environmental contaminant found in drinking, ground, and surface waters. Perchlorate contamination is attributed to the manufacture and use of ammonium perchlorate in solid propellant for rockets, missiles, fireworks, and elsewhere (e.g., production of matches, flares, pyrotechnics, ordnance, and explosives). Research on perchlorate in the environment has received attention because perchlorate poses a human health concern.

This application note demonstrates a 2D-IC system for determining trace concentrations of perchlorate in drinking waters. This method was developed based on AN 178 and EPA Method 314.2. Improvements were made by using a hybrid system in which perchlorate was resolved from the matrix on a 2 mm Dionex IonPac AS20 column, concentrated on a monolithic capillary concentrator, separated on a 0.4 mm Dionex IonPac AS16 column, and detected by suppressed conductivity detection.

Conditions

First Dimension

Columns:	Dionex IonPac AG20 Guard, 2 × 50 mm Dionex IonPac AS20 Analytical, 2 × 250 mm
Eluent Source:	Dionex EGC III KOH Eluent Generation Cartridge with Dionex CR-ATC Continuously Regenerated Anion Trap Column
Eluent:	35 mM KOH 0–30 min, step to 60 mM at 30.1 min, 60 mM 30.1–40 min, step to 35 mM at 40.1 min, 35 mM 40.1–45 min
Flow Rate:	0.25 mL/min
Injection Volume:	500 µL
Temperature:	15 °C (upper compartment) 30 °C (lower compartment)
Detection:	Suppressed conductivity, Dionex ASRS 300 suppressor, 2 mm, 38 mA, external water mode System
Backpressure:	~2185 psi
Background Conductance:	~0.500 µS
Noise:	~0.3 nS/min peak-to-peak
Run Time:	45 min

Conditions

Second Dimension

Columns:	Dionex IonPac AG16 Capillary Guard, 0.4 × 50 mm Dionex IonPac AS16 Capillary Analytical, 0.4 × 250 mm
Eluent Source:	Dionex EGC-KOH (Capillary) Cartridge with Dionex CR-ATC Continuously Regenerated Anion Trap Column (Capillary)
Eluent:	65 mM KOH
Flow Rate:	0.01 mL/min
Injection Volume:	1 mL (on the concentrator column from first dimension)
Temperature:	15 °C (upper compartment) 30 °C (Dionex IC Cube Cartridge)
Detection:	Suppressed conductivity, Dionex CES 300 suppressor, 12 mA, external water mode System
Backpressure:	~1230 psi
Background Conductance:	~0.400 µS
Noise:	~0.5 nS/min peak-to-peak
Run Time:	45 min





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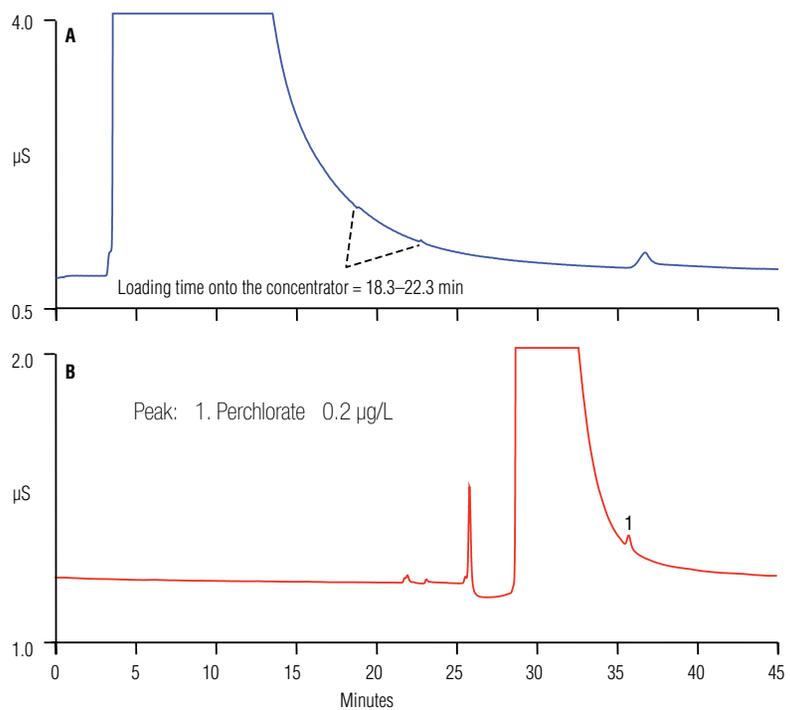
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Chromatograms of synthetic high inorganic water containing 1000 mg/L each of chloride, sulfate, and bicarbonate fortified with 0.2 µg/L perchlorate in (A) first dimension and (B) second dimension.



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Hexavalent Chromium

- Sensitive Determination of Hexavalent Chromium in Drinking Water
- Separation of Chromium (III) and Chromium (VI) by Ion Chromatography

Sensitive Determination of Hexavalent Chromium in Drinking Water



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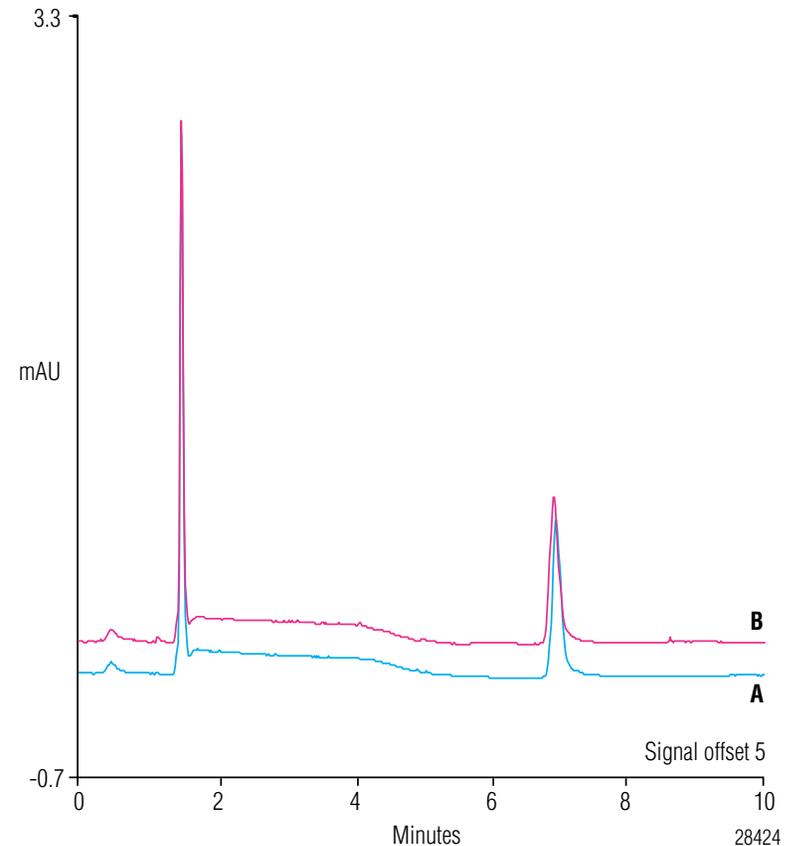
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Chromates are oxyanions (e.g., CrO_4^{2-} , $\text{Cr}_2\text{O}_7^{2-}$) of chromium in oxidation state +6. All hexavalent chromium Cr(VI) compounds are strong oxidizing agents and considered toxic and potentially carcinogenic. Hence, chromates are regulated in the environment and are a primary drinking water contaminant in the United States.

