thermo scientific

TECHNICAL GUIDE

Thermo Scientific Dionex Capillary Ion Chromatography

ThermoFisher SCIENTIFIC

Table of Contents

Capillary Ion Chromatography

Focus on Results. Uninterrupted.

In today's laboratory environment, time may be the most valuable asset when it comes to staying ahead of the curve. The ability to generate sample results quickly and accurately can require around-the-clock laboratory operation. What if your ion chromatography system operated on the same schedule, without the need to take any breaks?

Introducing Capillary Ion Chromatography
Capillary ion chromatography (IC) is a technique, developed solely by Thermo Fisher Scientific, that will save your laboratory time.

When using capillary IC, column size, injection volumes, and flow rates are scaled down by a factor of 25 to 100. Capillary IC can be used for most ion analysis applications, with the exception of polyacrylate-treated water, borated water, metals, and oligosaccharide analysis.

Always Ready
Start runs quickly by minimizing calibration and equilibration times with an always ready, truly on-demand IC system that comes with versatile detector options.

Lower Cost of Operation
The lower flow rate of capillary IC systems reduces eluent consumption and produces less waste, reducing disposal costs.

Analyze More with Less
For limited volume or precious samples, inject as little as 0.1 µL of sample even when performing trace-level analysis.

The Thermo Scientific™ Dionex™ ICS-6000 and Thermo Scientific™ Dionex™ ICS-4000 HPIC™ systems are our latest innovations in capillary ion chromatography.

Dionex ICS-6000 and Dionex ICS-4000 High-Pressure Ion Chromatography Systems

Benefits of Capillary Ion Chromatography

24/7 System Operation

Capillary IC requires only 15 mL of water a day. That is just 5.2 L a year. As a result, the system can operate with continuous eluent flow, thereby eliminating the need to wait for equilibration, and providing a true on-demand system.

Lower Cost of Operation

Capillary IC typically uses a flow rate of 10 µL/min, consuming only 15 mL of eluent per day. With automated Eluent Generation™, the Eluent Generator Cartridge (EGC) lasts 18 months at this flow rate, reducing the waste volume from 520 L to 5.2 L. This feature can be particularly beneficial for laboratories that analyze radioactive or highly toxic samples.

Optimum Balance Between Resolution and Throughput

Capillary IC systems operate at pressures up to 5000 psi. This feature enables the use of 4 µm-particle-size IC columns, available in 150 or 250 mm lengths, to help optimize speed and resolution for your application.

Improved Sensitivity with 2D-IC

Two dimensional IC (2D-IC) with a standard bore column in the first dimension (i.d. 4 mm) and a capillary column (i.d. 0.4 mm) in the second dimension, permits low detection limits in the ng/L range.

Table 1. Comparison of analytical versus capillary ion chromatography operating parameters.

	Analytical	Capillary
Column i.d.	4 mm	0.4 mm
Flow Rate	1.0 mL/min	0.010 mL/min
Injection Volume	40 µL	0.1 µL
Eluent Consumption/Waste Generated	43.2 L/month	0.432 L/month
EGC Cartridge Lifetime (at 39 mM)	5 months	18 Months

Capillary High-Pressure Ion Chromatography Systems

The high pressure capability of our capillary IC systems offers the flexibility to use small-particle columns for higher resolution, without sacrificing speed.

Dionex ICS-6000 HPIC System

Developed with flexibility and ease-of-use in mind, the Dionex ICS-6000 HPIC system gives you the freedom to push the boundaries of ion analysis. With the ability to analyze samples at capillary, microbore, or standard flow rates (or any combination of two, in a dual system) at up to 5000 psi, the system is our most adaptable IC system. The wide variety of Dionex ICS-6000 modules allows you to configure your ion chromatography system to provide the analytical solutions you need.

Dionex ICS-4000 HPIC System

The Dionex ICS-4000 Capillary HPIC system is a dedicated capillary ion chromatography system. High-pressure capability brings a new level of resolution and speed to ion chromatography, while the integrated design of the Dionex ICS-4000 HPIC system simplifies your workflow and increases your analytical efficiency and productivity. Additionally, with an array of detectors and options, the Dionex ICS-4000 HPIC system is an incredibly versatile system.

Best Practices

With the low flow rates and small volumes used in capillary IC, the number of connectors, void volumes, and the tubing lengths between modules must be minimized during installation. To achieve good chromatography by capillary IC, it is critical to:

1. Use easy-to-install Thermo Scientific™ Dionex™ IC PEEK Viper™ fittings. No training time is required to use these low-dead-volume fittings.
2. Flush and hydrate all consumables and cartridges according to the respective device and system manuals.
3. Flush air bubbles from the system initially and after any change to the system.
4. Keep the capillary IC system running to maintain stability.
5. If the system is shut down, open the prime valve on the pump to relieve pressure and minimize residual flow.

Maximizing the Benefits of Capillary IC

The above best practices will help ensure that you obtain good chromatographic results using the low volumes typical of capillary IC systems. Full details and additional instructions can be found in

Thermo Scientific Technical Note 113: Practical Guidance for Using Capillary Ion Chromatography.

Flow Path

Capillary reagent-free ion chromatography (RFIC™) systems have many similarities to standard bore RFIC systems. In RFIC systems, a dual piston pump delivers deionized water to an eluent generator cartridge (EGC), where the eluent is electrolytically generated in-line to the injection valve. At the valve, the autosampler introduces the sample through a sample loop (or concentrator column) to the ion-exchange column. The ions are then separated and eluted into the suppressor where the background conductivity is reduced to nearly zero, and the strong acid or base forms of the ions are detected by the conductivity detector.

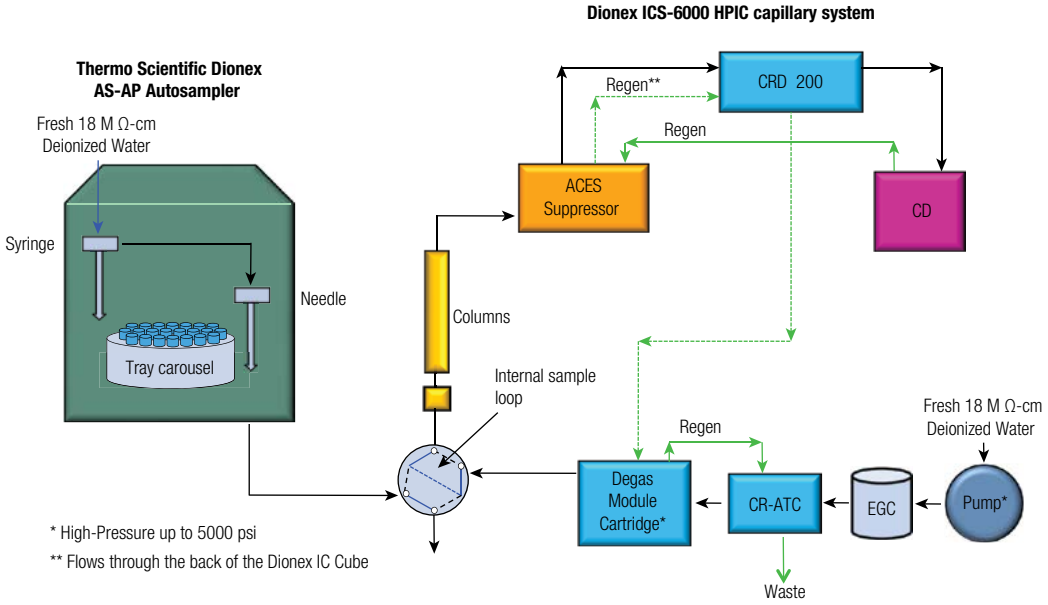


Figure 1. Flow diagram for a Dionex capillary IC System.

The Dionex IC Cube

Capillary IC uses 0.001 to 0.100 mL/min flow rates. The Thermo Scientific™ Dionex™ IC Cube™ module was designed to minimize the dead volume in the flow path (Figure 2). The Dionex IC Cube module contains the EG degas, columns, column tray, capillary electrolytic suppressor, and Thermo Scientific™ Dionex™ CRD 200 (Capillary) carbonate removal device cartridges, plus the injection valve. Injection valves are available in 400 nL, 200 nL, 100 nL internal loop, or 6-port valve options.

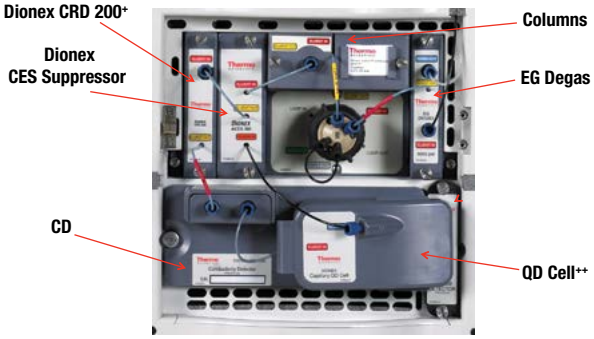


Figure 2. Components of the Dionex IC Cube module shown with the Dionex QD and CD detectors.