Conditions

Columns:	Dionex IonPac AG7 Guard, 2 × 50 mm Dionex IonPac AS7 Analytical, 2 × 250 mm
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
Backpressure:	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

The work shown here describes modification of the conditions described in EPA Method 218.6, including use of the column in the 2 mm format and a smaller reaction coil to increase method sensitivity.



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).

Separation of Chromium (III) and Chromium (VI) by Ion Chromatography

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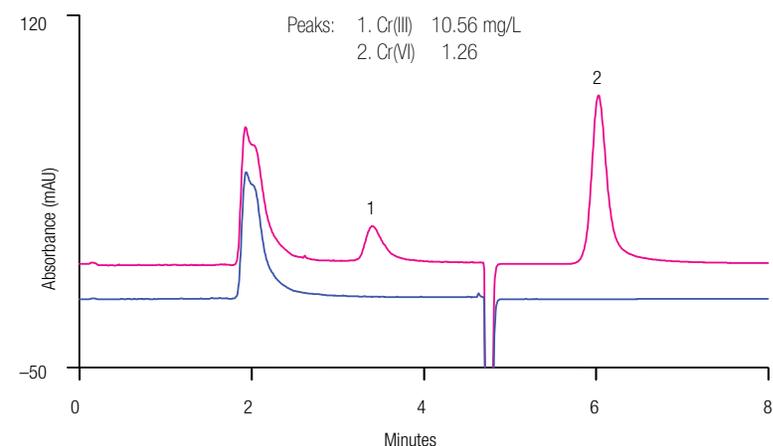
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Chromium in the environment exists primarily in two oxidation states: Cr(III) and Cr(VI). While the trivalent Cr(III) is only toxic at high concentrations, hexavalent Cr(VI), a strong oxidizer, is considered toxic to humans and the environment at $\mu\text{g/L}$ concentrations. Because of this toxicity, many countries and states strongly regulate the concentration of Cr(VI) in drinking and wastewaters and require that its concentration is measured and reported.

Conditions

Guard Column:	Dionex IonPac CG5A, 4 × 50 mm
Column:	Dionex IonPac CS5A, 4 × 250 mm
Eluent:	2 mM PDCA, 2 mM Na_2HPO_4 , 10 mM NaI, 50 mM $\text{CH}_3\text{CO}_2\text{NH}_4$, 2.8 mM LiOH
Flow Rate:	1.0 mL/min
Injection Volume:	50 μL
Expected System Pressure:	1500 psi
Detector Wavelength:	0.0–4.7 min, 335 nm; 4.7–8.0 min, 530 nm
Postcolumn Reagent:	2 mM DPC, 10% CH_3OH , 0.9 N H_2SO_4
Postcolumn Flow Rate:	0.5 mL/min*
Reaction Coil:	375 μL

This application update demonstrates separation and detection of Cr(III) and Cr(VI) using a Dionex IonPac CS5A column and absorbance detection. This method also describes a sample preparation technique that can be used to estimate the concentrations of Cr(III) and Cr(VI) in soil and wastewater samples.



Overlay of the chromatograms of the wastewater sample and the sampled spiked with 10 mg/L Cr(III) and 0.5 mg/L Cr(VI).

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Cyanide

- Determination of Total Cyanide in Municipal Wastewater and Drinking Water Using Ion-Exclusion Chromatography with Pulsed Amperometric Detection (ICE-PAD)
- Direct Determination of Metal Cyanides by Ion Chromatography with UV Absorbance Detection
- Determination of Metal Cyanide Complexes by Ion Chromatography with Online Sample Preconcentration and UV Absorbance Detection
- Direct Determination of Cyanide in Strongly Alkaline Solutions



Total Cyanide in Municipal Wastewater and Drinking Water

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Cyanide is a well known acute toxin that prevents cellular respiration by irreversibly binding with the iron in cytochrome C oxidase. In addition, thiocyanate, which is metabolized from cyanide, interferes with iodine uptake by the thyroid gland, causing goiters and other long-term iodine deficiency diseases. Cyanide is regulated as an environmental contaminant by the U.S. EPA for drinking water, surface water, and wastewater due to these health concerns.

In this application note, an ion-exclusion chromatography with pulsed amperometric detection (ICE-PAD) method is used as the determinative method for the U.S. EPA-approved Lachat MICRO DIST acid digestion system for preparing samples for determining µg/L concentrations of total

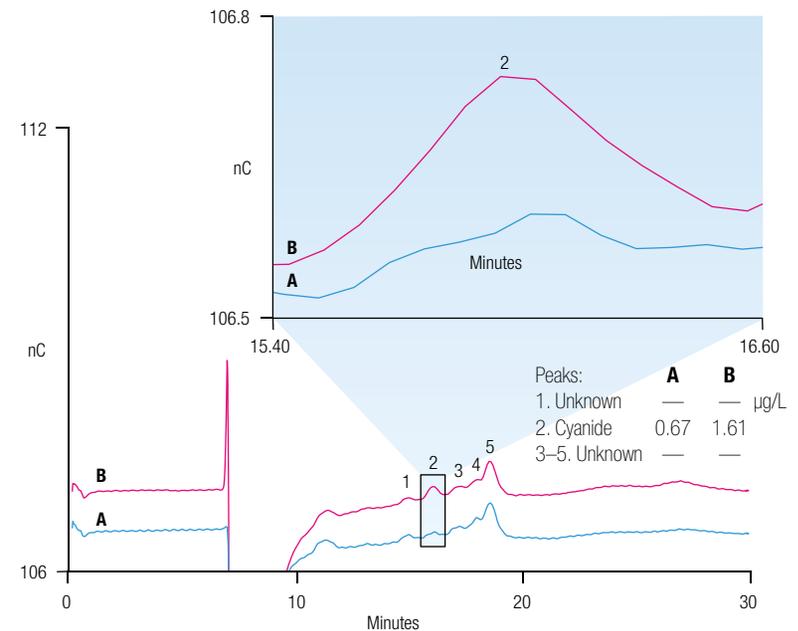
cyanide in municipal drinking water and municipal wastewater effluent. The method provides low detection limits and improvement of cyanide recoveries due to exclusion of chloride and resolution from sulfide.

Conditions

Columns:	Dionex IonPac ICE-AG1 Guard, 4 × 50 mm Dionex IonPac ICE-AS1 Analytical, 4 × 250 mm
Flow Rate:	0.2 mL/min
Eluent:	50 mM methanesulfonic acid
Column Temperature:	30 °C
Tray Temperature:	10 °C
Injection Volume:	50 µL
Detection:	PAD
Waveform:	See Table
Reference Electrode:	pH-Ag/AgCl electrode 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

Time (sec)	Potential vs. Ag/AgCl (V)	Gain Region ^a	Integration	Ramp ^a
0.00	+0.30	Off	Off	Ramp
0.31	+0.30	On	Off	Ramp
0.32	+1.15	On	Off	Ramp
0.64	+1.15	On	On (Start)	Ramp
0.66	+1.15	On	Off (End)	Ramp
0.67	-0.30	On	Off	Ramp
1.06	-0.30	Off	Off	Ramp
1.07	+0.30	Off	Off	Ramp

^aThe gain and ramp are instrument settings for the Dionex ICS-3000 IC electrochemical detector.



Comparison of A) municipal drinking water and B) sample A with 1 µg/L cyanide added.



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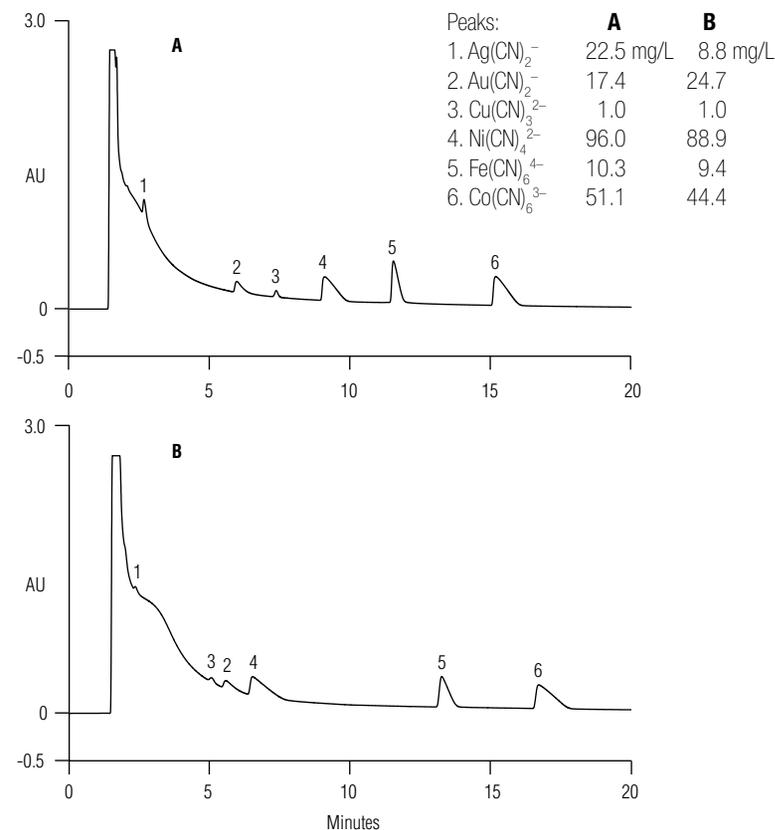
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Metal cyanide complexes are of environmental concern because they release cyanide upon dissociation. In environmental waters below pH 9.3, the cyanide ion converts to HCN, an extremely toxic substance. Metal cyanide complexes are also of interest in the mining and reclamation of precious metals, and in the metal finishing industry.

Conditions

Columns:	Dionex IonPac AS11 Analytical 2 × 250 mm Dionex IonPac AG11 Guard 2 × 50 mm Dionex IonPac ATC-3 (2 each)			
Temperature:	30 °C			
Injection:	25 µL			
Detection:	Absorbance at 215 nm			
Expected System Backpressure:	900 psi			
Noise:	1–5 mAU			
Run Time:	35 min			
Flow Rate:	0.25 mL/min			
Eluent A:	20 mM NaOH/150 mM NaCN			
Eluent B:	20 mM NaOH/300 mM NaClO ₄			
Eluent C:	20 mM NaOH			
Pump Program:	Time (min)	%A	%B	%C
	Init.	10	10	80
	0.0	10	10	80
	18.0	10	45	45
	22.0	10	45	45
	25.0	10	10	80
	35.0	10	10	80

This application update presents a thorough comparison of the performance expected from the different column sets, with data obtained during participation in a joint ASTM/EPA inter laboratory collaborative study to validate the use of IC for the determination of metal cyanide complexes in environmental waters. The method was evaluated for reproducibility, linearity, accuracy, precision, and spike recovery from various matrices.



Determination of metal cyanide complexes in wastewater from a former manufactured gas plant.



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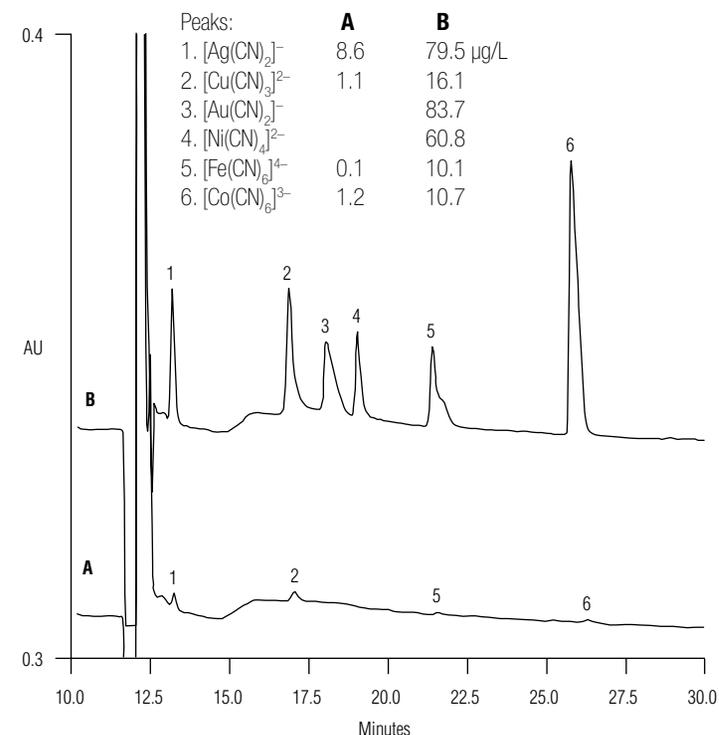
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Metal cyanides are negatively charged ionic complexes represented by the general formula $[M(CN)_6]^{x-}$, where several cyanide ions are bound to a single transition metal cation such as Ag^+ , Au^+ , or Fe^{2+} . Metal cyanides can dissociate to release highly toxic HCN into the environment. The toxicity of metal cyanides varies widely from one species to another. Weak metal cyanides that readily dissociate, such as $[Ag(CN)_2]^-$, pose a significant threat to health, whereas strong metal cyanide complexes that dissociate only under strongly acidic conditions, such as $[Fe(CN)_6]^{4-}$, pose a lesser risk.