Small Particles, Big Benefits

Chromatographic separations using packed columns benefit from a high number of theoretical plates per column. The number of theoretical plates can be increased by packing smaller resin particles into the columns. Typically ion chromatography columns use resin particles ranging from 7–9 µm in diameter. Recent developments in resin technology have allowed the use of 4 µm resin particles in ion exchange columns.

The benefits of columns packed with smaller particles include:

- Better resolution using 250 mm i.d. columns
- Faster run times using 150 mm i.d. columns

4 µm particle-size columns are available in 4 mm, 2 mm, and capillary i.d. formats, offering higher resolution and faster separations.

Shorter, 150 mm-length columns can be used at higher flow rates to increase productivity without sacrificing performance. Longer, 250 mm-length columns are utilized for higher resolution separations of complex sample matrices. As a result, these columns allow you to choose the optimum balance between resolution and throughput.

Figure 3 shows an example of a separation of inorganic cations and biogenic amines in less than nine minutes, using a capillary Thermo Scientific™ Dionex™ IonPac™ CS19-4µm column. To achieve these fast run times, the flow rate was increased from the standard of 0.010 mL/min to 0.020 mL/min and a steep gradient was applied.

Peaks:

1.	Lithium	0.05 mg/L
2.	Sodium	0.2
3.	Ammonium	0.25
4.	Potassium	0.5
5.	Magnesium	0.25
6.	Calcium	0.5
7.	Impurity	
8.	Putrescine	7.5
9.	Cadaverine	4.5
10.	Histamine	6.5
11.	Agmatine	5.0
12.	Spermine	3.0
13.	Spermidine	1.5

Note: Concentrations are approximate

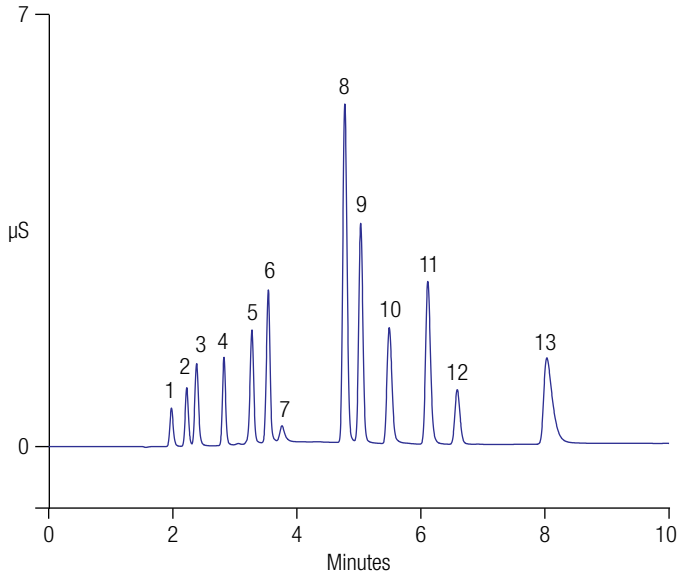


Figure 3. Fast separation of six inorganic cations and biogenic amines using the capillary Dionex IonPac CS19-4µm column.

Capillary Electrolytic Eluent Generation

An electrolytic eluent generator in a capillary format provides an ideal eluent generation and delivery platform for both isocratic (Figure 4) and gradient (Figure 5) capillary separations. The eluent generator:

- Provides a reliable and convenient eluent source and eliminates the errors associated with manual eluent preparation
- Is capable of providing precise and accurate gradients through precise current and flow rate controls
- Generates eluent concentrations up to 200 mM at capillary flow rates, enabling faster elution of highly retained ions
- With RFIC, delivers only water through the pump, prolonging pump seals and reducing maintenance costs

Thermo Scientific Dionex capillary RFIC systems are capable of highly reproducible separation of analytes under gradient elution by minimizing the error that comes with manually preparing eluents and eliminating contamination from the environment (e.g., carbon dioxide), as shown in Figure 5.

Overlay of 30 consecutive runs
Retention time RSD (n = 30):
0.060% (bromide) to 0.091% (fluoride)
Peak area RSD (n = 30):
0.24% (sulfate) to 0.50% (fluoride)

Peaks:
1. Fluoride
2. Bromate
3. Chloride
4. Nitrite
5. Chlorate
6. Bromide
7. Nitrate
8. Sulfate

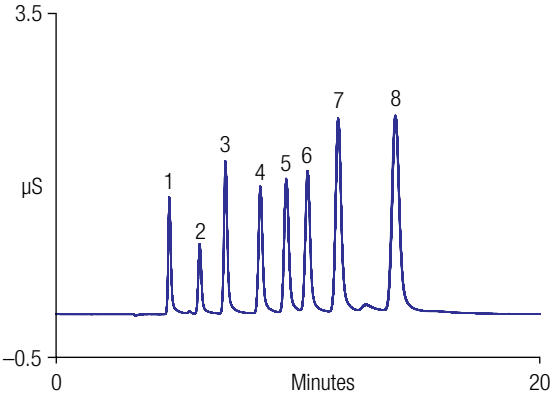


Figure 4. Isocratic separation of common anions obtained using a capillary Dionex IonPac AS19 column.

Peaks:
1. Fluoride
2. Acetate
3. Formate
4. Chlorite
5. Bromate
6. Chloride
7. Nitrite
8. Chlorate
9. Bromide
10. Nitrate
11. Sulfate
12. Malonate
13. Selenate
14. Oxalate
15. Iodide
16. Chromate
17. Thiosulfate
18. Phosphate
19. Fumarate
20. Arsenate
21. Thiocyanate
22. Perchlorate

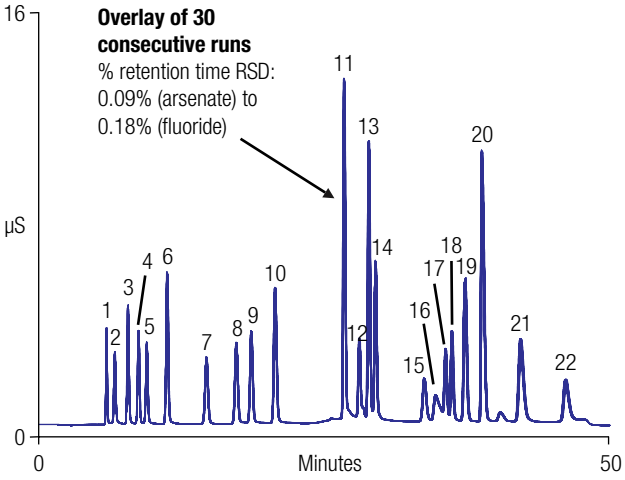


Figure 5. Gradient separation of 22 anions using a capillary Dionex IonPac AS19 column.

Enhanced Determination of Trace Analytes

When performing capillary IC, analytes can be determined with sample injections as small as 0.1 µL. This can be especially beneficial when working with precious samples (e.g., biological samples) or determining trace-level analytes. One approach is to perform a large-volume direct injection, which is suitable for samples with low levels of matrix ions. A 10 µL injection onto a 0.4 mm i.d. column in a capillary IC system is equivalent to a 1000 µL injection onto a 4 mm i.d. column. Another approach is loading a 250 µL sample onto a capillary concentrator rather than loading a 25 mL sample onto a 4 mm concentrator. Therefore, capillary IC systems can offer significant benefits in trace analysis, especially in applications where sample volumes are limited.

Figure 6 demonstrates the separation of inorganic anions at trace concentrations using a capillary Dionex IonPac AS19 column. With a 10 µL injection, a capillary RFIC system is capable of determining the target anions at concentrations ranging from 0.2 µg/L to 1.0 µg/L with excellent signal-to-noise ratios.