Conditions

Columns:	Dionex IonPac AS11 Analytical, 2 × 250 mm Dionex IonPac AG11 Guard, 2 × 50 mm, 2 each Dionex IonPac ATC-3
Temperature:	30 °C
Injection:	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
Eluent B:	20 mM sodium hydroxide/300 mM sodium perchlorate
Eluent C:	20 mM sodium hydroxide

In this application note, the method of online preconcentration allows determination of metal cyanide complexes at $\mu\text{g/L}$ 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.



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).

Cyanide in Strongly Alkaline Solutions



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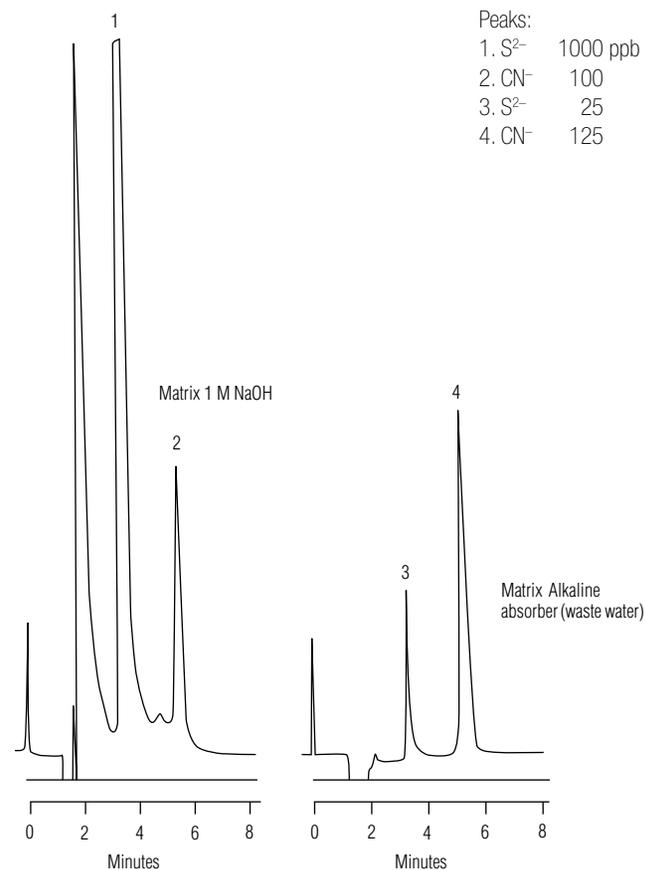
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Determination of total cyanide in water is usually done by refluxing the sample in an acid digest and trapping the liberated HCN gas in a strongly alkaline absorbing solution. Most methods for analyzing the trapping solution have an upper pH limit of about 12.5 to 13. This direct injection method can determine cyanide in solutions ranging up to pH 14. This allows absorbing solutions (such as the 1.25 M NaOH solution specified in U.S. EPA Method 335.2) to be quickly analyzed without dilution or other pretreatment. In addition, the technique is not subject to as many interferences as titrimetric or spectrophotometric methods, and it is well suited to automated analysis.

Conditions

Column:	Dionex IonPac AS7
Eluent:	0.5 M Sodium acetate 0.1 M Sodium hydroxide 0.5% (v/v) Ethylenediamine
Flow Rate:	1 mL/min
Detector:	Dionex ED40 detector, silver working electrode, 0.00 V vs Ag/AgCl reference



Cyanide in strongly alkaline solutions.

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Organic Acids

- Determination of Organic Acids in Wastewater Using Ion-Exclusion Chromatography and Online Carbonate Removal
- Direct Determination of Small Organic Acids in Seawater by IC-MS



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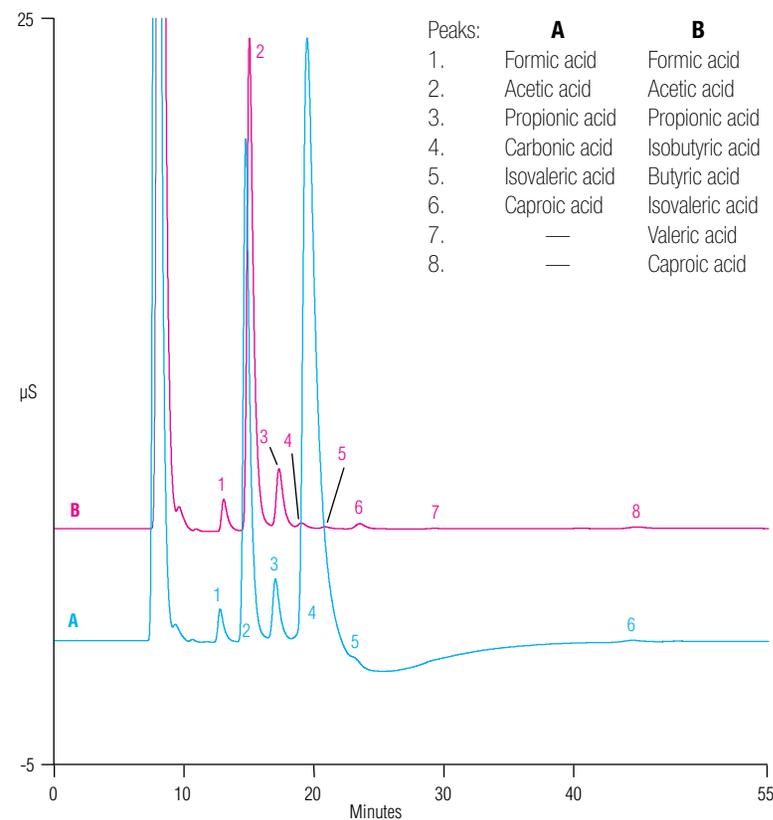
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Low-molecular mass carboxylic acids are important intermediates and metabolites in biological processes. These carboxylic acids are known as volatile fatty acids (VFAs) or short-chain fatty acids (SCFAs). The presence of VFAs in a sample matrix is often indicative of bacterial activity because they can originate from anaerobic biodegradation of organic matter. Therefore, VFAs are widely present in activated sludge, landfill leachates, and wastewater.

Conditions

Column:	Dionex IonPac ICE-AS1 Analytical, 4 × 250 mm Dionex IonPac ICE-AS1 Guard, 4 × 50 mm
Eluent:	0.5 mM HFBA
Flow Rate:	0.16 mL/min
Pressure:	750 psi
Sample Volume:	25 µL
Column Temperature:	30 °C
Sample Tray Temperature:	10 °C
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ AMMS™ ICE 300 Suppressor, 4 mm, Dionex CRD 200 Device, 2 mm, and 5 mM TBAOH regenerant delivered by pressurized bottle using N ₂ gas
Total Conductivity:	~41 µS

This application demonstrates the determination of organic acids in a wastewater sample using ion-exclusion chromatography and online carbonate removal. Ion-exclusion chromatography excludes the inorganic anions in the sample and the Thermo Scientific Dionex CRD 200 Carbonate Removal Device removes carbonate online. The results show that this combination allows the accurate determination of organic acids in a wastewater sample.



Overlay of chromatograms of spiked wastewater with and without a Dionex CRD 200 device installed.



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Studies show that low molecular weight species in aquatic environments are important sources of carbon for marine bacteria. Short-chain organic acids in the marine environment play important roles in adjusting the pH value, forming a variety of complexes and then increasing the solubility of trace metals in seawater. In light of the significant growth in aquaculture for farmed fish and shellfish, several feed additives—including acidifiers consisting of organic acids and their salts—may provide promising alternatives to the use of in-feed antibiotics in aquaculture.

In this application note, the analysis for small organic acids, using a 2D-IC system, allows low levels of target analytes to be detected in a high-background matrix. The Thermo Scientific™ MSQ Plus™ Mass Spectrometer provides additional selectivity and sensitivity through use of the SIM function to achieve low detection limits. No special sample preparation was required and the results show good precision and reproducibility.

Chromatographic Conditions

- Columns:
- First Dimension
- Dionex IonPac AG24 Guard Column (2 × 50 mm)
 - Dionex IonPac AS24 Analytical Column (2 × 250 mm)
- Second Dimension
- Dionex IonPac AG11 Guard Column (2 × 50 mm)
 - Dionex IonPac AS11-HC Analytical Column (2 × 250 mm)
 - Dionex IonPac UTAC-ULP1 Ultra Trace Anion Concentrator Ultralow Pressure (5 × 23 mm)

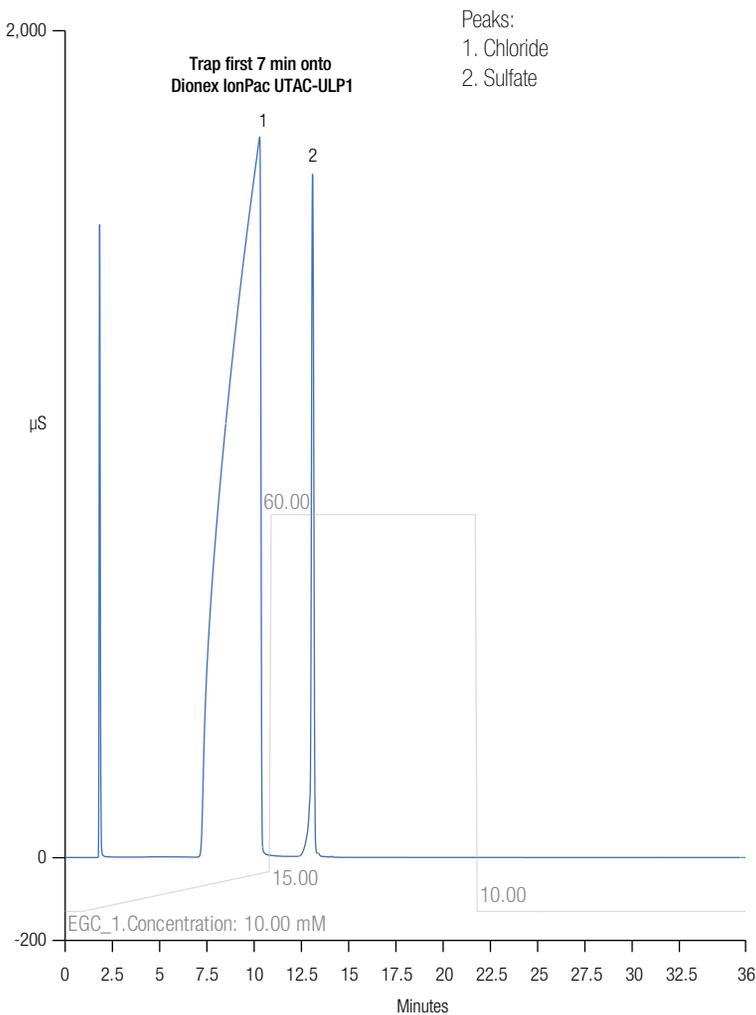
Mobile Phase:	Two Electrolytically	Generated KOH Gradients
Gradient 1:	Time (min)	Conc (mM)
	-5.0	10
	0.1	10
	10.0	15
	10.1	60
	20.9	60
	21.0	10
	33.1	10
Gradient 2:	Time (min)	Conc (mM)
	-5.0	1
	13.0	1
	23.0	4
	28.0	60
	33.0	60
	33.1	1
Flow Rate:	0.4 mL/min for both 1st and 2nd dimensions	
Injection Volume:	25 µL	
Temperature:	35 °C	
Detection:	Suppressed conductivity: Dionex ASRS 300 Suppressor, 2 mm, external water mode (2 mL/min)	
Suppressor 1:	Time (min)	Current mA
	-5.0	15
	10.0	15
	10.1	60
	28.0	15
Suppressor 2:	Time (min)	Current mA
	-5.0	2
	23.0	10
	28.0	60
	36.0	4