Peaks:	Concentration
1. Fluoride	0.2 µg/L
2. Chloride	0.3
3. Nitrite	1.0
4. Bromide	1.0
5. Nitrate	1.0
6. Carbonate	—
7. Sulfate	1.0

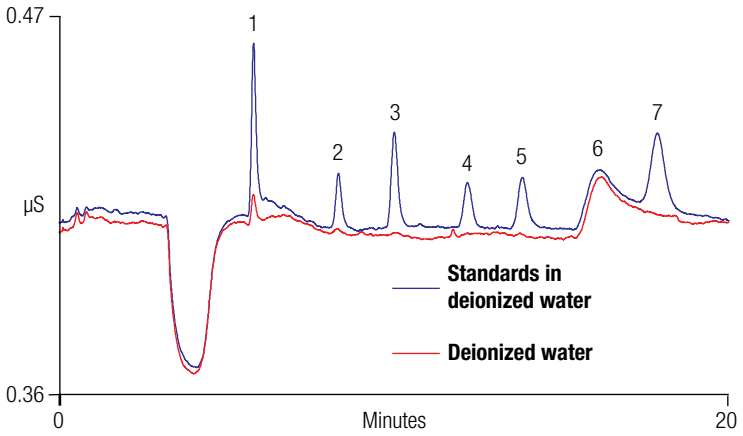


Figure 6. Separation of inorganic anions at trace concentrations using a capillary Dionex IonPac AS19 column with a 10 µL injection.

Capillary Electrolytic Suppressor

The Thermo Scientific™ Dionex™ CES™ 300 Capillary Electrolytic Suppressors are optimized for the eluent flow rates typically seen in capillary systems (5–30 µL/min). When used for anion analysis, the Thermo Scientific™ Dionex™ ACES™ 300 Anion Capillary Electrolytic Suppressor converts highly conductive hydroxide-based eluents into pure water, reducing the baseline conductivity. While suppressing the eluent, the Dionex ACES 300 suppressor also converts the analytes into their more conductive hydronium (acid) form, increasing their sensitivity under conductivity detection. Likewise, when used for cation analysis, the Thermo Scientific™ Dionex™ CCES™ 300 Cation Capillary Electrolytic Suppressor converts highly conductive methanesulfonic acid (MSA) eluents into pure water; simultaneously, the analytes are converted to their more conductive hydroxide form, increasing their sensitivity.

Dionex CES 300 Suppressor Technology

The Dionex CES suppressor uses a three-chamber design to minimize dead volume while maximizing suppression capacity and reducing noise. The eluent chamber includes an ion-exchange capillary membrane, which facilitates the efficient exchange of the eluent counterions for regenerant ions. The regenerant chambers are divided into ion-exchange and electrode chambers. Regenerant first passes through the ion-exchange chamber, which is filled with a bed of ion exchange resin; the ion exchange capillary membrane is coiled in this bed. The regenerant bed is an ion-exchange resin in the opposite form as the eluent. This bed of resin provides the regenerant ions for the capillary membrane eluent chamber. There are two electrode chambers that are separated from the ion-exchange chamber by a pair of ion-exchange membranes. After passing through the ion-exchange chamber, regenerant passes through the cathode and anode chambers serially. When current is passed through the electrodes, regenerant ions are generated in the first electrode chamber; these ions are pushed into the ion-exchange chamber via an electric field, maintaining the ion-exchange chamber in the regenerant form. After counterions exchange from the eluent ion exchange capillary membrane, they are then removed from the ion exchange chamber via the electric field into the second electrode chamber. Finally, the counterions are neutralized by the ions generated in the second electrode chamber.

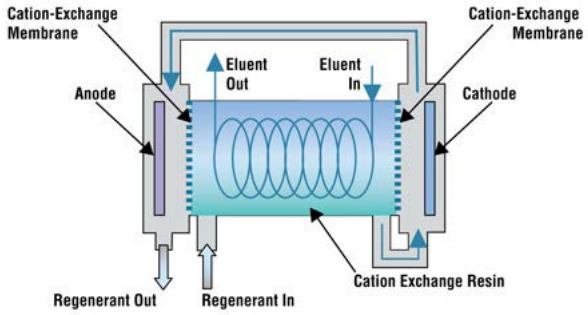


Figure 7. Dionex CES 300 Capillary Electrolytic Suppressor (above) and Dionex CES flow diagram (below).

Capillary Carbonate Removal Device

In capillary format anion determinations, a carbonate removal device, such as the Thermo Scientific™ Dionex™ CRD 180, 200 or 300 Carbonate Removal Device, is recommended for removing the carbonate peak that interferes with adjoining analyte peaks. For samples with low carbonate content, a carbonate removal device is not required, and analysts benefit from sharper peaks due to decreased void volume.

Capillary IC with Conductivity Detection

The Thermo Scientific Dionex Conductivity Detector handles any IC application, from single-column methods with high background signals to determinations of trace contaminants in high-purity water. Since all ions are electrically conductive, the conductivity detector is an ideal universal detector for most ion chromatography applications. Suppressed conductivity reduces the background conductivity of the eluent and the strong acid or base forms of the ions are detected by conductivity. The capillary conductivity detector is volume optimized for capillary flow rates and does not require a heat-exchanger.

Features of conductivity detection:

- Optimized for capillary applications
- Minimal cell volume (0.02 µL)
- Microprocessor-controlled digital signal processing detects high and trace concentrations
- Large dynamic range—up to 15,000 µS/cm
- Minimizes noise while maximizing sensitivity

Figure 8. Dionex CD Conductivity Detector.

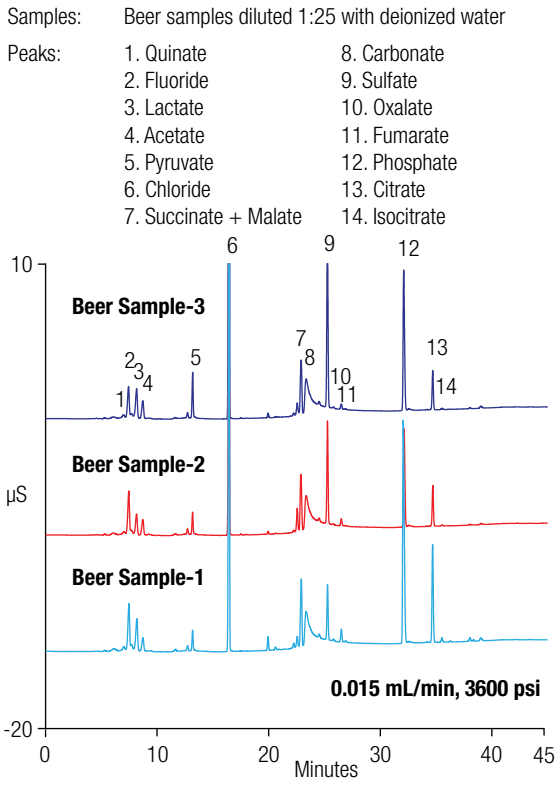


Figure 9. Analysis of anions in fermentation products using a capillary Dionex IonPac AS11-HC-4µm column.

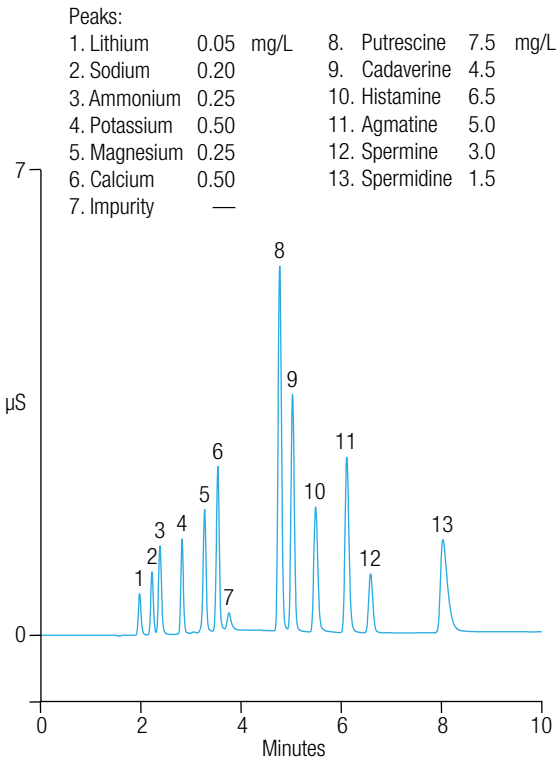


Figure 10. Fast separation of six common cations and biogenic amines using the Dionex IonPac CS19-4µm capillary column.

Capillary IC with Electrochemical Detection

Electrochemical detection is used in combination with ion chromatography to detect the presence of compounds that contain an oxidizable or reducible moiety within their structure. Target compounds are separated on a column and enter the electrochemical detector. The compound is either oxidized or reduced and the flow of electrons is detected as current by the electrochemical detector (ECD). The current linearly correlates to the analyte concentration.

Features of electrochemical detection:

- Volume and flow optimized for performance and low backgrounds
- High sensitivity (femtomole detection capability)
- Optional palladium hydrogen reference electrode for superior reproducibility and long lifetime
- Microprocessor-controlled digital signal processing
- DC amperometry, cyclic voltammetry, and integrated amperometry including 3-D (current, voltage, retention)

Figure 11. Thermo Scientific Dionex Electrochemical Detector Cell for capillary-scale applications.