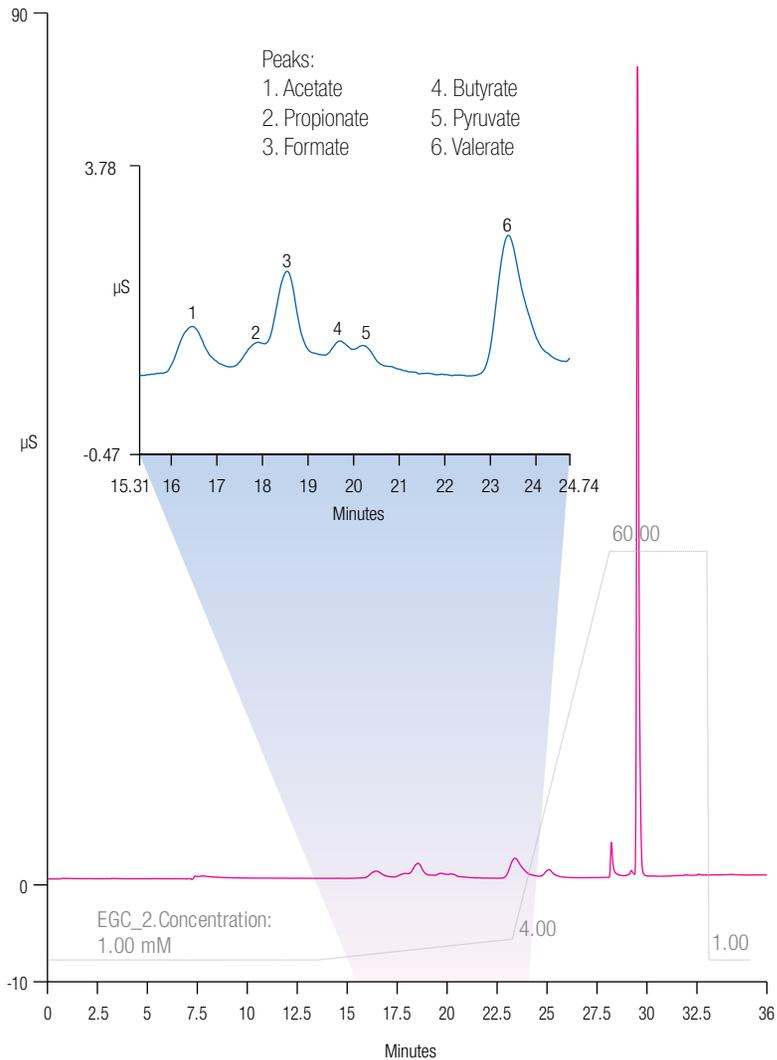


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First dimension (Dionex IonPac AS24 column).



Second dimension (Dionex IonPac AS11-HC column).



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Herbicides

- Sensitive and Fast Determination of Endothall in Water Samples by IC-MS/MS



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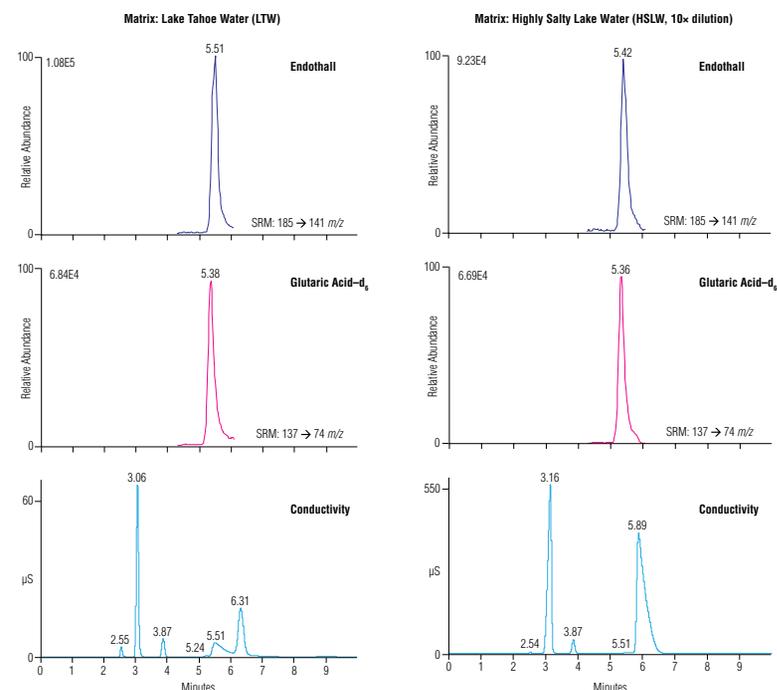
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Endothall is a widely used herbicide for both terrestrial and aquatic weeds. Major uses of endothall include defoliation of cotton, the control of aquatic weeds and algae, and as a desiccating agent for alfalfa and potatoes. Human exposure to endothall in excess of the maximum contamination level (MCL) may cause gastrointestinal problems. Endothall is regulated by the U.S. EPA with an MCL at 0.1 mg/L or 100 ppb for drinking water; and the California EPA developed the Public Health Goal (PHG) of 0.58 mg/L for this compound.

Conditions

Columns:	Dionex IonPac AG16/AS16 hydroxide selective anion-exchange set (2 mm)	
Eluent Source:	Dionex EGC II KOH Cartridge with Dionex CR-ATC Column, 2 mm	
Eluent:	Hydroxide gradient	
	Min	Conc
	-4.0	15 mM
	0.0	15
	5.0	15
	6.0	80
	9.0	80
	9.5	15
	10.0	5
Flow Rate:	400 μ L/min	
Temperature:	30 $^{\circ}$ C	
Solvent:	200 μ L/min acetonitrile delivered by a Dionex AXP-MS Pump	
Detector 1:	Suppressed Conductivity with Dionex ASRS 300 Suppressor, 2 mm (external water at 0.5 mL/min delivered by a Dionex AXP-MS Pump)	
Detector 2:	Thermo Scientific™ TSQ Quantum Access™ Mass Spectrometer	

A fast and sensitive IC-MS/MS method was presented for the quantitative determination of trace level endothall in environmental water samples. The detection limit was estimated at 0.56 ppb, and linear response was observed from 1 ppb to 1000 ppb. The significantly improved sensitivity enables direct analysis of water samples without labor-intensive sample enrichment and derivatization. The total chromatographic run time was halved from the 20 min GC run in U.S. EPA Method 548.1.



Conductivity and SRM chromatograms of endothall spiked in different samples: left, 5 ppb endothall in salty lake water with 10-fold dilution.

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Metals

- Determination of Chelating Agents in Drinking Water and Wastewater Samples

Chelating Agents in Drinking Water and Wastewater Samples



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Aminopolycarboxylate chelating agents are used extensively in many domestic products and industrial processes, with the most important applications in cleaning compounds, pulp and paper manufacturing, and agriculture. Chelating agents form stable water-soluble complexes with alkali and transition metal ions, thus increasing metal solubility and preventing metal-catalyzed reactions. Therefore, chelating agents prevent metals from interfering with the detergent's ability to remove soils and stains from clothing, from degrading oxidizing and bleaching agents in paper and textile manufacturing, and from precipitating in fertilizers. Chelating agents are also used in soil remediation to remove heavy metal contamination.

This application note describes a direct, sensitive, and accurate method to determine $\mu\text{g/L}$ concentrations of NTA, EDTA, EGTA, and DTPA in surface water, municipal drinking water, and wastewater samples. This method takes advantage of the selectivity of the Dionex IonPac AS7 column to separate large hydrophobic anionic compounds, such as chelating agents, and the selectivity of PAD to determine low concentrations of these compounds without detecting common anions that are typically present at high concentrations in wastewater samples.

Conditions

Column:	Dionex IonPac AG7 Guard, 2 × 50 mm Dionex IonPac AS7 Analytical, 2 × 250 mm
Eluent:	A: Degassed deionized water B: 200 mM Methanesulfonic acid (MSA)
Gradient:	17.5% B (35 mM MSA) from -5 to 1 min, step to 50% B (100 mM MSA) at 1 min, 50% B from 1 to 12 min, step to 17.5% B at 12 min, 17.5% B from 12 to 16 min
Flow Rate:	0.3 mL/min
Trap Column:	Dionex IonPac CTC-1, 9 × 24 mm
Temperature:	30 °C
Injection Volume:	25 μL
Detection:	PAD, Pt disposable WE, waveform (see Table)
Data Collection Rate:	0.9 Hz
Typical Background:	130–180 nC
Typical Noise:	60–80 pC
Typical pH:	0.9–1.1
Run Time:	16 min



Chelating Agents in Drinking Water and Wastewater Samples



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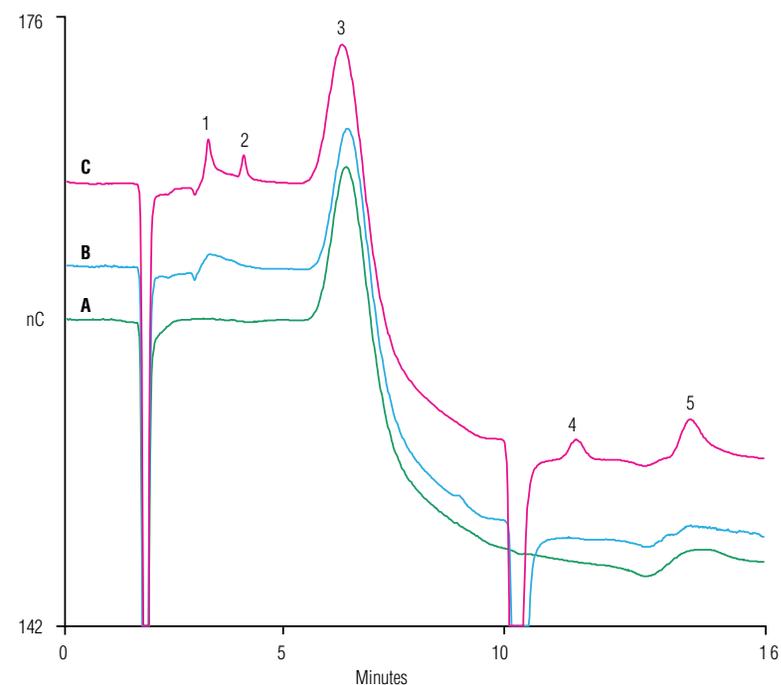
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Time (sec)	Potential vs Ag/AgCl (V)	Gain Region ^a	Integration	Ramp ^a
0.00	+0.30	Off	Off	Ramp
0.31	+0.30	Off	Off	Ramp
0.32	+1.15	Off	Off	Ramp
0.64	+1.15	On	On (Start)	Ramp
0.66	+1.15	On	Off (End)	Ramp
0.67	-0.30	Off	Off	Ramp
1.06	-0.30	Off	Off	Ramp
1.07	+0.30	Off	Off	Ramp

^a The gain and ramp are instrument settings for the Dionex ICS-3000 and Dionex ICS-5000 electrochemical detector.

Peaks:	B	C
1. EDTA	0.024	0.203 mg/L
2. NTA	—	0.210
3. System	—	—
4. DTPA	—	0.259
5. EGTA	0.18	0.698



Comparison of 1:10 dilution of City A municipal wastewater effluent B) without and C) with chelates added.



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Innovative Solutions

- Sample Preparation Solutions
- Matrix Elimination
- Ion Analysis

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Sample Preparation Solutions

The separation and detection of trace-level ionic contaminants in various environmental matrices can be compromised if there are interfering compounds. Overcoming interference effects requires effective sample preparation solutions to accurately determine the contaminant of interest using ion chromatography.

Inline Filtration

Filtration removes particulate contamination from samples prior to injection onto an ion chromatograph which will also preserve the lifetime and performance of IC columns. When performing filtration, the unpredictable clogging of filters and sample carry over can be problematic. Inline filtration alleviates these issues by automating the filtration process.

Both the Thermo Scientific Dionex AS-DV Autosampler and the use of inline high-pressure filters are options to perform inline filtration. The Dionex AS-DV Autosampler is a metal-free, automated sample loading device that handles various types of samples including ones with a high particulate count. A sample is dispensed into an inert sample vial and a plunger cap with an integral filter is installed in the top of the vial (caps without filters are available for samples not requiring filtration). Larger particles are deposited in the bottom of a tube as filtration occurs top down, assisted by



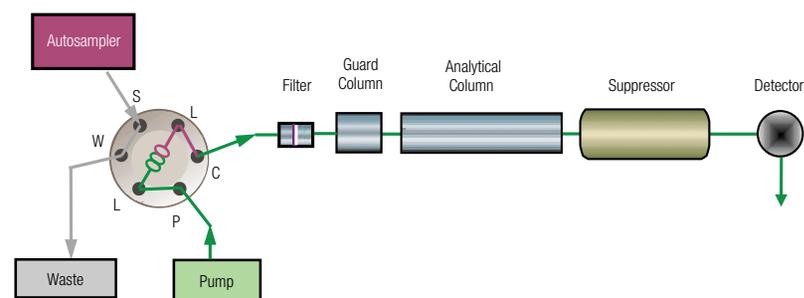
Dionex AS-DV Autosampler sample displacement filtration.

gravity. The filter cap has a large capacity due to its large cross-section and volume. Sample carry over is eliminated as each sample has its own filter.

Inline high-pressure filters are placed in the high-pressure flow path of the ion chromatograph, after the injection valve. This allows particulates to be removed from the sample. In addition, the guard column does not clog due to particulate accumulation in the inlet frit of the column. Inline high pressure filters should not be used with samples that are highly contaminated.

Sample carry over is eliminated as the filter is exposed to the flow of eluent. An additional benefit is the ability to monitor filter condition by monitoring the pressure of the ion chromatography system. All Thermo Scientific Dionex IC systems are capable of monitoring system pressure.

Inline high-pressure filtration schematic.



Inline high-pressure filter assembly.



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AutoDilution

Environmental samples can have high analyte concentrations that necessitate loading less sample volume or diluting prior to injection to avoid exceeding column capacity and ensure that the concentrations determined fall within the linear calibration range. The latter is particularly important when regulated methods are used, which typically prescribe a concentration range for each analyte.

Using the AutoDilution feature of Chromeleon CDS software, the need for sample dilution can be easily automated. Samples are run undiluted and then, based on the peak height or area of analytes in the resultant chromatogram, the amount of sample injected can be reduced before re-analysis using:

- A partial loop injection
- A smaller volume injection loop
- Dilution by the autosampler

Dionex AS-AP Autosampler

The Thermo Scientific Dionex AS-AP Autosampler provides high-performance, automated sample processing for IC applications on any Dionex IC system. The autosampler can be configured for simultaneous or sequential sample delivery. In simultaneous delivery mode, samples are delivered through a splitter to two injection valves for dual full-loop injections. With this configuration, you can perform two separate analyses on one sample (e.g., anions and cations). For sequential delivery, valves are configured to divert the sample stream to the appropriate injection valve for loading.