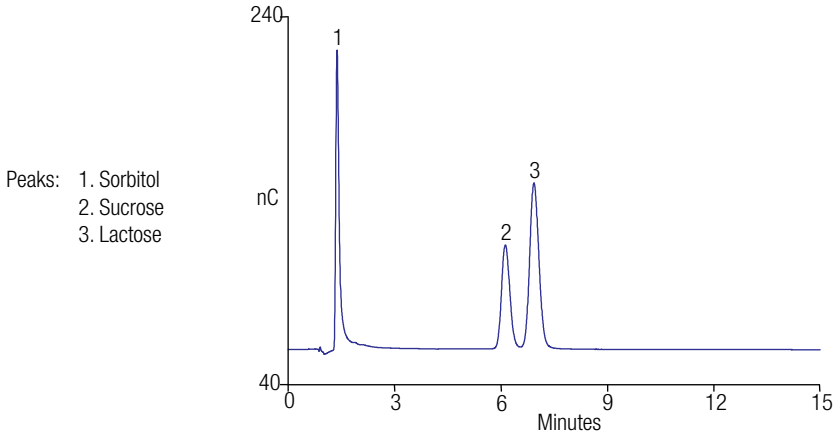


Figure 12. Separation of an alditol and disaccharides using a capillary Dionex CarboPac PA20 column (0.4 × 150 mm).

Capillary Ion Chromatography Column Guide

Thermo Fisher Scientific provides a wide range of capillary ion chromatography columns, and our unique column chemistries are designed for specific applications. We offer a variety of selectivities and capacities for simple and complex sample matrices to address all your separation needs.

Dionex IonPac capillary cation- and anion-exchange columns

Column	Market	Application	Analytes
Dionex IonPac AS9-HC	Environmental	Compliance testing of drinking water	Inorganic anions and oxyhalides
Dionex IonPac AS11-HC-4µm	Food and Beverage	Adulteration, profiling of foods and beverages	Organic acids, inorganic anions
Dionex IonPac AS11-HC	Food and Beverage	Adulteration, profiling of foods and beverages	Organic acids, inorganic anions
Dionex IonPac AS15	Power/Semiconductor	Trace contamination of high purity water	Inorganic anions, low molecular weight organic acids
Dionex IonPac CS16-4µm	Power/Chemical	Disparate ratios in industrial samples	Sodium and ammonium
Dionex IonPac CS16-Fast-4µm	Power/Chemical	Disparate ratios in industrial samples	Sodium and ammonium
Dionex IonPac AS16-4µm	Environmental	Compliance testing of drinking water	Trace perchlorate
Dionex IonPac AS16	Environmental	Compliance testing of drinking water	Trace perchlorate
Dionex IonPac AS18-Fast-4µm	Environmental	Fast compliance testing of drinking water	Inorganic anions
Dionex IonPac AS18-Fast	Environmental	Fast compliance testing of drinking water and wastewater	Inorganic anions
Dionex IonPac AS18	Environmental	Compliance monitoring of drinking water and wastewater	Inorganic anions
Dionex IonPac AS19-4µm	Environmental	Compliance monitoring of drinking water	Trace bromate, inorganic anions, oxyhalides
Dionex IonPac AS19	Environmental	Compliance monitoring of drinking water	Trace bromate, inorganic anions, oxyhalides
Dionex IonPac AS20	Environmental	Compliance testing of drinking water	Trace perchlorate
Dionex IonPac CS20	Power/Chemical	Profiling, monitoring in industrial samples	Inorganic cations and amines
Dionex IonPac AS22-Fast-4µm	Environmental	Compliance monitoring of drinking water and wastewater	Inorganic anions
Dionex IonPac AS22	Environmental	Compliance monitoring of drinking water and wastewater	Inorganic anions
Dionex IonPac AS23	Environmental	Compliance monitoring of drinking water and wastewater	Inorganic anions and oxyhalides
Dionex IonPac AS24A	Environmental	Drinking water analysis	Haloacetic acids
Dionex IonPac AS25	Chem/Petrochem	Process monitoring	Sulfur speciation, polyvalent anions
Dionex IonPac AS26	Environmental	Drinking water analysis	Haloacetic acids
Dionex IonPac AS27	Environmental	Compliance testing of drinking water preserved with ethylenediamine (EDA)	Trace bromate
Dionex IonPac AS28-Fast-4µm	Power/Semiconductor	Trace contaminants in high purity water	Inorganic anions, low molecular weight organic acids

Dionex IonPac capillary cation-exchange columns

Column	Market	Application	Analytes
Dionex IonPac CS19-4µm	Power/Chemical	Profiling, monitoring in industrial samples	Inorganic cations, small polar amines
Dionex IonPac CS12A-8µm	Environmental	Monitoring of Drinking Water	Inorganic cations plus ammonium
Dionex IonPac CS12A-5µm	Environmental	Monitoring of Drinking Water	Inorganic cations plus ammonium
Dionex IonPac CS16	Power/Chemical	Disparate ratios in industrial samples	Sodium and ammonium
Dionex IonPac CS17	Power/Chemical	Profiling, monitoring in industrial samples	Polyvalent and moderately hydrophobic amines, including diamines
Dionex IonPac CS19	Power/Chemical	Profiling, monitoring in industrial samples	Inorganic cations, small polar amines

Dionex CarboPac capillary carbohydrate columns

Column	Market	Application	Analytes
Dionex CarboPac PA20	Food and Beverage	Fast monitoring in food and beverage samples	Mono- and disaccharides
Dionex CarboPac PA10	Food and Beverage	Monitoring in food and beverage samples	Mono- and disaccharides

Determination of Carbohydrates in Urine by Capillary HPAE-PAD

Summary

Researchers have studied the presence of certain carbohydrates such as mannitol, rhamnose, xylose, 3-O-methylglucose, and lactulose in both urine and serum samples to evaluate disease states in animals and humans. Because carbohydrates are poor chromophores, they are difficult to detect by UV absorption without lengthy and costly derivitization. However, carbohydrates can be determined directly by High Performance Anion-Exchange chromatography with Pulsed Amperometric Detection (HPAE-PAD), a well-established technique that eliminates the need for derivitization, in turn saving time and money (i.e., reagent and disposal costs). This technical note demonstrates a method to determine mono- and disaccharides in synthetic urine on a capillary ion chromatography system.

Download the Technical Note

Mono- and Disaccharides in Urine

Equipment

- Dionex ICS-4000 Capillary HPIC System*
- Dionex IC Cube
- Dionex Electrochemical Detector (ED)
- Dionex Electrochemical Cell, reference electrode with gasket, and working electrode with gasket
- Dionex AS-AP Autosampler
- Thermo Scientific™ Chromeleon™ Chromatography Data system 7 (CDS), version 7.1 with SR2 MUa build or later

*Dionex ICS-6000 HPIC system can be used for equivalent results

Reagents and Standards

- 18 MΩ-cm resistivity degassed deionized water
- pH Buffer solutions, pH 7 (Fisher Scientific, P/N SB108-500); pH 10 (Fisher Scientific, P/N SB115-500)

Conditions

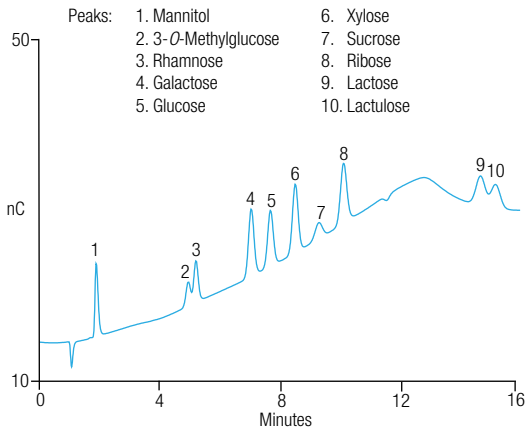
Columns:	Dionex CarboPac PA20 column (0.4 × 150 mm)
Eluent Source:	Thermo Scientific Dionex EGC-KOH Eluent Generator Cartridge (Capillary)
Eluent:	10 mM KOH (–7 to 1 min); 10–30 mM KOH (1 to 9 min); 30–35 mM KOH (9 to 16 min); 35 mM KOH (16 to 21 min); 10 mM KOH (21 to 37 min)
Flow Rate:	0.008 mL/min
Column Temp.:	30 °C
Compartment Temp.:	27 °C
Inj. Volume:	0.4 µL
Detection:	PAD, Gold on PTFE, 0.001" thick gasket, Four-Potential Carbohydrate waveform
Reference Electrode:	pH-Ag/AgCl
Background:	10–20 nC
Noise:	< 10 pC

Analysis

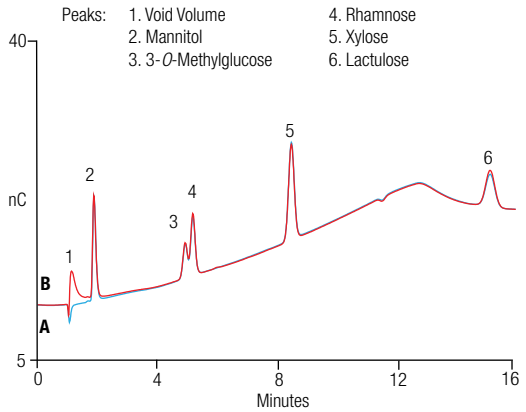
Capillary HPAE-PAD

Results

See chromatograms below.

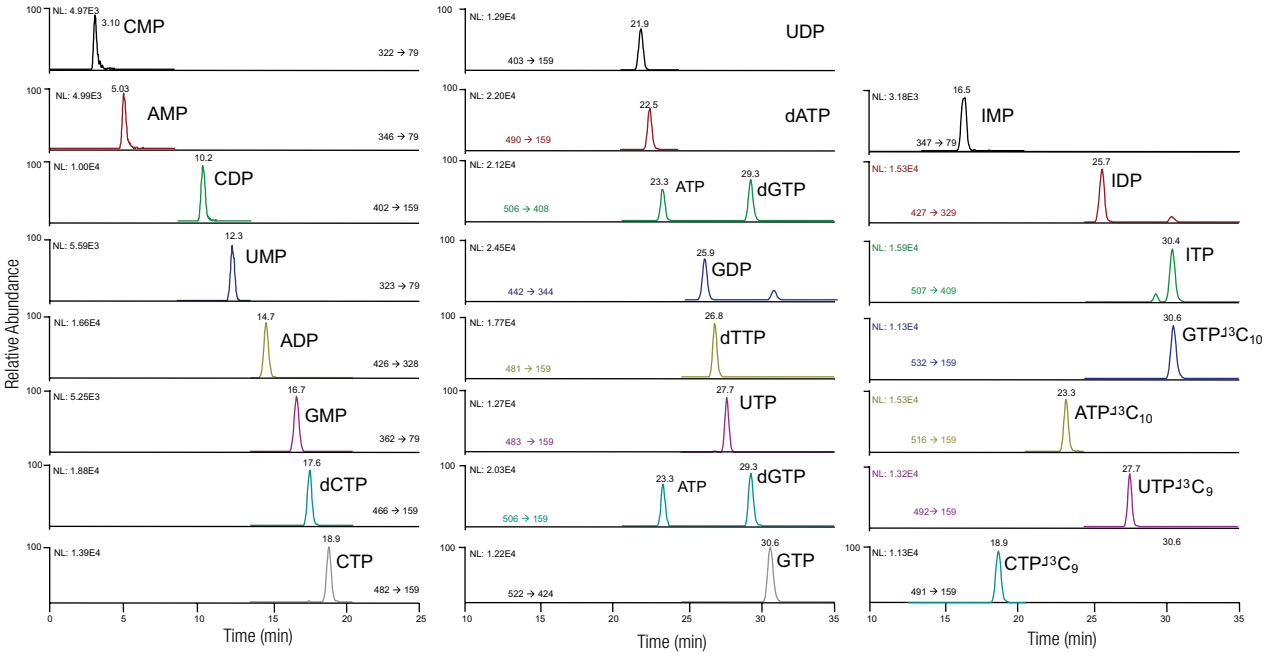


Carbohydrates of interest for urine analysis.



Carbohydrates in A) water and B) synthetic urine.

Quantitative Profiling of Nucleotides Using Capillary IC-MS/MS



Using a Dionex ICS-5000 RFIC system coupled to a TSQ Quantum Access MAX triple quadrupole MS, a Dionex IonSwift MAX-100 anion-exchange column, and an electrolytically generated hydroxide gradient, 19 native and 2 modified nucleotides were separated and detected within 40 minutes.

Improved Determination of Trace Perchlorate in Drinking Water Using 2D-IC

Summary

Perchlorate is identified as an environmental contaminant found in drinking, ground, and surface waters. Research on perchlorate in the environment has received attention because perchlorate poses a human health concern. Perchlorate impairs normal thyroid function by interfering with iodine uptake by the thyroid. Ion chromatography is recognized as an effective tool for the determination of perchlorate in drinking water and other samples. This study demonstrates a 2D-IC system for determining trace concentrations of perchlorate in drinking waters in accordance with EPA Method 314.2 using a capillary format in the second dimension.

Download the Application Note

Perchlorate in Drinking Water

Equipment

- Dionex ICS-5000 Hybrid (Analytical/Capillary) system,** including:

– DP Dual Pump

– Dionex EG Eluent Generator

– DC Detector/Chromatography Compartment

– Dionex AS-AP Autosampler* with Sample Syringe, 5.0 mL (P/N 074308) and 8.5 mL buffer line assembly (P/N 075520)

- Dionex Potassium Hydroxide Eluent Generator Cartridge (EGC III KOH, P/N 074532) and Capillary Cartridge (EGC KOH, P/N 072076)
- Thermo Scientific Dionex CR-ATC Continuously Regenerated Anion Trap Column (P/N 060477) and Capillary Trap Column (P/N 072078)
- Dionex CRD 200 Carbonate Removal Device, 2 mm (P/N 062986) and Capillary Dionex CRD 200 Carbonate Removal Device, (P/N 072054)
- Dionex IonSwift MAC-200 Monolith Anion Concentrator Column (P/N 075461)
- Vial Kit, Polystyrene with Caps and Blue Septa, 10 mL (P/N 074228)
- Dionex IC Cube Cartridge with six-port valve (P/N 078841)
- Corning™ Syringe Filter, surfactant-free cellulose acetate (SFCA), 0.2 µm pore, 26 mm (Fisher Scientific (P/N 09-754-13)
- Disposable syringe, 20 mL (24 mL) Luer Lock, Sterile (Fisher Scientific P/N 14-817-33)
- Sterile sample container, 125 mL, I-Chem™ Sterile Nalgene™ Bottles (Fisher Scientific P/N N411-0125)
- PEEK tubing, 38 cm (15 in.) piece of 0.0 25 mm (0.001 in.) i.d. (P/N 074582) for conditioning a new capillary Dionex EGC cartridge

*A Dionex AS or AS-DV Autosampler can also be used for sample delivery

**The Dionex ICS-4000 HPIC system or Dionex ICS-6000HPIC system can be used for equivalent results

Reagents and Standards

- Deionized water (DI), Type I reagent grade, 18 MΩ-cm resistance or better
- Sodium perchlorate, 98%, extra pure (Fisher Scientific P/N AC34218)
- Sodium chloride, crystalline, 99.0% (Fisher Scientific P/N S671)
- Sodium sulfate, anhydrous (Fisher Scientific P/N S429)
- Sodium bicarbonate, certified ACS (Fisher Scientific P/N S233)

First Dimension Conditions

Columns:	Dionex IonPac AG20 Guard column, 2 × 50 mm (P/N 063066)
	Dionex IonPac AS20 Analytical column, 2 × 250 mm (P/N 063065)
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, Thermo Scientific™ Dionex™ ASRS™ 300 Anion Self-Regenerating 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

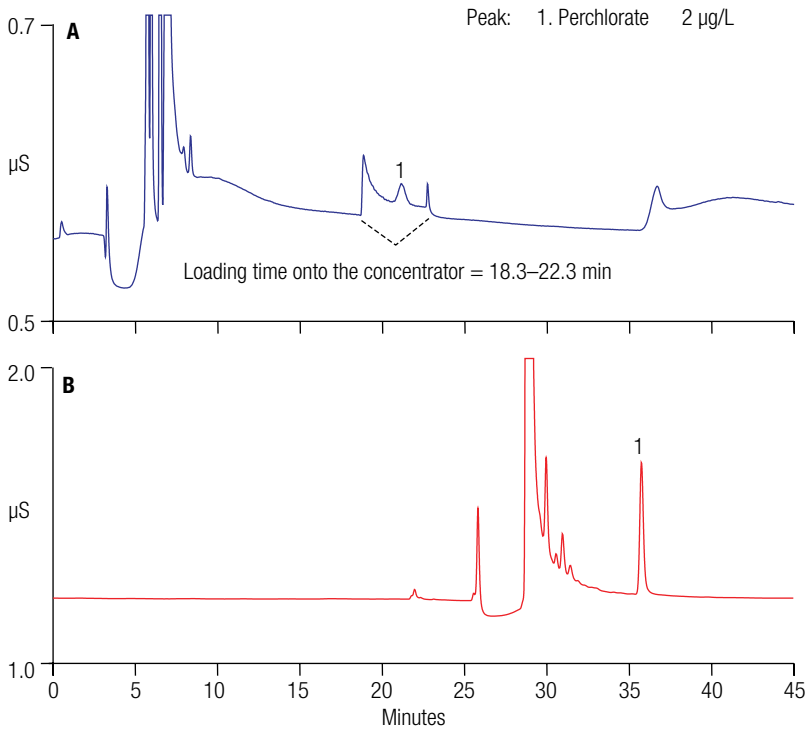
Second Dimension Conditions	
Columns:	Dionex IonPac AG16 Capillary Guard column, 0.4 × 50 mm (P/N 082316) Dionex IonPac AS16 Capillary Analytical column, 0.4 × 250 mm (P/N 082315)
Eluent Source:	Dionex EGC-KOH Cartridge (Capillary) with Dionex CR-ATC Continuously Regenerated Anion Trap Column (Capillary)
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 ACES 300 suppressor (P/N 072052), 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

Analysis

2D-IC

Results

See chromatogram below.