Dionex AS-AP Sample Conductivity and pH Accessory

The inline sample conductivity and pH measurement option offers a level of automation and convenience unmatched by any other IC autosampler. The Dionex AS-AP Sample Conductivity and pH Accessory allows inline measurement of the sample conductivity and pH prior to injection. Chromeleon CDS software triggers can then be used to modify the sample as needed prior to injections. This option saves time by not having to rerun samples and puts less stress on the column, extending their lifetime.



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Matrix Elimination

Dionex OnGuard II Cartridges

Environmental samples may require removal of components that could interfere with analysis. These samples can be pretreated to remove matrix interferences like metals, anions, cations, and hydrophobic compounds using the Thermo Scientific™ Dionex™ OnGuard™ II line of disposable cartridges.



Dionex InGuard II Cartridges

The Thermo Scientific™ Dionex™ InGuard™ cartridges serve the same purpose as the Dionex OnGuard II sample pretreatment cartridges. However, they are installed inline between the Dionex AS-AP Autosampler and the IC injection valve enabling the automation of the interferant elimination process.



Dionex IonPac Guard Columns

In order to prevent sample contaminants from binding to an analytical column, an additional guard column can be placed prior to the analytical column. For example, the Dionex IonPac NG1 Guard column can be used to prevent decreased retention time or peak efficiency loss of the specific analytical cartridge being used.

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Ion Analysis

Since the development of IC analysis over 30 years ago, we have continually pioneered the development of new and innovative IC systems. Our HPIC systems include the 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 RFIC system that is commercially available. The Dionex ICS-4000 system is always ready for the next analysis delivering high-pressure IC on demand. RFIC eliminates daily tasks of eluent and regenerant preparation, saving time, preventing errors, and increasing convenience. RFIC systems with eluent generation (RFIC-EG) use electrolytic technologies to generate eluent on demand from deionized water, and to suppress the eluent back to pure water, delivering unmatched sensitivity. RFIC systems with eluent regeneration (RFIC-ER) are designed to use carbonate, carbonate/bicarbonate, or MSA eluents for isocratic separations. At the heart of our IC systems is a unique set of column chemistries that provide high selectivities and efficiencies with excellent peak shape and resolution. 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.thermoscientific.com/IC.

High-Pressure Ion Chromatography

High-pressure ion chromatography systems allow continuous operation up to 5000 psi when configured as an RFIC system for standard, microbore and capillary scale flow rates. As a result, these instruments can use high resolution 4 μ m particle ion-exchange columns which create higher backpressures. Higher backpressures are possible with the Dionex ICS-5000+ system and the Dionex ICS-4000 systems. The Dionex IonPac 4 μ m 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. HPIC systems enable the use of the 4 μ m columns, thus yielding yield fast separations with short (150 mm) columns, and high resolution with standard length (250 mm) columns.



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Reagent-Free Ion Chromatography

Minimize unintentional variations in the preparation of eluents and regenerants. Advances in eluent generation and electrolytic suppression technologies are enabling a wider variety of applications and increased productivity. RFIC-EG or RFIC-ER systems 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 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 H₂ and O₂ 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 at: www.thermoscientific.com/eluentregeneration.



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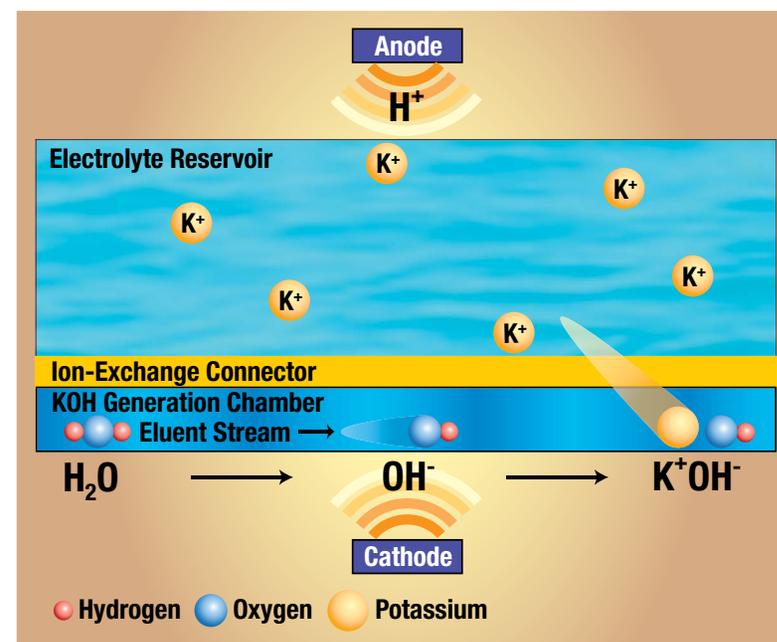
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RFIC-Eluent Generation

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 counterion 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 EGC Eluent Generation Cartridge 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 Thermo Scientific Dionex Electrolytic pH Modifier

(EPM III) can be added for generating carbonate/bicarbonate eluents. 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 at: www.thermoscientific.com/eluentgeneration.



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 electrolytic chamber.



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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.

IC-MS/(MS)

Trace analysis for environmental samples requires the removal of matrix interferences to achieve sensitive detection. An ideal analytical solution for many of these trace-level ionic contaminants utilizes the power of ion-exchange chromatography coupled with mass detection (IC-MS). Dionex RFIC systems with high-capacity anion-exchange separation improve gradient separations and trace level detection. The increased capacity and selectivity of Dionex IonPac columns help resolve trace analytes in very challenging matrices. These columns allow the separation of trace contaminants even if there are high amounts of interfering anions. During separation, the interfering ions are removed prior to detection by mass spectrometry when the eluent is diverted to waste. As a result, detection limits for the trace analyte are improved. The mobile phase is desalted by continuously-regenerated suppressors which minimizes ion suppression and allows similar response factors despite variable chromatographic conditions. Thermo Scientific provides a total solution for IC-MS with Dionex RFIC systems and the MSQ Plus Mass Spectrometer.



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Titration IC

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 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

Manual versus automated sample handling time with water quality analysis.



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Austria +43 810 282 206	Finland +358 9 3291 0200	Latin America +1 561 688 8700	Sweden +46 8 556 468 00
Belgium +32 53 73 42 41	France +33 1 60 92 48 00	Middle East +43 1 333 50 34 0	Switzerland +41 61 716 77 00
Brazil +55 11 3731 5140	Germany +49 6103 408 1014	Netherlands +31 76 579 55 55	Taiwan +886 2 8751 6655
Canada +1 800 530 8447	India +91 22 6742 9494	New Zealand +64 9 980 6700	UK/Ireland +44 1442 233555
China 800 810 5118 (free call domestic) 400 650 5118	Italy +39 02 950 591	Norway +46 8 556 468 00	USA +1 800 532 4752

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