Chromatogram of a 2 µg/L perchlorate standard in reagent water in (A) first dimension and (B) second dimension.

Determination of Nitrite and Nitrate in Wastewater Using Capillary IC with UV Detection

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 Application Update

Nitrite and Nitrate in Wastewater

Equipment

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

*Dionex ICS-4000 HPIC sytem or Dionex ICS-6000 HPIC sytem 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/min
Inj. Volume:	0.4 µL
Detection:	A: Suppressed conductivity, Dionex ACES 300 suppressor, recycle mode B: UV, 210 nm, capillary

Analysis

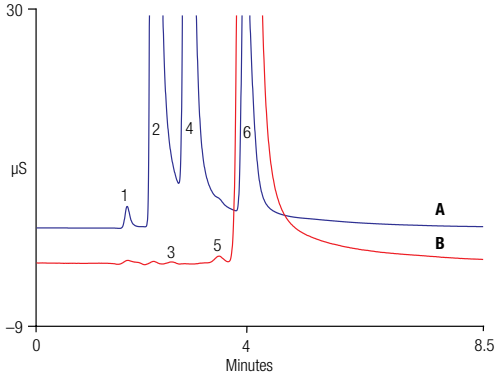
Capillary Ion Chromatography

Results

See chromatogram below.

Column:	Dionex IonPac AS18-Fast column, 0.4 mm × 150 mm
Eluent Source:	EGC-KOH, cartridge (Capillary)
Eluent:	33 mM KOH
Flow Rate:	15 µL/min
Inj. Volume:	0.4 µL
Column Temp.:	30 °C
Detection:	A. Suppressed conductivity, capillary B. UV, 210 nm, capillary
Sample Prep:	Filtered prior to analysis

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



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

Determination of Inorganic Anions in Acid Rain Using a Dedicated High-Pressure Capillary Ion Chromatography System

Summary

Acid rain is closely monitored by many countries due to its negative effects on plants, aquatic animals, infrastructures, and human health. The cause of acid rain is related to the reaction of water in the atmosphere with sulfur dioxide (SO_2) and nitrous oxides (NO_x) released from natural phenomena, such as volcanic eruption and lightning strikes. Some human activities, for example, coal power plants and motor vehicles, contribute to the level of SO_2 and NO_x in the air as well. Air and rain monitoring has become an important task worldwide for investigating the effects of pollutants on the global ecology and assessing the advancement of pollution control. This method demonstrates rapid analysis of determining inorganic anions in rainwater with a high-pressure capillary IC and 4 µm particle-size columns.

Download the Technical Note

Inorganic Anions in Acid Rain

Equipment

- Dionex ICS-4000 HPIC system*
- Dionex AS-AP Autosampler
- Chromeleon CDS software 7.1 with SR2 MUa build or later

*Dionex ICS-6000 HPIC sytem can be used for equivalent results

Reagents and Standards

- 18 MΩ-cm degassed deionized water
- Fluoride standard 1000 mg/L (Thermo Scientific Dionex P/N 037158)
- Chloride standard 1000 mg/L (Thermo Scientific Dionex P/N 037159)
- Sulfate standard 1000 mg/L (Thermo Scientific Dionex P/N 037160)
- Nitrate standard 1000 mg/L (Thermo Scientific Dionex P/N 056497)
- Bromide standard 1000 mg/L (Ultra Scientific P/N ICC-001)
- Nitrite standard 1000 mg/L (Ultra Scientific P/N ICC-007)
- Phosphate standard 1000 mg/L (Ultra Scientific P/N ICC-005)

Conditions

Columns:	Dionex IonPac AG18-4µm column, 0.4 × 50 mm
	Dionex IonPac AS18-4µm column, 0.4 × 150 mm
Eluent Source:	Dionex EGC-KOH Cartridge (Capillary)
Eluent:	23 mM KOH
Flow Rate:	A: 0.025 mL/min for sample B: 0.010, 0.020, and 0.025 mL/min for standards
Column Temp.:	30 °C
Compartment Temp.:	15 °C
Inj. Volume:	0.4 µL (full loop injection mode)
Detection:	Suppressed conductivity, Dionex ACES 300 suppressor, recycle mode, 8 mA at 0.010 mL/min; 13 mA at 0.025 mL/min
Background Conductance:	< 1 µS
Noise:	< 1 nS
System Backpressure:	~1600 psi (0.010 mL/min); ~2900 psi (0.020 mL/min); ~3500 psi (0.025 mL/min)

Analysis

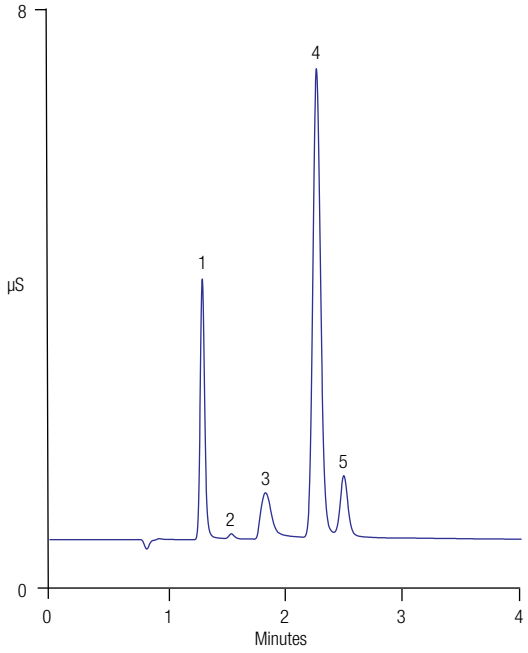
Capillary Ion Chromatography

Results

See chromatogram below.

Column :	Dionex IonPac AG18 column, AS18-4µm column, capillary
Eluent Source:	Dionex EGC-KOH Cartridge (Capillary)
Eluent:	23 mM KOH
Col. Temp.:	30 °C
Inj. Volume:	0.4 µL
Flow Rate:	0.025 mL/min
Detection:	Suppressed Conductivity, Dionex ACES 300 suppressor
Sample:	Rain sample

Peaks:			
	1. Chloride	1.0	mg/L
	2. Nitrite	0.03	
	3. Carbonate	—	
	4. Sulfate	5.2	
	5. Nitrate	0.78	



Fast determination of inorganic anions in a rain water sample.

Determinations of Monosaccharides and Disaccharides in Beverages by Capillary HPAE-PAD

Summary

Mono- and disaccharide sugar determinations are often used in the food and beverage industry to ensure the quality of a formulated product, to maintain or select for desired sweetness, and to characterize and confirm the source of the carbohydrates. Carbohydrates have poor chromophores and are therefore problematic to detect by UV absorption without lengthy and costly derivitization. However, carbohydrates can be determined directly by High Performance Anion-Exchange chromatography and Pulsed Amperometric Detection (HPAE-PAD), a well-established method that eliminates the need for derivitization, saving time and money including reagent costs. This technical note demonstrates mono- and disaccharides determinations in two-fold to 10,000-fold diluted beverage samples by HPAE-PAD at capillary flow rates on the Dionex ICS-4000 HPIC Integrated capillary system.

Download the Technical Note

Mono- and Disaccharides in Beverages

Equipment

- Dionex ICS-4000 Capillary HPIC System*
- Dionex IC Cube
- Dionex Electrochemical Detector (ED)
- Thermo Scientific Dionex Electrochemical Cell, reference electrode with gasket, and working electrode with gasket
- Dionex AS-AP Autosampler
- Chromeleon CDS software, version 7.1 with SR2 MUa build or later

*Dionex ICS-6000 HPIC system can be used for equivalent results

Reagents and Standards

- 18 MΩ-cm degassed deionized water
- ACS Grade reagents, Fisher Scientific
- Thermo Scientific™ Dionex™ MonoStandard, Mixture of Six, 100 nmol each (P/N 043162)
- pH Buffer solutions, (pH 7 / pH 10) (Fisher Scientific, P/N SB108-500 / SB115-500)

Conditions

Columns:	Dionex CarboPac PA20 column set, 0.4 × 150 mm
Eluent Source:	Dionex EGC-KOH Eluent Generator Cartridge (Capillary)
Eluent*:	10 mM KOH
Flow Rate:	0.008 mL/min
Column Temp.:	30 °C
Compartment Temp.:	27 °C
Inj. Volume:	0.4 µL
Detection:	PAD, Gold on PTFE, 0.001“ or 0.015” gasket, Four-Potential Carbohydrate waveform
Reference Electrode:	pH-Ag/AgCl
Background:	10–20 nC
Noise:	< 10 pC

*Column wash/10 samples: 5 min at 100 mM KOH, 7 min equilibration at 10 mM KOH.

Analysis

Capillary HPAE-PAD

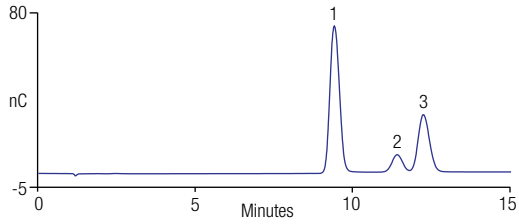
Results

See chromatograms below.

Column:	Dionex CarboPac PA20 column, 0.4 × 150 mm
Eluent Source:	Dionex EGC-KOH Cartridge (Capillary)
Eluent:	10 mM KOH (15 min)
Flow Rate:	0.008 mL/min
Inj. Volume:	0.4 µL
Column Temp.:	30 °C
Detection:	PAD, Au disposable, 0.001” gasket, 4-Potential Carbohydrate waveform
Ref. Electrode:	Ag/AgCl
Sample Prep.:	10,000-fold dilution

Peaks:

	mg/L	Total	% Ratio
1. Glucose	4.6	46 g/L	39
2. Sucrose	1.3	13	11
3. Fructose	5.9	59	50

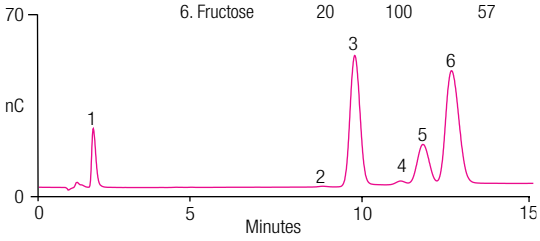


Glucose, sucrose, and fructose in tea beverage.

Column:	Dionex CarboPac PA20 column set, 0.4 × 150 mm
Eluent Source:	Dionex EGC-KOH Cartridge (Capillary)
Eluent:	10 mM KOH (15 min)
Flow Rate:	0.008 mL/min
Inj. Volume:	0.4 µL
Column Temp.:	30 °C
Detection:	PAD, Au disposable, 0.001” gasket, 4-Potential Carbohydrate waveform
Ref. Electrode:	Ag/AgCl
Sample Prep.:	10,000-fold dilution

Peaks:

	mg/L	Total	% Ratio
1. Void Volume			
2. Galactose	< 0.02	— g/L	—
3. Glucose	11	55	32
4. Mannose	0.4	2	1
5. Sucrose	3.4	17	10
6. Fructose	20	100	57



Diluted apple cider with native sugar only.

Quantitative Determination of Bisphosphonate Pharmaceuticals and Excipients by Capillary IC-MS

Summary

Bisphosphonates are a group of compounds that are used as active pharmaceutical ingredients (APIs) to treat bone disorders including osteoporosis, Paget's disease, and hypercalcemia. Typical methods for bisphosphonates analysis include liquid chromatography (LC) with derivatization and/or ion pairing, ion chromatography (IC), capillary electrophoresis (CE), and gas chromatography (GC) with derivatization. This application note discusses a quantitative approach for the direct analysis of bisphosphonates and excipients in pharmaceuticals using capillary IC with suppressed conductivity and mass spectrometric detection. This method is used to provide chromatographic retention and resolution for target analytes, and the elimination of derivatization steps simplifies the workflow and improves method throughput.

Download the Application Note

Bisphosphonate and Common Excipients in Pharmaceuticals

Equipment

- Dionex ICS-5000 Capillary HPIC System* with Eluent Generation
- Thermo Scientific MSQ Plus Mass Spectrometer (single quadrupole)
- Dionex AXP-MS Auxiliary pump (×2)
- Chromeleon CDS software, version 6.8 SR11
- Thermo Scientific™ Xcalibur 2.0.7 software with MSQ™ 2.0 SP1

*Dionex ICS-6000 HPIC system or Dionex ICS-4000 HPIC system can be used for equivalent results

Reagents and Chemicals*

- Etidronate disodium hydrate (PN P5248)
- Clodronate disodium (PN D4434)
- Tiludronate disodium hydrate (PN T4580)
- Benzoic acid sodium salt (PN B3375)
- *p*-Hydroxybenzoic acid (PN H5376)
- Citric acid (PN 27788)
- Isotope labeled internal standard citric acid-d4 (C/D/N Isotopes, Inc., PN D-3745)
- Deionized (DI) water with 18.2 MΩ-cm resistivity
- Acetonitrile (LC/MS grade, Fisher Scientific or equivalent)

All chemical standard chemicals were purchased from Sigma-Aldrich unless noted.

Chromatographic Conditions

System:	Dionex ICS-5000 capillary IC system with eluent generation
Column:	Dionex IonPac AS18-Fast Capillary Column (0.4 × 150 mm, PN 072062) Dionex IonPac AG18-Fast Capillary Guard Column (0.4 × 35 mm, PN 072063)
Eluent:	Hydroxide gradient

Time (min)	Concentration (mM)
–4.0	40
0.0	40
5.0	50
8.0	100
13.9	100
14.0	40

Eluent Source:	Dionex EGC-KOH (Capillary) Cartridge (PN 072076)
Flow Rate:	0.020 mL/min
Injection:	2 µL
Temperature:	40 °C
Detection:	1) Suppressed conductivity with Dionex ACES 300 suppressor (external water mode, 30 µL/min DI water delivered by AXP-MS pump) 2) MSQ Plus single quadrupole mass spectrometer

Mass Spectrometric Conditions

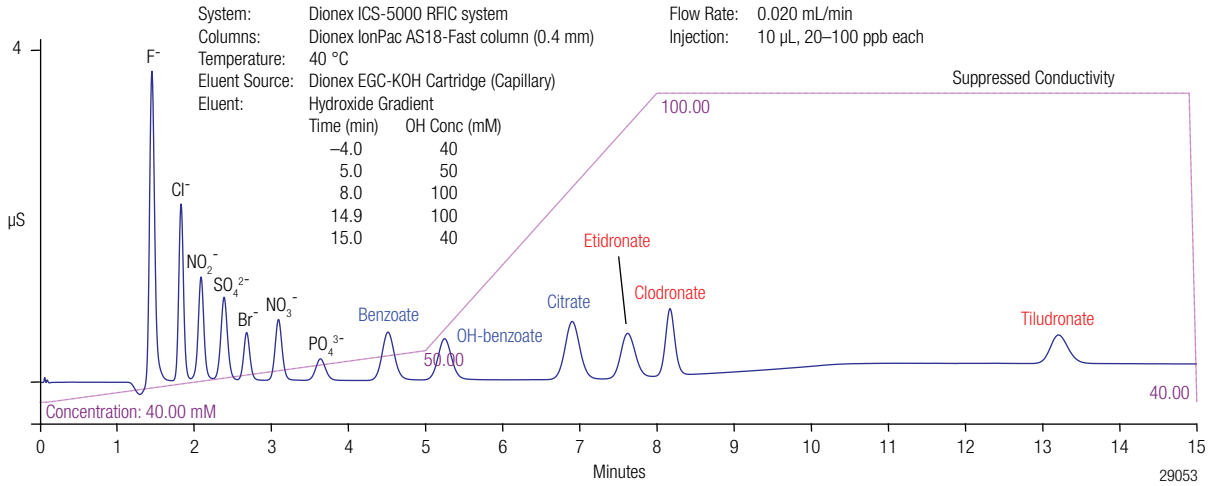
System:	MSQ Plus mass spectrometer, single quadrupole
Interface:	Capillary low-flow electrospray ionization (ESI) negative polarity
Probe:	MSQ Plus ESI probe with low-flow option (PN 078996)
Probe Temperature:	300 °C
Needle Voltage:	3500 V
Desolvation Solvent:	20 µL/min acetonitrile delivered by a Dionex AXP-MS pump
Nebulizer Gas:	Nitrogen at 65 psi
Acquisition:	Selected ion monitoring (SIM) with cone set at 55 V for each SIM with 0.3 amu span

Analysis

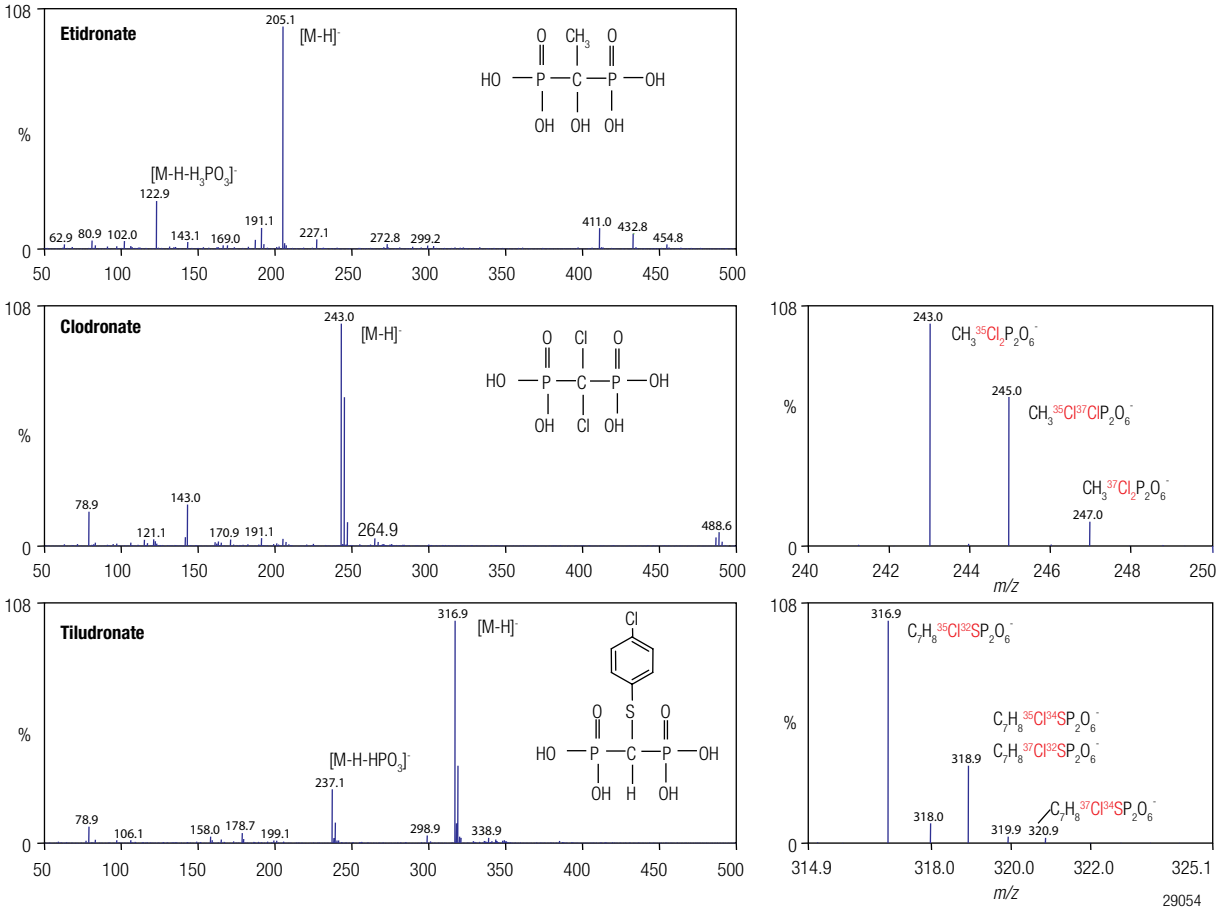
Capillary IC-MS

Results

See chromatogram and figures below.



Total resolution of bisphosphonates, excipients and anions.



FMS Spectra of three bisphosphonate pharmaceuticals.

Implementation of a Walk-Up High-Pressure Capillary Ion Chromatograph for the Fast Separation of Pharmaceutical Relevant Inorganic Anions and Cations

Summary

Ion chromatography (IC) with suppressed conductivity detection is a well-established technique for the determination of inorganic and organic ions in pharmaceuticals. This work describes the development of a walk-up IC system using high-pressure to provide ultrafast separations of inorganic anions and cations relevant to the pharmaceutical industry. Data will be presented on the identification, quantification, and control of inorganic impurities that are important during drug development, and the benefits an Always Ready system brings to IC analysis. Additionally, ion exchange with suppressed conductivity detection provides a highly sensitive and selective detection mode for the analysis of counter ions in pharmaceutical formulations.

Download the Application Note

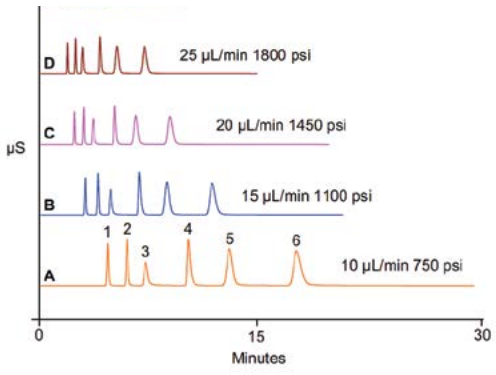
Anions and Cations in Pharmaceuticals

Equipment

- Dionex ICS-5000 Reagent-Free Capillary IC system* consisting of:
  - DP Dual isocratic capillary pump
  - DC Detector and Chromatography Module
  - Dionex IC Cube capillary module compartment
  - CD Capillary Conductivity Detector for Anions and Cations
  - EG Eluent Generator
  - Dionex AS-AP Autosampler with diverter valve
- Chromeleon CDS software

*Dionex ICS-6000 HPIC system or Dionex ICS-4000 HPIC system can be used for equivalent results

Column:	Dionex IonPac CS16 column, Capillary 0.5 × 250 mm		
Eluent Source:	Dionex EGC-MSA Cartridge (Capillary)		
Eluent:	30 mM MSA		
Peaks:	1. Lithium	0.5	mg/L
	2. Sodium	2.0	
	3. Ammonium	2.5	
	4. Potassium	5.0	
	5. Magnesium	2.5	
	6. Calcium	5.0	



Fast cation determinations by high-pressure capillary IC.

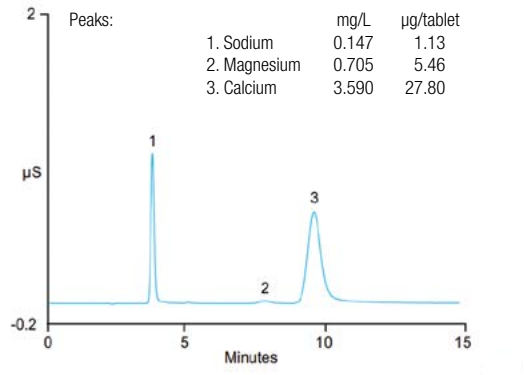
Analysis

Capillary Ion Chromatography

Results

See chromatograms below.

Column:	Dionex IonPac CS12A column, 0.4 × 250 mm		
Eluent Source:	Dionex EGC-MSA Cartridge (Capillary)		
Gradient:	20 mM MSA		
Flow Rate:	0.010 mL/min		
Inj. Volume:	0.4 µL		
Column Temp.:	30 °C		
Detection:	Dionex CCES 300 suppressor		
Sample:	1.29 mg/mL of 10 mg tablet in water		
Sample Prep:	Filtered, Thermo Scientific™ Dionex™ OnGuard™ RP Cartridge, 1:10 dilution		
Peaks:		mg/L	µg/tablet
	1. Sodium	0.147	1.13
	2. Magnesium	0.705	5.46
	3. Calcium	3.590	27.80



Counterion determinations in a atorvastatin calcium tablet by capillary IC.

Thermo Scientific

AppsLab Library of Analytical Applications

The AppsLab Library of Analytical Applications is a fully searchable online, analytical method repository where you can find applications with detailed method information, chromatograms and related compound information.

Discover the latest applications from Thermo Fisher Scientific for LC, IC, GC, GC-MS and LC-MS instruments. Search by compound, column, instrument or any other method parameter and view key method parameters.

Download one-click eWorkflows, created and tested by Thermo Fisher Scientific application scientists, which can be directly executed in your chromatography data system.

Visit: https://appslab.thermofisher.com

Find out more at **www.thermofisher.com/CapillaryIC**

ThermoFisher SCIENTIFIC

TG70668-EN 1217S